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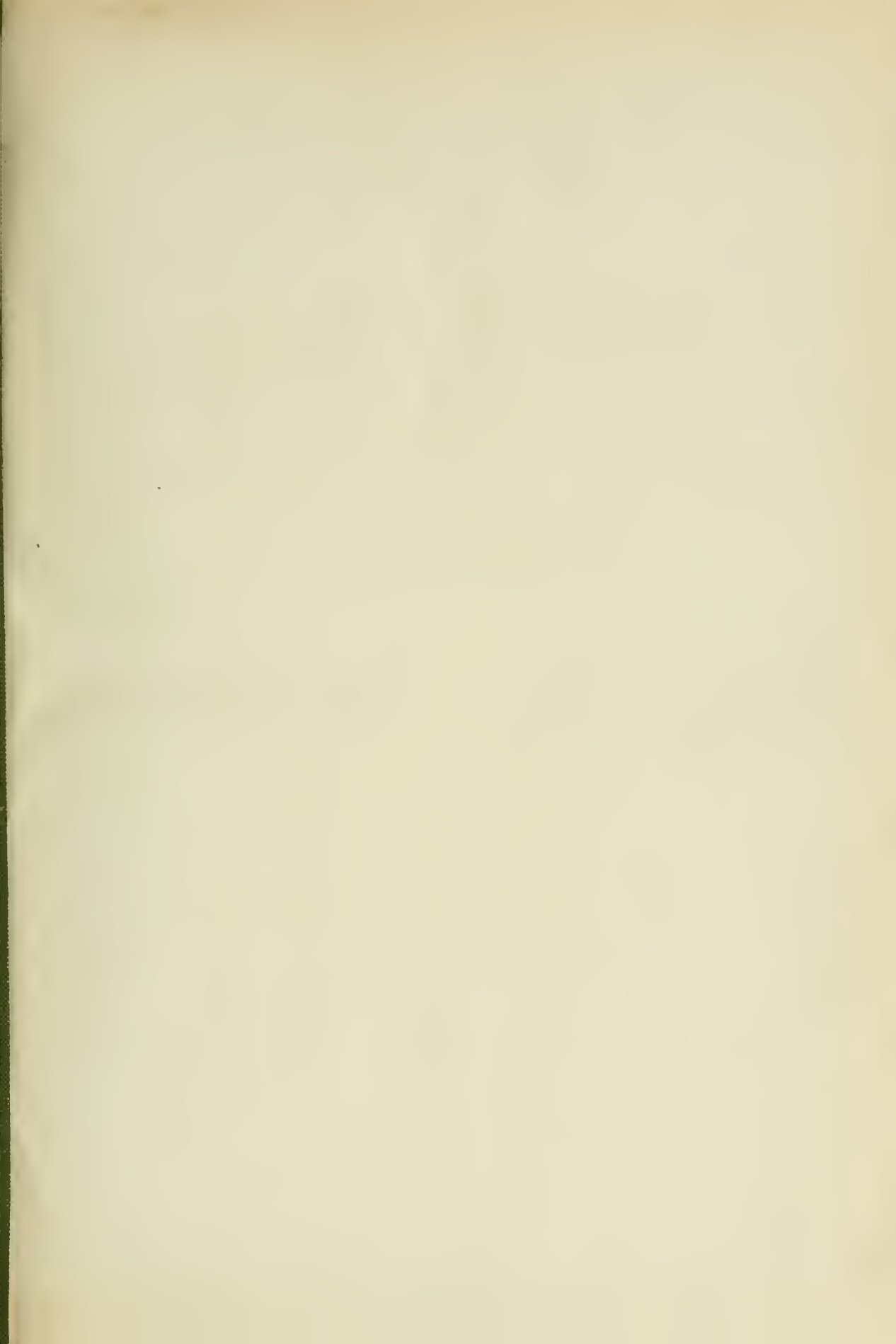
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100,777	124A	107,589	301A	111,309	30A	112,165	81A	113,002	130A	113,777	418A	114,620	357A
101,219	235A	107,593	528A	111,313	30A	112,166	107A	113,010	146A	113,778	337A	114,641	419A
101,220	362A	107,599	482A	111,314	30A	112,177	96A	113,012	177A	113,779	362A	114,645	296A
101,723	45A	107,600	525A	111,317	3A	112,179	94A	113,015	173A	113,780	495A	114,651	289A
101,875	45A	107,754	67A	111,318	14A	112,188	81A	113,017	149A	113,785	735A	114,658	293A
102,049	35A	107,770	627A	111,321	5A	112,196	89A	113,025	176A	113,794	243A	114,663	302A
102,146	517A	107,961	235A	111,328	4A	112,199	93A	113,036	156A	113,810	209A	114,680	282A
102,375	15A	107,966	86A	111,340	37A	112,220	94A	113,048	174A	113,812	242A	114,684	308A
102,507	31A	107,969	553A	111,341	37A	112,223	106A	113,062	146A	113,825	235A	114,700	321A
102,915	525A	108,298	131A	111,355	31A	112,227	83A	113,069	177A	113,827	249A	114,706	289A
102,967	71A	108,303	317A	111,365	5A	112,232	102A	113,079	177A	113,834	247A	114,743	301A
102,971	263A	108,308	212A	111,377	12A	112,260	576A	113,083	390A	113,835	241A	114,794	419A
102,976	96A	108,311	96A	111,383	33A	112,282	500A	113,094	729A	113,836	247A	114,810	289A
103,290	60A	108,312	518A	111,385	12A	112,293	129A	113,105	191A	113,838	249A	114,816	707A
103,474	425A	108,454	295A	111,401	38A	112,321	94A	113,106	192A	113,839	248A	114,817	416A
103,479	407A	108,459	266A	111,403	1A	112,322	94A	113,131	177A	113,844	237A	114,853	308A
103,480	541A	108,460	76A	111,412	3A	112,329	88A	113,141	202A	113,853	237A	114,859	293A
103,645	340A	108,671	297A	111,413	1A	112,336	94A	113,162	203A	113,856	233A	114,863	289A
103,648	84A	108,850	301A	111,422	38A	112,338	88A	113,181	202A	113,860	254A	114,873	315A
103,649	121A	109,056	506A	111,434	2A	112,339	81A	113,182	176A	113,892	208A	114,878	313A
103,650	121A	109,243	8A	111,466	688A	112,341	83A	113,195	202A	113,900	256A	114,879	313A
103,651	121A	109,247	373A	111,469	707A	112,362	113A	113,197	192A	113,937	233A	114,886	303A
103,652	121A	109,252	45A	111,480	537A	112,365	89A	113,198	192A	113,981	233A	114,888	289A
103,653	146A	109,254	570A	111,485	243A	112,370	118A	113,202	185A	113,983	223A	114,896	302A
103,654	461A	109,255	158A	111,495	48A	112,377	96A	113,203	176A	113,992	229A	114,921	290A
103,655	121A	109,257	185A	111,507	65A	112,380	88A	113,209	151A	114,002	229A	114,933	323A
103,664	52A	109,258	308A	111,508	71A	112,395	120A	113,211	180A	114,012	249A	114,936	292A
103,665	328A	109,264	182A	111,515	25A	112,405	95A	113,232	187A	114,016	256A	114,937	295A
103,670	113A	109,266	374A	111,521	58A	112,412	307A	113,239	177A	114,025	247A	114,945	327A
103,671	447A	109,267	173A	111,533	39A	112,417	637A	113,240	178A	114,026	232A	114,953	309A
103,672	336A	109,435	185A	111,539	58A	112,419	663A	113,259	591A	114,040	250A	114,954	309A
103,812	417A	109,452	771A	111,548	72A	112,460	434A	113,264	760A	114,072	266A	114,971	295A
103,814	377A	109,789	507A	111,556	45A	112,473	135A	113,272	704A	114,105	271A	114,974	380A
103,822	573A	109,790	150A	111,580	61A	112,474	120A	113,276	466A	114,126	226A	114,976	309A
103,827	14A	109,795	142A	111,605	50A	112,482	121A	113,285	178A	114,164	231A	114,977	336A
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104,174	133A	110,154	636A	111,679	61A	112,512	113A	113,333	193A	114,190	283A	115,087	357A
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104,673	328A	110,360	727A	111,738	52A	112,590	129A	113,355	200A	114,228	244A	115,140	327A
104,676	202A	110,364	747A	111,757	62A	112,614	353A	113,373	201A	114,236	241A	115,163	327A
104,684	53A	110,535	520A	111,762	58A	112,615	660A	113,374	192A	114,237	271A	115,193	357A
104,885	250A	110,537	30A	111,774	52A	112,630	688A	113,393	185A	114,257	264A	115,211	357A
105,064	222A	110,540	769A	111,775	58A	112,649	127A	113,408	217A	114,277	275A	115,214	358A
105,065	180A	110,544	301A	111,801	48A	112,653	114A	113,453	203A	114,282	272A	115,218	358A
105,072	280A	110,547	229A	111,826	45A	112,658	128A	113,454	203A	114,288	230A	115,222	426A
105,222	132A	110,691	5A	111,835	519A	112,672	136A	113,457	206A	114,304	687A	115,223	643A
105,543	173A	110,715	6A	111,843	621A	112,678	146A	113,483	222A	114,328	271A	115,247	380A
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105,558	271A	110,750	217A	111,858	262A	112,700	114A	113,486	220A	114,339	263A	115,254	358A
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105,563	292A	110,758	185A	111,871	50A	112,741	143A	113,493	217A	114,353	261A	115,261	387A
105,744	223A	110,906	282A	111,875	56A	112,747	141A	113,494	203A	114,358	274A	115,278	367A
105,745	167A	110,907	45A	111,883	49A	112,766	606A	113,505	222A	114,359	271A	115,311	359A
105,746	193A	110,910	613A	111,890	58A	112,791	141A	113,508	212A	114,370	261A	115,316	373A
105,912	520A	110,941	6A	111,904	52A	112,792	154A	113,510	203A	114,371	263A	115,323	378A
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106,086	167A	111,101	1A	111,916	93A	112,865	165A	113,557	208A	114,442	320A	115,409	378A
106,087	193A	111,102	726A	111,933	49A	112,876	154A	113,560	220A				

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115,543	378A	116,334	434A	116,946	518A	117,649	579A	118,313	571A	118,993	651A	119,639	678A
115,546	359A	116,348	486A	116,952	528A	117,666	600A	118,323	594A	119,007	669A	119,642	723A
115,556	438A	116,357	483A	116,958	495A	117,683	540A	118,332	574A	119,018	661A	119,700	727A
115,573	363A	116,360	463A	116,966	525A	117,693	579A	118,334	566A	119,049	682A	119,706	723A
115,609	359A	116,361	418A	116,974	518A	117,704	538A	118,346	628A	119,050	739A	119,711	742A
115,644	678A	116,365	466A	117,016	554A	117,713	602A	118,353	571A	119,052	713A	119,712	742A
115,662	448A	116,366	461A	117,022	528A	117,715	576A	118,355	706A	119,066	682A	119,713	742A
115,686	442A	116,384	475A	117,023	554A	117,721	538A	118,357	592A	119,074	710A	119,723	726A
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115,693	371A	116,406	404A	117,039	503A	117,755	579A	118,394	631A	119,100	706A	119,779	739A
115,696	373A	116,418	443A	117,042	577A	117,761	589A	118,397	584A	119,101	700A	119,783	768A
115,697	378A	116,438	466A	117,047	513A	117,781	550A	118,398	584A	119,106	714A	119,787	739A
115,705	381A	116,445	456A	117,054	518A	117,785	554A	118,425	628A	119,117	704A	119,817	739A
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115,750	378A	116,458	458A	117,087	501A	117,823	556A	118,448	620A	119,156	678A	119,834	745A
115,847	659A	116,477	418A	117,095	502A	117,824	606A	118,461	662A	119,164	706A	119,835	726A
115,865	429A	116,486	482A	117,103	496A	117,828	599A	118,465	613A	119,173	688A	119,878	742A
115,866	428A	116,490	713A	117,104	558A	117,842	539A	118,466	631A	119,200	704A	119,881	765A
115,867	406A	116,503	473A	117,117	554A	117,843	539A	118,470	616A	119,207	682A	119,885	726A
115,870	426A	116,512	453A	117,121	519A	117,852	603A	118,482	634A	119,210	710A	119,902	742A
115,872	440A	116,521	480A	117,147	513A	117,857	554A	118,488	628A	119,249	717A	119,911	728A
115,877	432A	116,530	453A	117,173	496A	117,864	607A	118,489	636A	119,250	717A	119,923	747A
115,899	413A	116,532	474A	117,180	518A	117,870	546A	118,511	620A	119,252	708A	119,924	766A
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115,936	405A	116,603	438A	117,286	517A	117,917	603A	118,664	622A	119,288	679A	120,018	747A
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115,937	404A	116,620	480A	117,308	500A	117,923	569A	118,668	670A	119,298	688A	120,076	756A
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116,118	414A	116,735	416A	117,372	617A	117,987	576A	118,701	623A	119,366	700A	120,219	767A
116,119	406A	116,745	468A	117,383	513A	117,991	580A	118,702	622A	119,367	678A	120,223	761A
116,123	477A	116,749	399A	117,387	500A	118,000	589A	118,712	622A	119,368	687A	120,230	761A
116,129	462A	116,754	407A	117,414	502A	118,021	570A	118,727	619A	119,376	682A	120,255	768A
116,139	426A	116,758	436A	117,417	529A	118,037	590A	118,730	618A	119,399	682A	120,256	781A
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116,181	426A	116,769	398A	117,469	496A	118,063	546A	118,750	659A	119,430	713A	120,276	755A
116,185	456A	116,786	398A	117,470	519A	118,116	700A	118,768	629A	119,440	760A	120,277	776A
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116,235	426A	116,839	399A	117,483	506A	118,156	580A	118,812	659A	119,485	691A	120,293	781A
116,253	593A	116,853	429A	117,486	531A	118,182	658A	118,825	660A	119,486	677A	120,294	781A
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116,321	466A	116,935	521A	117,586	497A	118,289	742A	118,940	669A	119,591	691A	120,520	780A
116,322	477A	116,936	555A	117,637	538A	118,291	575A	118,947	660A	119,594	687A		
116,323	433A	116,939	501A	117,658	550A	118,296	628A	118,960	647A	119,599	723A		
116,324	477A	116,940	501A	117,640	574A	118,305	631A	118,983	658A	119,618	771A		
116,326	433A	116,943	518A	117,645	540A	118,312	590A						

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(Note.—The letter "R" indicates that a reissue of the patent is referred to.)

1,087,422R	775A	1,241,884	1A	1,245,634	13A	1,246,344	46A	1,246,867	30A	1,247,516	34A	1,248,065	74A
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1,123,448R	467A	1,244,901	20A	1,245,690	6A	1,246,379	64A	1,246,939	25A	1,247,541	38A	1,248,139	75A
1,223,121R	17A	1,244,902	20A	1,245,694	6A	1,246,514	45A	1,247,059	67A	1,247,542	38A	1,248,213	94A
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1,243,811	17A	1,245,273	14A	1,245,975	27A	1,246,641	47A	1,247,165	56A	1,247,619	56A	1,248,389	65A
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1,244,444	4A	1,245,349	31A	1,245,980	33A	1,246,802	62A	1,247,278	56A	1,247,757	54A	1,248,463	66A
1,244,453	19A	1,245,353	4A	1,245,981	34A	1,246,805	48A	1,247,280	56A	1,247,782	64A	1,248,486	59A
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1,244,467	3A	1,245,383	30A	1,245,983	27A	1,246,807	48A	1,247,337	31A	1,247,833	106A	1,248,501	65A



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1,248,864	75A	1,250,934	164A	1,252,742	149A	1,254,438	200A	1,255,913	226A	1,257,737	238A	1,259,536	302A
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1,248,885	48A	1,251,038	144A	1,252,785	183A	1,254,457	191A	1,255,951	223A	1,257,772	234A	1,259,594	308A
1,248,884	71A	1,251,039	144A	1,252,865	165A	1,254,510	187A	1,255,983	242A	1,257,829	234A	1,259,595	310A
1,248,888	66A	1,251,057	124A	1,252,887	156A	1,254,518	198A	1,255,995	244A	1,257,854	231A	1,259,631	320A
1,248,924	96A	1,251,058	124A	1,252,962	143A	1,254,519	198A	1,256,032	199A	1,257,882	231A	1,259,632	320A
1,248,925	95A	1,251,064	117A	1,252,973	158A	1,254,521	181A	1,256,067	249A	1,257,886	239A	1,259,633	320A
1,248,933	75A	1,251,170	131A	1,252,975	169A	1,254,531	185A	1,256,068	241A	1,257,894	242A	1,259,634	320A
1,248,950	49A	1,251,182	131A	1,252,976	148A	1,254,588	187A	1,256,084	256A	1,257,895	233A	1,259,635	320A
1,248,951	47A	1,251,189	128A	1,252,985	204A	1,254,606	173A	1,256,171	256A	1,257,906	244A	1,259,636	320A
1,249,001	49A	1,251,201	123A	1,252,986	204A	1,254,676	181A	1,256,189	238A	1,257,923	244A	1,259,637	320A
1,249,019	53A	1,251,202	123A	1,252,999	144A	1,254,677	181A	1,256,200	244A	1,257,943	272A	1,259,651	303A
1,249,028	59A	1,251,203	123A	1,253,000	144A	1,254,689	173A	1,256,229	230A	1,257,956	226A	1,259,669	479A
1,249,041	50A	1,251,204	123A	1,253,026	174A	1,254,693	173A	1,256,233	233A	1,257,990	272A	1,259,674	263A
1,249,050	65A	1,251,237	146A	1,253,048	202A	1,254,700	178A	1,256,237	243A	1,257,995	272A	1,259,675	289A
1,249,061	95A	1,251,251	146A	1,253,055	157A	1,254,727	179A	1,256,240	237A	1,257,996	264A	1,259,683	266A
1,249,075	94A	1,251,261	146A	1,253,060	150A	1,254,734	174A	1,256,272	241A	1,257,997	273A	1,259,702	302A
1,249,125	57A	1,251,273	144A	1,253,061	150A	1,254,736	184A	1,256,273	241A	1,258,017	261A	1,259,717	302A
1,249,177	46A	1,251,275	132A	1,253,064	154A	1,254,805	186A	1,256,285	248A	1,258,047	280A	1,259,718	302A
1,249,180	66A	1,251,285	118A	1,253,238	148A	1,254,810	179A	1,256,291	198A	1,258,055	261A	1,259,719	302A
1,249,181	66A	1,251,295	124A	1,253,252	145A	1,254,833	193A	1,256,293	283A	1,258,076	280A	1,259,757	283A
1,249,205	71A	1,251,296	181A	1,253,261	158A	1,254,836	193A	1,256,295	241A	1,258,083	264A	1,259,758	283A
1,249,226	95A	1,251,302	156A	1,253,262	158A	1,254,841	193A	1,256,296	242A	1,258,104	262A	1,259,786	293A
1,249,232	49A	1,251,309	144A	1,253,334	190A	1,254,844	186A	1,256,301	234A	1,258,106	275A	1,259,788	310A
1,249,252	53A	1,251,317	142A	1,253,342	145A	1,254,866	188A	1,256,319	249A	1,258,165	261A	1,259,793	313A
1,249,259	69A	1,251,333	141A	1,253,347	164A	1,254,908	193A	1,256,324	243A	1,258,168	266A	1,259,794	313A
1,249,272	66A	1,251,341	127A	1,253,362	155A	1,254,914	203A	1,256,356	237A	1,258,190	262A	1,259,806	313A
1,249,278	49A	1,251,359	135A	1,253,397	189A	1,254,970	202A	1,256,368	213A	1,258,191	262A	1,259,839	297A
1,249,287	53A	1,251,368	120A	1,253,401	142A	1,254,987	186A	1,256,429	247A	1,258,196	262A	1,259,840	297A
1,249,314	88A	1,251,369	200A	1,253,403	142A	1,254,992	182A	1,256,455	209A	1,258,203	265A	1,259,842	311A
1,249,355	101A	1,251,412	156A	1,253,404	158A	1,255,007	198A	1,256,456	209A	1,258,225	265A	1,259,898	311A
1,249,360	100A	1,251,422	181A	1,253,411	145A	1,255,015	200A	1,256,460	233A	1,258,227	272A	1,260,020	286A
1,249,368	106A	1,251,426	143A	1,253,428	142A	1,255,018	200A	1,256,494	232A	1,258,261	272A	1,260,037	308A
1,249,392	114A	1,251,485	128A	1,253,445	144A	1,255,020	181A	1,256,495	256A	1,258,265	273A	1,260,072	312A
1,249,444	84A	1,251,486	115A	1,253,487	141A	1,255,047	200A	1,256,496	216A	1,258,266	273A	1,260,103	321A
1,249,479	96A	1,251,511	155A	1,253,497	149A	1,255,049	204A	1,256,513	242A	1,258,282	267A	1,260,119	310A
1,249,480	104A	1,251,522	130A	1,253,534	150A	1,255,066	186A	1,256,535	251A	1,258,320	266A	1,260,135	290A
1,249,511	104A	1,251,533	146A	1,253,556	174A	1,255,096	187A	1,256,536	251A	1,258,321	266A	1,260,286	292A
1,249,557	114A	1,251,535	150A	1,253,560	149A	1,255,116	183A	1,256,537	251A	1,258,334	261A	1,260,298	312A
1,249,575	86A	1,251,538	142A	1,253,571	150A	1,255,138	200A	1,256,541	244A	1,258,348	280A	1,260,312	310A
1,249,636	89A	1,251,564	133A	1,253,590	150A	1,255,139	213A	1,256,570	237A	1,258,405	262A	1,260,327	347A
1,249,637	89A	1,251,564	136A	1,253,591	150A	1,255,144	206A	1,256,593	242A	1,258,427	280A	1,260,330	290A
1,249,677	86A	1,251,568	155A	1,253,615	167A	1,255,149	200A	1,256,605	242A	1,258,529	272A	1,260,398	303A
1,249,684	95A	1,251,569	155A	1,253,616	167A	1,255,162	197A	1,256,623	248A	1,258,542	265A	1,260,406	291A
1,249,708	88A	1,251,614	121A	1,253,617	167A	1,255,164	198A	1,256,634	280A	1,258,543	265A	1,260,407	291A
1,249,728	88A	1,251,621	130A	1,253,622	150A	1,255,170	198A	1,256,664	251A	1,258,544	265A	1,260,448	297A
1,249,739	88A	1,251,741	124A	1,253,653	155A	1,255,191	211A	1,256,675	224A	1,258,545	265A	1,260,472	311A
1,249,740	106A	1,251,742	131A	1,253,667	183A	1,255,192	216A	1,256,697	230A	1,258,546	265A	1,260,492	301A
1,249,787	95A	1,251,841	125A	1,253,674	165A	1,255,196	198A	1,256,703	272A	1,258,562	277A	1,260,493	301A
1,249,821	88A	1,251,842	125A	1,253,691	167A	1,255,197	213A	1,256,730	230A	1,258,563	277A	1,260,508	297A
1,249,835	114A	1,251,862	131A	1,253,732	156A	1,255,268	206A	1,256,758	255A	1,258,568	264A	1,260,535	296A
1,249,854	94A	1,251,863	131A	1,253,747	144A	1,255,283	213A	1,256,789	315A	1,258,587	284A	1,260,570	297A
1,249,863	88A	1,251,888	124A	1,253,760	176A	1,255,286	198A	1,256,802	248A	1,258,627	280A	1,260,584	293A
1,249,864	84A	1,251,931	124A	1,253,775	150A	1,255,292	220A	1,256,810	230A	1,258,654	292A	1,260,621	349A
1,249,886	114A	1,251,932	124A	1,253,793	158A	1,255,293	220A	1,256,825	256A	1,258,666	309A	1,260,622	349A
1,249,937	89A	1,251,938	123A	1,253,793	150A	1,255,335	223A	1,256,864	249A	1,258,747	315A	1,260,656	347A
1,249,960	89A	1,251,951	129A	1,253,840	165A	1,255,340	183A	1,256,875	242A	1,258,800	309A	1,260,660	307A
1,249,997	91A	1,251,954	117A	1,253,844	183A	1,255,358	221A	1,256,894	256A	1,258,869	309A	1,260,661	310A
1,250,052	84A	1,252,000	118A	1,253,853	146A	1,255,359	221A	1,256,929	234A	1,258,885	309A	1,260,668	310A
1,250,063	88A	1,252,001	131A	1,253,854	146A	1,255,390	193A	1,256,930	234A	1,258,886	309A	1,260,681	302A
1,250,071	95A	1,252,010	128A	1,253,987	174A	1,255,391	213A	1,256,935	242A	1,258,888	317A	1,260,707	283A
1,250,072	105A	1,252,013	125A	1,253,990	167A	1,255,423	193A	1,256,939	248A	1,258,889	317A	1,260,731	294A
1,250,079	106A	1,252,023	127A	1,254,006	204A	1,255,433	212A	1,256,952	255A	1,258,895	302A	1,260,740	308A
1,250,088	114A	1,252,024	124A	1,254,007	176A	1,255,434	212A	1,256,954	272A	1,258,912	292A	1,260,787	307A
1,250,094	115A	1,252,032	144A	1,254,009	165A	1,255,435	212A	1,256,958	249A	1,258,913	265A	1,260,803	310A
1,250,106	120A	1,252,033	144A	1,254,033	190A	1,255,436	212A	1,256,959	249A	1,258,934	310A	1,260,811	340A
1,250,132	166A	1,252,038	128A	1,254,045	186A	1,255,437	212A	1,256,972	247A	1,258,984	295A	1,260,812	340A
1,250,141	156A	1,252,058	131A	1,254,048	226A	1,255,438	212A	1,256,974	251A	1,258,988	289A	1,260,852	296A
1,250,146	156A	1,252,090	136A	1,254,056	186A	1,255,439	212A	1,256,981	284A	1,258,996	320A	1,260,899	319A
1,250,183	129A	1,252,104	142A	1,254,073	197A	1,255,440	212A	1,257,004	231A	1,258,997	320A	1,260,940	310A
1,250,216	123A	1,252,178	197A	1,254,077	186A	1,255,450	208A	1,257,080	238A	1,259,006	290A	1,260,944	302A
1,250,228	118A	1,252,183	197A	1,254,078	186A	1,255,474	206A	1,257,136	251A	1,259,008	290A	1,260,970	294A
1,250,258	115A	1,252,185	129A	1,254,079	186A	1,255,499	212A	1,257,150	243A</				



No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.	No. of Patent.	Page.
1,261,395	428A	1,263,607	418A	1,265,274	525A	1,266,957	543A	1,269,006	559A	1,270,919	569A	1,272,801	565A
1,261,398	399A	1,263,618	363A	1,265,275	525A	1,267,004	550A	1,269,054	551A	1,270,962	539A	1,272,851	574A
1,261,410	405A	1,263,620	360A	1,265,298	399A	1,267,005	550A	1,269,067	537A	1,270,963	539A	1,272,855	581A
1,261,498	412A	1,263,634	386A	1,265,318	399A	1,267,012	506A	1,269,078	592A	1,270,989	580A	1,272,880	582A
1,261,499	412A	1,263,635	386A	1,265,326	476A	1,267,022	497A	1,269,080	557A	1,270,990	566A	1,272,917	591A
1,261,500	412A	1,263,656	379A	1,265,330	575A	1,267,023	496A	1,269,100	600A	1,270,997	579A	1,272,918	566A
1,261,501	412A	1,263,685	332A	1,265,332	463A	1,267,024	496A	1,269,110	551A	1,271,002	580A	1,272,922	609A
1,261,526	415A	1,263,695	428A	1,265,346	604A	1,267,081	558A	1,269,120	565A	1,271,013	636A	1,272,952	593A
1,261,542	443A	1,263,705	414A	1,265,360	466A	1,267,102	565A	1,269,128	592A	1,271,028	553A	1,272,979	570A
1,261,572	415A	1,263,706	415A	1,265,369	528A	1,267,141	552A	1,269,132	537A	1,271,051	551A	1,272,987	607A
1,261,606	428A	1,263,707	415A	1,265,370	469A	1,267,204	558A	1,269,134	539A	1,271,052	547A	1,273,050	570A
1,261,615	433A	1,263,708	419A	1,265,378	459A	1,267,209	544A	1,269,141	545A	1,271,067	544A	1,273,054	607A
1,261,645	406A	1,263,709	418A	1,265,453	474A	1,267,317	513A	1,269,150	550A	1,271,071	540A	1,273,058	566A
1,261,694	427A	1,263,710	418A	1,265,459	518A	1,267,332	499A	1,269,157	550A	1,271,093	592A	1,273,072	603A
1,261,695	415A	1,263,727	378A	1,265,464	437A	1,267,347	545A	1,269,168	596A	1,271,111	560A	1,273,091	570A
1,261,696	415A	1,263,747	386A	1,265,485	593A	1,267,380	616A	1,269,170	580A	1,271,112	537A	1,273,110	581A
1,261,736	399A	1,263,796	389A	1,265,545	468A	1,267,400	551A	1,269,173	592A	1,271,114	539A	1,273,127	566A
1,261,742	425A	1,263,813	433A	1,265,547	474A	1,267,410	500A	1,269,189	599A	1,271,115	539A	1,273,144	603A
1,261,743	425A	1,263,817	387A	1,265,549	469A	1,267,419	557A	1,269,222	547A	1,271,118	553A	1,273,145	603A
1,261,750	336A	1,263,823	367A	1,265,551	474A	1,267,428	506A	1,269,223	545A	1,271,136	550A	1,273,146	590A
1,261,767	330A	1,263,831	379A	1,265,573	404A	1,267,473	522A	1,269,224	545A	1,271,144	576A	1,273,171	584A
1,261,810	310A	1,263,832	379A	1,265,574	404A	1,267,513	501A	1,269,236	545A	1,271,146	552A	1,273,177	569A
1,261,811	349A	1,263,852	371A	1,265,582	522A	1,267,565	499A	1,269,270	537A	1,271,172	552A	1,273,191	617A
1,261,858	365A	1,263,893	360A	1,265,641	487A	1,267,578	497A	1,269,287	600A	1,271,192	545A	1,273,202	591A
1,261,900	303A	1,263,906	443A	1,265,655	469A	1,267,611	519A	1,269,292	709A	1,271,200	552A	1,273,208	581A
1,261,907	339A	1,263,950	363A	1,265,665	458A	1,267,638	507A	1,269,339	709A	1,271,229	545A	1,273,220	590A
1,2 1,911	340A	1,263,959	381A	1,265,684	457A	1,267,646	499A	1,269,340	687A	1,271,246	604A	1,273,223	591A
1,261,921	333A	1,263,962	351A	1,265,694	523A	1,267,653	553A	1,269,350	575A	1,271,267	552A	1,273,233	574A
1,261,930	330A	1,263,981	358A	1,265,700	528A	1,267,669	551A	1,269,392	540A	1,271,268	615A	1,273,297	614A
1,261,948	339A	1,264,043	360A	1,265,746	454A	1,267,683	503A	1,269,399	602A	1,271,269	633A	1,273,345	585A
1,261,987	339A	1,264,092	432A	1,265,800	531A	1,267,686	514A	1,269,418	561A	1,271,271	566A	1,273,346	585A
1,262,019	339A	1,264,162	414A	1,265,809	462A	1,267,696	519A	1,269,439	572A	1,271,280	592A	1,273,357	593A
1,262,020	339A	1,264,185	432A	1,265,836	460A	1,267,699	599A	1,269,441	590A	1,271,293	566A	1,273,358	590A
1,262,015	339A	1,264,186	432A	1,265,837	460A	1,267,701	503A	1,269,442	590A	1,271,349	537A	1,273,370	582A
1,262,057	333A	1,264,206	432A	1,265,838	525A	1,267,709	558A	1,269,443	590A	1,271,364	537A	1,273,392	600A
1,262,062	339A	1,264,229	428A	1,265,855	477A	1,267,711	501A	1,269,476	574A	1,271,367	537A	1,273,396	614A
1,262,063	339A	1,264,236	425A	1,265,863	454A	1,267,737	503A	1,269,520	571A	1,271,371	600A	1,273,432	593A
1,262,072	331A	1,264,237	425A	1,265,864	531A	1,267,772	497A	1,269,562	604A	1,271,387	539A	1,273,457	607A
1,262,115	303A	1,264,254	410A	1,265,865	531A	1,267,801	551A	1,269,565	589A	1,271,392	555A	1,273,465	591A
1,262,126	310A	1,264,263	400A	1,265,892	414A	1,267,819	572A	1,269,566	593A	1,271,393	555A	1,273,474	571A
1,262,146	291A	1,264,288	432A	1,265,893	414A	1,267,849	513A	1,269,599	610A	1,271,425	604A	1,273,477	581A
1,262,190	310A	1,264,316	418A	1,265,923	473A	1,267,888	540A	1,269,627	554A	1,271,506	538A	1,273,506	591A
1,262,228	339A	1,264,317	418A	1,265,944	473A	1,267,976	551A	1,269,666	593A	1,271,549	576A	1,273,523	617A
1,262,248	310A	1,264,336	438A	1,265,945	474A	1,268,015	506A	1,269,678	599A	1,271,550	576A	1,273,529	585A
1,262,251	295A	1,264,347	399A	1,265,973	590A	1,268,030	551A	1,269,700	537A	1,271,560	590A	1,273,547	585A
1,262,267	330A	1,264,360	440A	1,265,975	488A	1,268,070	529A	1,269,747	539A	1,271,575	594A	1,273,568	638A
1,262,263	330A	1,264,435	405A	1,266,008	454A	1,268,076	531A	1,269,751	620A	1,271,576	594A	1,273,571	632A
1,262,295	333A	1,264,459	426A	1,266,013	454A	1,268,100	500A	1,269,754	603A	1,271,578	608A	1,273,595	614A
1,262,302	340A	1,264,479	398A	1,266,041	415A	1,268,142	520A	1,269,792	560A	1,271,591	574A	1,273,598	709A
1,262,305	335A	1,264,509	414A	1,266,073	461A	1,268,193	543A	1,269,795	539A	1,271,633	580A	1,273,619	569A
1,262,384	327A	1,264,510	415A	1,266,080	499A	1,268,217	584A	1,269,863	575A	1,271,652	584A	1,273,628	571A
1,262,432	339A	1,264,511	415A	1,266,088	404A	1,268,233	559A	1,269,895	538A	1,271,654	601A	1,273,629	571A
1,262,448	327A	1,264,512	415A	1,266,092	541A	1,268,240	507A	1,269,905	547A	1,271,681	566A	1,273,653	614A
1,262,452	339A	1,264,513	415A	1,266,108	463A	1,268,273	497A	1,269,913	593A	1,271,712	615A	1,273,673	595A
1,262,453	339A	1,264,514	415A	1,266,109	463A	1,268,274	497A	1,269,914	593A	1,271,713	581A	1,273,675	595A
1,262,472	346A	1,264,535	416A	1,266,110	463A	1,268,323	551A	1,269,915	593A	1,271,738	575A	1,273,690	581A
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1,262,576	330A	1,264,551	433A	1,266,133	454A	1,268,430	502A	1,269,926	551A	1,271,790	607A	1,273,758	571A
1,262,589	334A	1,264,564	437A	1,266,147	458A	1,268,433	506A	1,269,931	709A	1,271,810	596A	1,273,762	591A
1,262,738	327A	1,264,572	416A	1,266,198	479A	1,268,441	544A	1,269,934	576A	1,271,846	607A	1,273,772	581A
1,262,769	349A	1,264,586	427A	1,266,199	479A	1,268,446	547A	1,269,974	550A	1,271,869	581A	1,273,791	565A
1,262,770	331A	1,264,600	435A	1,266,200	419A	1,268,447	551A	1,269,994	537A	1,271,925	603A	1,273,797	608A
1,262,808	330A	1,264,604	408A	1,266,203	478A	1,268,455	574A	1,270,042	613A	1,271,926	603A	1,273,817	614A
1,262,872	366A	1,264,628	427A	1,266,212	467A	1,268,465	550A	1,270,055	709A	1,271,961	710A	1,273,824	622A
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1,262,984	379A	1,264,747	419A	1,266,335	476A	1,268,513	543A	1,270,270	531A	1,272,053	604A	1,273,920	659A
1,263,031	383A	1,264,796	433A	1,266,339	415A	1,268,532	514A	1,270,271	531A	1,272,054	604A	1,273,927	619A
1,263,045	411A	1,264,802	416A	1,266,354	523A	1,268,533	514A	1,270,290	620A	1,272,059	570A	1,273,927	619A
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1,263,119	367A	1,264,832	427A	1,266,406									



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1,274,247	622A	1,275,184	613A	1,276,290	662A	1,277,056	652A	1,277,896	732A	1,278,854	772A	1,279,828	768A
1,274,249	659A	1,275,206	662A	1,276,307	652A	1,277,057	686A	1,277,898	733A	1,278,885	729A	1,279,860	772A
1,274,250	660A	1,275,231	643A	1,276,377	654A	1,277,085	679A	1,277,899	733A	1,278,991	768A	1,279,888	775A
1,274,310	622A	1,275,232	704A	1,276,383	708A	1,277,136	647A	1,277,900	733A	1,279,037	740A	1,279,889	772A
1,274,323	651A	1,275,242	659A	1,276,385	708A	1,277,136	652A	1,277,904	729A	1,279,040	772A	1,279,928	773A
1,274,324	651A	1,275,251	666A	1,276,386	678A	1,277,144	733A	1,277,922	724A	1,279,063	757A	1,279,932	756A
1,274,343	666A	1,275,276	666A	1,276,387	678A	1,277,145	733A	1,277,925	687A	1,279,086	772A	1,279,975	768A
1,274,351	649A	1,275,285	663A	1,276,421	643A	1,277,149	682A	1,277,931	711A	1,279,090	766A	1,279,990	762A
1,274,360	659A	1,275,289	663A	1,276,427	643A	1,277,155	667A	1,277,935	683A	1,279,101	771A	1,280,043	755A
1,274,395	620A	1,275,308	669A	1,276,428	645A	1,277,185	686A	1,277,961	723A	1,279,106	775A	1,280,101	757A
1,274,396	620A	1,275,324	669A	1,276,429	646A	1,277,227	700A	1,277,989	705A	1,279,108	772A	1,280,129	775A
1,274,405	617A	1,275,337	647A	1,276,451	644A	1,277,244	660A	1,278,010	701A	1,279,109	767A	1,280,139	767A
1,274,430	663A	1,275,352	656A	1,276,477	670A	1,277,322	663A	1,278,023	683A	1,279,110	772A	1,280,153	775A
1,274,479	622A	1,275,354	657A	1,276,481	663A	1,277,328	647A	1,278,044	710A	1,279,145	767A	1,280,162	775A
1,274,480	616A	1,275,374	660A	1,276,487	647A	1,277,329	647A	1,278,127	724A	1,279,146	773A	1,280,178	761A
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1,274,523	620A	1,275,412	660A	1,276,508	774A	1,277,372	704A	1,278,155	724A	1,279,180	772A	1,280,221	773A
1,274,524	620A	1,275,417	671A	1,276,509	774A	1,277,378	747A	1,278,161	749A	1,279,181	772A	1,280,249	773A
1,274,525	620A	1,275,535	666A	1,276,517	659A	1,277,431	704A	1,278,164	734A	1,279,192	773A	1,280,268	773A
1,274,560	634A	1,275,547	669A	1,276,537	673A	1,277,475	691A	1,278,166	739A	1,279,200	729A	1,280,278	784A
1,274,725	618A	1,275,583	644A	1,276,555	710A	1,277,493	700A	1,278,173	739A	1,279,240	781A	1,280,279	784A
1,274,728	661A	1,275,648	647A	1,276,629	670A	1,277,503	691A	1,278,176	740A	1,279,248	784A	1,280,280	780A
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1,274,916	623A	1,276,058	660A	1,276,957	678A	1,277,708	708A	1,278,493	766A	1,279,458	772A	1,280,625	777A
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1,275,178	623A	1,276,284	648A	1,277,048	672A	1,277,895	724A	1,278,744	740A	1,279,757	760A		

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295,091	672A	302,294	349A	302,786	351A	303,058	347A	303,730	367A	304,603	400A	305,369	443A
299,072	371A	302,305	379A	302,792	407A	303,062	362A	303,731	366A	304,645	428A	305,413	401A
299,546	456A	302,306	377A	302,803	332A	303,064	337A	303,737	348A	304,658	634A	305,417	581A
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301,452	672A	302,322	331A	302,816	379A	303,079	345A	303,754	436A	304,681	467A	305,419	467A
301,583	336A	302,323	369A	302,817	351A	303,095	487A	303,764	426A	304,682	467A	305,427	410A
301,585	440A	302,329	343A	302,825	340A	303,115	337A	303,786	332A	304,737	389A	305,446	419A
301,830	346A	302,330	344A	302,826	340A	303,121	367A	303,799	427A	304,772	410A	305,447	419A
301,929	351A	302,342	347A	302,827	330A	303,122	360A	304,002	370A	304,820	401A	305,448	419A
301,932	337A	302,350	334A	302,834	335A	303,126	472A	304,003	412A	304,802	425A	305,449	419A
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301,971	377A	302,358	377A	302,838	389A	303,144	351A	304,024	382A	304,857	331A	305,702	476A
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302,155	334A	302,635	337A	302,893	409A	303,447	337A	304,078	360A	304,909	432A	305,771	457A
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302,227	440A	302,674	328A	302,973	400A	303,507	473A	304,127	348A	305,239	464A	305,915	466A
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302,255	345A	302,704	336A	302,995	341A	303,557	370A	304,459	405A	305,263	389A	305,956	575A
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302,279	379A	302,753	400A	303,047	386A	303,657	425A	304,574	350A	305,349	457A	306,032	629A
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302,283	377A	302,768	377A	303,053	332A	303,718	362A	304,602	613A	305,367	442A	306,055	585A

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306,059	662A	306,261	740A	306,707	688A	306,892	740A	307,221	723A	307,433	747A	307,857	749A
306,060	570A	306,283	618A	306,725	740A	306,893	764A	307,212	741A	307,434	747A	307,867	764A
306,103	571A	306,505	614A	306,726	734A	306,906	717A	307,244	667A	307,441	747A	307,890	757A
306,104	743A	306,506	615A	306,727	616A	306,921	669A	307,267	682A	307,471	741A	307,902	734A
306,107	575A	306,514	735A	306,748	614A	306,938	636A	307,284	619A	307,483	733A	307,961	761A
306,173	724A	306,515	614A	306,751	746A	306,943	761A	307,312	724A	307,489	724A	307,987	760A
306,193	581A	306,517	745A	306,804	636A	306,952	735A	307,332	706A	307,524	741A	308,036	728A
306,194	581A	306,529	743A	306,805	717A	306,956	684A	307,357	783A	307,525	733A	308,107	733A
306,198	607A	306,567	622A	306,836	618A	306,975	735A	307,359	679A	307,545	780A	308,144	764A
306,214	616A	306,621	618A	306,837	623A	306,978	636A	307,378	705A	307,564	766A	308,259	727A
306,219	619A	306,653	735A	306,839	746A	306,979	672A	307,380	767A	307,579	725A		
306,257	621A	306,658	618A	306,853	740A	306,987	723A	307,399	764A	307,808	769A		
306,259	614A	306,670	614A	306,880	616A	306,988	733A	307,406	705A	307,834	739A		



## List of Journals Abstracted, with Abbreviations Used, and Addresses of Publishers and Prices.

JOURNAL.	ABBREVIATION.	ADDRESS OF PUBLISHER AND PRICE.*
Agricultural Bulletin of the Federated Malay States	Agric. Bull. F.M.S. ...	Dept. of Agriculture, Kuala Lumpur, F.M.S. 1s. 3d.
Agricultural Journal of India	Agric. J. India ...	W. Thacker and Co., 2, Creed Lane, London. 3s.
Agricultural Ledger ...	Agric. Ledger... ...	Reporter on Economic Products to the Government of India, Calcutta.
Agricultural Research Institute, Pusa, Report and Bulletins	Rep. (Bull.) Agric. Res. Inst., Pusa.	Supt. Govt. Printing, Calcutta. Price varies.
Allgemeine Zeitschrift für Bierbrauerei und Malzfabrikation	Allgem. Z. Bierbrau. u. Malzfabr.	Michaelerstrasse 25, Vienna XVIII/1. Krone 12 per annum.
American Journal of Pharmacy.	Amer. J. Pharm. ...	145, North Tenth Street, Philadelphia, Pa., U.S.A. 30 cents.
American Journal of Science	Amer. J. Sci. ...	New Haven, Conn., U.S.A. 50 cents.
Anales de la Sociedad Española de Física y Química	Anal. Soc. Espan. Fis. Quim.	Calle de Núñez de Balboa, núm. 21, Madrid. 15 pesetas per annum.
Anales de la Sociedad Química Argentina	Anal. Soc. Quim. Argentina	1790, Lavalle, Buenos Aires. 60 c.
Analyst ... ..	Analyst ... ..	Simpkin, Marshall, Hamilton, Kent, and Co., Ltd., 2, 4, 6, 8, Orange Street, London, S.W. 2s. (3s. from Jan., 1919.)
Annalen der Chemie ...	Annalen ... ..	C. F. Winter'sche Verlagshandlung, Leipzig, Germany. M. 28 per annum.
Annales de Chimie ... ..	Ann. Chim. ... ..	Imprimerie Gauthier-Villars et Cie., Quai des Grands-Augustins, 55, Paris. 28 fr. per annum.
Annales de Chimie Analytique	Ann. Chim. Analyt. ...	M. Crinon, 20, Boulevard Richard-Lenoir, Paris, 1 fr. 50.
Annales des Falsifications...	Ann. Falsif. ... ..	56, Rue Madame, Paris, 6c. 3 fr.
Annali di Chimica Applicata	Annali Chim. Appl. ...	Via Panisperna N. 896, Rome. Lire 34 per annum.
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Apotheker-Zeitung ... ..	Apoth.-Zeit. ... ..	Levetzowstrasse 16b, Berlin, N.W. 87. 25 Pf.
Archiv der Pharmazie ...	Arch. Pharm... ..	Selbstverlag des Deutschen Apotheker-Vereins, Berlin, Germany. M. 12 per annum.
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Australasian Pharmaceutical Notes and News	Austr. Pharm. Notes and News	Elliott Bros., Ltd., O'Connell St., Sydney, N.S.W. 3d.
Berichte der Deutschen Chemischen Gesellschaft	Ber. ... ..	R. Friedländer und Sohn, Karlstrasse 11, Berlin, N.W. 6, Germany. M. 32 per annum.
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Bio-chemical Journal ...	Bio-chem. J. ... ..	Cambridge University Press, Fetter Lane, London, E.C. 7s.
Biochemische Zeitschrift ...	Biochem. Zeits. ...	Verlag von J. Springer, Berlin, W. 9, Linkstrasse 23/24. M. 22 per volume.
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Brewers' Journal ... ..	Brewers' J. ... ..	F. W. Lyon, Eastcheap Buildings, Eastcheap, London, E.C. 3. 3s.
British and Colonial Pharmacist	Brit. and Col. Pharm.	194-200, Bishopsgate, London, E.C. 1s.

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Bulletin of the Forest Experiment Station, Meguro, Tokyo	Bull. Forest Exp. Stat., Meguro	Bureau of Forestry, Dept. of Agriculture and Commerce, Tokyo.
Bulletin of the Kentucky Agricultural Experiment Station	Bull. Kentucky Agric. Exp. Stat.	University of Kentucky, Lexington, Ky., U.S.A.
Bulletin of the School of Mines and Metallurgy, University of Missouri	Bull. School Mines and Met., Univ. Missouri	Director of Mining Experiment Station, Rolla, Mo., U.S.A.
Bulletin de la Société Chimique de Belgique	Bull. Soc. Chim. Belg.	M. J. Wauters, Palais du Midi (Galerie du Travail 7), Brussels.
Bulletin de la Société Chimique de France	Bull. Soc. Chim. ...	Masson et Cie., 120, Boulevard Saint-Germain, Paris, 6e. 38 fr. per annum.
Bulletin de la Société Française de Photographie	Bull. Soc. Franç. Phot.	51, Rue de Clichy, Paris, 9e. 1 fr. 50.
Bulletin de la Société Industrielle du Nord de la France	Bull. Soc. Ind. Nord ...	Rue de l'Hôpital Militaire 116, Lille, France.
Bulletin de la Société Industrielle de Mulhouse	Bull. Soc. Ind. Mulhouse	Berger-Levrault et Cie., 5, Rue des Beaux-Arts, Paris. 60 fr. per annum.
Bulletin de la Société d'Encouragement pour l'Industrie Nationale	Bull. Soc. d'Encour....	Secrétariat, Rue de Rennes 44, Paris, France. 36 fr. per annum.
Canadian Chemical Journal	Canad. Chem. J. ...	Biggar Press, Ltd., 540-541, Confederation Life Building, Toronto. 20 cents.
Le Caoutchouc et la Gutta-Percha	Caoutchouc et Gutta-Percha	A. D. Cillard fils, 49, Rue des Vinaigriers, Paris, X. 30 fr. per annum.
Chamber of Commerce Journal	Ch. of Comm. J. ...	Oxford Court, Cannon Street, London, E.C. 1s.
Chemical and Metallurgical Engineering	Chem. and Met. Eng.	McGraw-Hill Co., 10th Avenue at 36th Street, New York. 25 cents.
Chemical News ... ..	Chem. News ... ..	E. J. Davey, 16, Newcastle Street, Farringdon Street, London, E.C. 4d.
Chemical Trade Journal ...	Chem. Trade J. ...	Davis Bros., Danes Inn House, 265, Strand, London, W.C. 6d.
Chemiker-Zeitung ... ..	Chem.-Zeit. ... ..	Dr. v. Vietinghoff-Scheel, Cöthen, Anhalt, Germany. M. 9 per quarter.
Chemisch Weekblad ...	Chem. Weekblad ...	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 10 fr. per annum.
Chemische Industrie ...	Chem. Ind. ... ..	Weidmann'sche Buchhandlung, Zimmerstrasse 94, Berlin, S.W., Germany. M. 20 per annum.
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Chemische Zeitschrift ...	Chem. Zeitschr. ...	E. Buntrock, Berlin, S.W. 68.
Chemisches Zentralblatt ...	Chem. Zentr. ... ..	R. Friedländer und Sohn, Karlstrasse 11, Berlin, N.W. 6, Germany. M. 40 per vol.
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Gazzetta Chimica Italiana ...	Gazz. Chim. Ital. ...	La Direzione della "Gazzetta Chimica," Via Panisperna, 89, Rome. Lire 4.
Gerber ... ..	Gerber ... ..	VI $\frac{1}{2}$ Gumpendorferstrasse 89, Wien, Austria. Kr. 17.50 per annum.
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International Sugar Journal	Int. Sugar J. ...	2, St. Dunstan's Hill, London, E.C. 1s. 4d.
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Journal of the Washington Academy of Sciences	J. Wash. Acad. Sci. ...	211, Church Street, Easton, Pa., U.S.A. 25 cents; 50 cents for July, Aug. and Sept. numbers.
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New Zealand Journal of Science and Technology	N.Z.J. Sci. and Technol	The Director, Dominion Museum, Wellington, N.Z. 1s. 6d.
Oesterreichische Zeitschrift für Berg- und Hüttenwesen	Oesterr. Z. Berg- u. Hüttenw.	Kohlmarkt 20, Wien I, Austria. Kr. 28 per annum.
Oil, Paint, and Drug Reporter	Oil, Paint, and Drug Rep.	100, William Street, New York City, U.S.A. \$5 per annum U.S.A., \$6 Canada, \$7 other countries.
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Pharmaceutisch Weekblad...	Pharm. Weekblad ...	D. B. Centen, O.Z. Voorburgwal 115, Amsterdam. 10 fr. per annum.
Pharmazentische-Zeitung ...	Pharm.-Zeit. ... ..	Link-Strasse 23-24, Berlin, W. 9. 35 Pf.
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Philippine Journal of Science	Philippine J. Sci. ...	Bureau of Science, Manila, Philippine Islands. 50 cents.
Philosophical Magazine and Journal of Science	Phil. Mag. ... ..	Taylor and Francis, Red Lion Court, Fleet Street, London, E.C. 2s. 6d.
Photographic Journal ...	Phot. J. ... ..	Harrison and Sons, 44-47, St. Martin's Lane, W.C. 2.
Proceedings of the American Society of Civil Engineers	Proc. Amer. Soc. Civ. Eng.	220, West 57th Street, New York City, U.S.A. \$8 per annum.
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Proceedings of the Australasian Institute of Mining Engineers	Proc. Austral. Inst. Min. Eng.	57-59, Swanston Street, Melbourne, Victoria, Australia.
Proceedings of British Foundrymen's Association	Proc. Brit. Foundrymen's Assoc.	165, Strand, W.C.
Proceedings of the Engineers' Society of Western Pennsylvania	Proc. Eng. Soc. W. Pa.	2511, 568, Union Arcade Building, Pittsburgh, Pa., U.S.A. 50 cents.
Proceedings of the Faraday Society	Proc. Faraday Soc. ...	The Secretary, 82, Victoria Street, London, S.W.
Proceedings of the Institution of Civil Engineers	Proc. Inst. Civ. Eng...	The Secretary, Great George Street, London, S.W.
Proceedings of the Institution of Mining and Metallurgy	Proc. Inst. Min. and Met.	1, Finsbury Circus, London, E.C. 21s. per annum.
Proceedings of Koninklijke Akademie van Wetenschappen te Amsterdam	Proc. K. Akad. Wetensch. Amsterdam.	J. Muller, Amsterdam.
Proceedings of the Royal Society	Roy. Soc. Proc. ...	Harrison and Sons, 45, St. Martin's Lane, London, W.C. Price varies.
Proceedings and Transactions of Nova Scotian Institute of Science	Proc. and Trans. Nova Scotian Inst. Sci.	Halifax, Nova Scotia. 50 cents.
Pulp and Paper Magazine...	Pulp and Paper Mag.	Industrial and Educational Press, Ltd., Garden City Press, St. Anne de Bellevue, Canada. 15 cents.
Recueil des Travaux Chimiques de Pays-Bas	Rec. Trav. Chim. Pays-Bas	A. W. Sijthoff, Leiden, Holland. 6 fl. per volume.
Revue Générale des Matières Colorantes	Rev. Gén. Mat. Col. ...	L. Lefevre, 4, rue de Stockholm, Gare St. Lazare, Paris, France. 3 fr.
Revue de Métallurgie ...	Rev. Mét. ... ..	H. Dunot and E. Pinat, 49, Quai des Grands Augustins, Paris. 4 fr. 50.
School of Mines Quarterly...	Sch. Mines Quart. ...	T. H. Harrington, Columbia University, New York. 50 cents.
Schweizerische Chemiker-Zeitung	Schweiz. Chem.-Zeit.	Meyer & Co., Oerlikon-Zürich, Switzerland. 20 fr. per annum.
Science Reports of Tohoku Imperial University	Sci. Reports, Tohoku Imp. Univ.	The Maruzen Co., Ltd., Sendai, Japan. Price varies.
Scientific American... ..	Scient. Amer. ... ..	233, Broadway, New York City, U.S.A. 10 cents.
Scientific Proceedings of the Royal Dublin Society	Scient. Proc. Roy. Dublin Soc.	Leinster House, Dublin. Price varies.
Silikat-Zeitschrift ... ..	Silikat-Zeits. ... ..	Verlag von Müller und Schmidt, Coburg, Germany. M. 12 per annum.
Sitzungsberichte der Königlich Preussischen Akademie der Wissenschaften	Sitzungsber. Kgl. Preuss Akad. Wiss.	Verlag der Königlichen Akademie der Wissenschaften, Berlin. M. 12 per annum.
Soil Science ... ..	Soil Sci. ... ..	Williams & Wilkins Co., 2419-2421, Greenmount Avenue, Baltimore, U.S.A.; Cambridge University Press, Fetter Lane, London, E.C. 50 cents.
South African Journal of Industries	S. African J. Ind. ...	Government Printer, Box 373, Pretoria. 6d.
Sprechsaal ... ..	Sprechsaal ... ..	Verlag von Müller und Schmidt, Coburg, Germany. M. 14 per annum.
Stahl und Eisen ... ..	Stahl u. Eisen ... ..	Breitestr. 27, Düsseldorf 74, Germany. M. 18 per half-year.
Teknisk Tidskrift ... ..	Tekn. Tidsk. ... ..	Jakobsg. 19, Stockholm, 16, Sweden.
Tonindustrie-Zeitung ... ..	Tonind.-Zeit. ... ..	Dreysestrasse 4, Berlin, N.W. 21, Germany. M. 20 per annum.
Transactions of the American Electrochemical Society	Trans. Amer. Electrochem. Soc.	Lehigh University, South Bethlehem, Pa., U.S.A. \$3 per vol.

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Transactions of the American Foundrymen's Association	Trans. Amer. Foundrymen's Assoe.	R. Moldenke, Watchung, N.J., U.S.A.
Transactions of the American Institute of Chemical Engineers	Tr. Amer. Inst. Chem. Eng.	D. Van Nostrand Co., New York, and F. and N. Spon, Ltd., London.
Transactions of the English Ceramic Society	Trans. Engl. Ceram. Soc.	County Pottery Laboratory, Stoke-on-Trent, Staffordshire. 30s. per annum.
Transactions of the Institute of Metals	Trans. Inst. Metals ...	Institute of Metals, 36, Victoria Street, Westminster, London, S.W.
Transactions of the Iron and Steel Institute	Trans. Iron and Steel Inst.	28, Victoria Street, London, S.W.
Transactions of the North of England Institute of Mining and Mechanical Engineers	Tr. N. Eng. Inst. Min. and Mech. Eng.	Newcastle-on-Tyne.
Transactions of the Royal Society of Canada	Trans. Roy. Soc. Canada	J. Hope & Son, Ottawa; B. Quaritch, London. Price 50 cents.
Tropenpflanzer ... ..	Tropenpflanzer ...	Pariser Platz, 7, Berlin, N.W., Germany. M. 15 per annum.
United States Bureau of Mines, Bulletins and Technical Papers	U.S. Bureau of Mines, Bull. & Tech. Papers	Bureau of Mines, Washington, D.C., U.S.A. Price varies.
United States Bureau of Standards, Bulletins and Technological Papers	U.S. Bureau of Standards, Bull. and Technol. Papers	Superintendent of Documents, Washington, D.C., U.S.A. Price varies.
United States Commerce Reports, Daily Consular and Trade Reports	U.S. Comm. Rept. ...	Superintendent of Documents, Washington, D.C., U.S.A. \$2.50 per annum.
United States Department of Agriculture Bulletins	Bull. U.S. Dept. Agric.	Department of Agriculture, Washington, D.C., U.S.A. Price varies.
Wasser u. Gas ... ..	Wasser u. Gas ...	G. Stalling, Oldenburg i. Gr., Germany.
West Indian Bulletin ...	West Ind. Bull. ...	Imperial Dept. of Agriculture, Barbados, W.I. 6d.
Wochenblatt für Papierfabrikation	Wochenbl. Papierfabr.	Günter-Staib in Bieberach a. d. Riss, Württemberg, Germany. 50 Pf.
Wochenschrift für Brauerei	Woch. Brau. ...	P. Parey, Hedemannstrasse 10, Berlin, S.W., Germany. M. 40 per annum.
Zeitschrift der analytischen Chemie	Z. anal. Chem. ...	C. W. Kreidel's Verlag, Wiesbaden, Germany. M. 2.
Zeitschrift für angewandte Chemie	Z. angew. Chem. ...	Verlag von Otto Spamer, Nürnberger Strasse 48, Leipzig, Germany. M. 35 per annum.
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Zeitschrift des Vereins der deutschen Zucker-Industrie	Z. Ver. deut. Zuckerind.	Vereins-Direktorium, Kleiststrasse 32, Berlin, W. 62, Germany. M. 45 per annum.
Zeitschrift für das gesammte Brauwesen	Z. ges. Brauw. ...	R. Oldenbourg, Glückstrasse 8, München, Germany. M. 20 per annum.
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Zeitschrift für öffentliche Chemie	Z. öffentl. Chem. ...	A. Kell's Buchhandlung, Marktstrasse 15, Plauen i. V., Germany. M. 1.
Zeitschrift für physikalische Chemie	Z. physik. Chem. ...	W. Engelmann, Mittelstrasse 2, Leipzig, Germany. M. 76 per annum.
Zeitschrift für physiologische Chemie	Z. physiol. Chem. ...	Verlag von K. J. Trübner, Strassburg, Germany. M. 18 per vol.
Zeitschrift für Spiritusindustrie	Z. Spiritusind. ...	P. Parey, Hedemannstrasse 10, Berlin, S.W., Germany. M. 30 per annum.
Zeitschrift für Untersuchung der Nahrungs- und Genussmittel	Z. Unters. Nahr. Genussm.	Julius Springer, Linkstrasse 23/24, Berlin, W. 9, Germany. M. 48 per annum.
Zeitschrift für Zuckerindustrie in Böhmen	Z. Zuckerind. Böhm....	Die Administration, Havlikovo Nam, Nr.32, Prag II., Bohemia. M. 17.20 per annum.





INCOME AND EXPENDITURE ACCOUNT FOR YEAR ENDING 31st DECEMBER, 1917.

[illegible]



## BALANCE SHEET, 31ST DECEMBER, 1917.

To Sundry Creditors.....	£ s. d.		£ s. d.
" Subscriptions received in advance .....	1858 2 8		2315 9 1
" Research Prize Fund.....	846 2 0		81 5 0
" Accumulated Fund.....	300 0 0		1082 17 5
Balance 31st Decemr. or 1916.....	22,128 0 11		
Add Excess of Income over Expenditure, 1917 .....	556 4 7		
	<u>23,084 5 6</u>		

Valuation : 31st Dec., 1917.	£ s. d.	£ s. d.
3021 12 1 at 67½		4163 2 1
830 0 0 "	63	870 0 0
604 1 2 "	70	1277 4 8 New Zealand 3% (1945)
816 11 10 "	99½	(1905) .....
330 0 0 "	65	850 13 10 War Stock 4½%
685 14 4 "	54	600 0 Gas Light & Coke Co. 3% Debentures .....
336 14 4 "	45½	1084 13 4 North British 3% Con- solidated .....
451 4 0 "	47	736 0 Midland Railway 2½%
1033 13 3 "	68½	do do .....
511 9 7 "	76	Guaranteed Prof.... }
612 10 0 "	61½	Great Eastern Railway 4% Irredeemable .....
112 0 0 "	56	673 0 South Eastern Railway 4½% Preference .....
1176 0 0 "	91	1000 0 Metropolitan Water Board 3½% "B" } Debentures .....
472 0 0 "	59	200 0 Nottingham Joint Station 3% .....
330 15 0 "	73½	1250 0 Great Western Rail- way 5% Guaranteed .....
463 12 3 "	64	800 0 Great Northern Rail- way 3% Debentures .....
459 19 0 "	55	450 0 Nottingham and Gran- tham Canal 4½% Con- solidated .....
1022 0 0 "	73	724 8 India 3½%
		834 9 India 3%
		1400 0 Dominion of Canada (Canadian Pacific) 3½% Land Grant Bonds (1938) .....
		709 0 Canada 4% (1940 - 1960) ...
		703 8 West Australian Govern- ment 4% .....
		700 0 War Loan 3½%
		1500 0 War Loan 6% (Boat) <u>22,698 18 5</u>
		16 666 10 10

Add—Purchased during year War Loan 5% .....	10 0 3
	<u>22,708 18 8</u>
	£ 46,684 10 2

Depreciation = 26·6 % on cost.

To the Members of the Society of Chemical In

20 to the Members of the Society of Chemical Ind-

We have audited the above Balance Sheet, dated 31st December, 1917, and have verified the Investments, the Society at 31st December, 1917.

28 Basinghall Street, London, E.C.,  
28th June, 1918

(Signed) FEASEY & CO.,  
Chartered Accountants.

In our opinion the Balance Sheet shows correctly the position of

# JOURNAL OF THE SOCIETY OF CHEMICAL INDUSTRY

Vol. XXXVII. No. 1

January 15th, 1918

## TRANSACTIONS

### Glasgow Section.

*Meeting held at Glasgow, on Friday, October 19th, 1917.*

MR. JAMES MACLEOD IN THE CHAIR.

#### RECOVERY OF POTASH FROM BLAST FURNACE DUST.

BY PROF. R. A. BERRY, F.I.C., AND D. N. MCARTHUR, B.Sc.

That the dust deposited from blast furnaces and kiln gases contained potash salts<sup>1</sup> is by no means a new discovery. In 1884 a patent was taken out by Hugh Barclay and Robert Simpson of the Harrington Iron Works, Cumberland, for the recovery of salts, especially potash salts, from coke-fed furnaces. Reference to similar patents occur in American literature.<sup>2</sup> At the Riverside Portland Cement Company's Works in California, a Cottrell electrical precipitation plant<sup>3</sup> was installed in 1911 for the purpose of precipitating the dust from the kiln gases and the dust was found to contain potassium salts, the raw material containing 0.2%  $K_2O$ . Owing to the low price of potash salts at that time, the dust was not considered to be of any value but it was allowed to accumulate. With the outbreak of war, however, the whole position changed and this accumulated dust then became a product of considerable value. In 1915 the same company, every twenty-four hours, was recovering, from five kilns, from 20 to 25 tons of dust containing 5 to 20% of water-soluble potash ( $K_2O$ ). De Beers<sup>4</sup> calculated that 80,000 tons of potash could be obtained annually from such sources in the United States. Wysor<sup>5</sup> found that the yellowish dust, which accumulates at the bottom of the stone chequer work in the stoves and gas fired boilers, contained 15% of water soluble potash ( $K_2O$ ). Since the war started, he has gone into the question of the potash content of the raw material and of its recovery from the furnace gases and has drawn up a balance-sheet to show the weight of potash charged into the furnace and the amount recovered and lost. He estimates the greatest loss to be in the primary and secondary washers, amounting to some 56% of the whole, while in the slag some 20% is lost. The amount recovered in the dust from the stoves and boilers is under 1% of the potash charged. In 1916 an experimental Cottrell plant was installed, being connected to the gas main leaving one of the dust catchers and he found that practically all the dust entering the plant could be successfully precipitated. This dust contained on an average about 10% of potash ( $K_2O$ ) and the colour varied from a light to a dark grey. The quantity of potash charged was 22.4 lb. per ton of pig iron produced and

deducting the amount of potash in the slag and dust catcher, namely, 6 lb., about 15 lb. per ton of pig iron appear to be left in the gases, which were then recoverable by the Cottrell process.

According to Burchard's<sup>6</sup> figures the potash content of the iron ores varies greatly, from 0.17% to 2.56%  $K_2O$  in some American ores, the average being 0.63%. Grasby<sup>7</sup>, however, gives the average as 1.32%, the lowest being 0.70%  $K_2O$  and the highest 2.29%.

The production of pig iron in the United States in 1916 amounted to over 39,000,000 tons<sup>8</sup>, so that if Wysor's figures are correct then the flue dust from the coke-fed furnaces in the States is a great addition to the potash supply.

In Scotland, however, the recovery of potash from blast-furnace gases is a rather different problem, as not only is the potash content of the iron ores used probably lower, but coal instead of coke is normally used. In the case of the coal-fed furnaces much tarry matter is produced. With the coke-fed furnaces, as in England, this is absent so that the gases are not washed but led from the dust catcher direct to the stoves and boilers, the dust being deposited in the dust catchers, tubes, stoves and at the base of the chimney stack.

Cranfield<sup>9</sup> states that from one furnace per week 20 tons of a coarse black dust are deposited in the dust catcher, 5 tons of a reddish dust in the stoves and boilers, and 1 ton of a cream-coloured dust at the base of the chimney stacks. The potash ( $K_2O$ ) content of each is 2.5%, 7.0%, and 10% respectively and a total annual yield from the furnaces in Great Britain of 15,000 tons of potash ( $K_2O$ ) is estimated. His estimate is, however, too high as he assumes that the Scottish furnaces are producing dust at the same rate as the English ones. Coke fed furnaces are driven harder with a blast of about 12 lb. compared with 5 lb. in the Scottish practice and consequently more dust is produced in the English works.

As the amount of dust produced in the Scottish practice is relatively very small, the spent liquor, which according to Wysor is the main avenue of loss, seems more deserving of attention. So far as we are aware there are no published analyses of the spent liquors; while the analytical data showing the potash content of the raw materials are almost equally unsatisfactory. The potassium is present mostly in combination with silica and its amount will vary largely. Probably the silicates of potassium, at the high temperature reached at the base of the furnace, interact with other components of the charge liberating the potassium as oxide which, in turn, reacts with the sulphate and chlorides present to form potassium salts. Potassium cyanide is found at certain regions in the furnace, but is again decomposed, the nitrogen being given off as ammonia and free nitrogen, and the potassium probably as oxide or carbonate. The high temperature vaporises the potassium compounds and the



vapours, as they pass into the cooler parts of the furnace, condense in the form of fine dust particles which are carried over along with the dust from

potash ( $K_2O$ ) was first determined in each sample and subsequently the total potash in several samples.

TABLE I.  
FLUE DUSTS.

Firm No.	Water-sol. potash.	Colour.	Action towards litmus paper.	Yield per annum.	Value at 15s. per unit of $K_2O$ .	Value at 4s. per unit of $K_2O$ .
	%			Tons.	£	£
1 .....	11.06	Grey	Alkaline	21	174.2	46.5
2 .....	19.49	Yellowish brown	Neutral	10	146.2	40.0
3 .....	6.59	Yellowish Brown	Neutral	5	25.2	6.6
4 .....	4.59	Greyish white	Neutral	6	20.7	5.5
7 .....	4.07	Reddish white	Neutral	2	6.1	1.6
8 .....	8.58	Light brown	Neutral	2	12.9	3.2
14 .....	8.05	Purple	Alkaline	20	120.8	32.2
16 .....	8.46	Cream	Neutral	5	31.7	8.5
17* .....	3.13	Grey	Alkaline	300	704.3	187.8

\*No. 17. Coke-fed furnace.

Average % water-sol. potash in dust from 8 coal-fed furnaces .....	8.86
Total yearly yield of fine dust from 8 coal-fed furnaces .....	71 tons
Total yearly value of fine dust at 15s. per unit $K_2O$ .....	£550 approx.
Total yearly value of fine dust at 4s. per unit $K_2O$ .....	£144 approx.

the fuel and ores. The heavier particles are deposited in the dust catcher and form a coarse black powder. Inside the main tube a thick coating of carbonaceous matter is formed which is periodically cleaned out and forms, along with the dust from the dust catcher, the "tube cleanings."

The furnace gases contain a large proportion of tarry matter, from the coal, which condenses and encrusts much of the mineral matter. Most of the heavy tarry matter separates in the condensers and in settling carries down with it a considerable quantity of the fine dust. The rest of the tarry matter, the ammonia, and most of the remaining fine dust are then caught by the water during the cleaning of the gas in the scrubbers. The gases on leaving the scrubbers should only contain a very little mineral matter and this is caught in the stove and boilers forming the stove and boiler dust. The spent liquor, from which the bulk of tarry matter and the ammonia have been extracted, is at present allowed to escape except at Messrs. Baird and Co.'s Lugar and Muirkirk plants and at the Dalmeilington Iron Co.'s Works, Dunaskin, where for several years the potassium salts have been recovered by evaporating the spent liquors. Samples of the spent liquors and of the dusts were received for analysis towards the end of May, 1917, from the different plants in Scotland and their potash content was determined with a view to forming an estimate of the quantity of potash which might possibly be recovered.

Consideration may be first given to the dusts:

**Flue dust.**—Nine samples were received. The dusts were of a very fine powdery nature but

The water soluble potash ( $K_2O$ ) varied from 4.07% to 19.49%, the average being 8.86%. It was not possible to correlate colour and potash content.

A qualitative examination of the dust usually showed in addition to potassium, the presence of iron, aluminium, calcium, magnesium, sodium, and silica, while in many lead and zinc were also found. Chlorides, sulphates, and carbonates were the predominating acid radicles.

The highest yield of dust per annum at any one of the plants was 21 tons. The one plant using coke fuel gave a yield of 300 tons per annum—due to a greater blast, and to the absence of tarry matter, but the potash content was low, namely, 3.13%. Excluding the latter the total production per annum of flue dust in Scotland from the coal-fed furnaces does not reach 100 tons, and taking the present value of the potash at 15s. per unit, the estimated total value is £550, whilst the estimated value of dust from the coke-fed furnaces amounts to £700 per annum as against £150 and £190 respectively if valued at the pre-war rate of 4s. per unit of potash ( $K_2O$ ).

**Stove dust.**—Only three samples were received. The potash content varied between 6% and 8% water-soluble  $K_2O$ . With the exception of the plant using coke fuel, the yields are negligible. In this case the yield was 150 tons per annum, the total potash content being 13.6%  $K_2O$  and the water-soluble potash 7.6%. These values are given in Table II.

The total potash was determined in five samples of dust and the percentage of the total potash soluble in water ranged from 40 to 89.

TABLE II.  
STOVE DUSTS.

Firm No.	Water-sol. potash.	Colour.	Yield per annum.	Value at 15s. per unit of $K_2O$ .	Value at 4s. per unit of $K_2O$ .
	%		Tons.	£	£
3 .....	7.87	Reddish brown	1	5.9	1.5
14 .....	6.86	Grey	6	30.9	8.2
17* .....	7.65	White	150	860.6	229.5

\*No. 17. Coke-fed furnace.

Total yearly yield of dust from 2 coal-fed furnaces .....	7 tons
Total yearly value of dust from 2 coal-fed furnaces at 15s. per unit $K_2O$ ..	£37 approx.
Total yearly value of dust from 2 coal-fed furnaces at 4s. per unit $K_2O$ ..	£10 approx.

microscopic examination revealed a large number of minute crystals, too small for identification. The colour varied from cream to purple and some dusts were distinctly alkaline while others were neutral to litmus paper. The water soluble

**Tube cleanings.**—Samples from 15 works were received. They were black and contained a considerable amount of carbonaceous matter. The percentage of ash in the samples analysed varied from 53% to 74%. As the ash contained

TABLE III.  
COMPARISON OF WATER-SOLUBLE AND TOTAL POTASH IN DUST.

Firm No.	Dust.	Total water-sol. salts.	Total potash (K <sub>2</sub> O) sol. in HCl.	Water-soluble potash, K <sub>2</sub> O.	Total potash water-sol.
		%	%	%	%
2	Flue	56.9	21.96	19.49	88.7
14	Flue	25.8	11.13	8.06	72.4
15	Burnt tube cleanings	11.5	2.60	1.04	40.0
17	Flue	11.3	6.67	3.13	46.9
17	Stove	—	13.58	7.65	59.3

In the dust average % total potash (K<sub>2</sub>O) water-soluble is 69.3.

only a very small percentage of water soluble potash, the highest being 2.7% and the lowest under 1%, only a few samples were analysed. The value of these dusts is however high in comparison with the other dusts on account of the great quantities of cleanings annually produced. According to estimates supplied, the total quantity of tube cleanings produced per annum from the Scottish furnaces amounts to nearly 15,000 tons.

in blast, the structural differences in individual plants, the efficiency of the washing and the potash content of the raw materials. Considerable variation must be expected in the raw materials according to the nature of the coal, the iron ore, and the limestone used. As stated above, published data showing the potassium content of the raw materials are not available.

The potassium salts could be extracted with

TABLE IV.  
TUBE CLEANINGS.

Firm No.	Ash.	Water-sol. potash K <sub>2</sub> O in ash.	Yearly yield ash (app.).	Yearly yield of tube cleanings.	Value at 15s. per unit of K <sub>2</sub> O.	Value at 4s. per unit of K <sub>2</sub> O.
	%		Tons.	Tons.		
3	60.5	1.1	151	250	124.6	33.0
7	74.4	2.7	350	470	708.75	189.0
9	59.1	0.3	236	400	53.1	14.2
12	53.5	2.5	1605	3000	3009.4	802.5
17*	65.8	1.0	312	520	275.5	68.4

\*No. 17. Coke-fed furnace.

Average % water-sol. potash in ash from 4 coal-fed furnaces..... 1.6  
Total yearly yield of cleanings from 4 coal-fed furnaces ..... 4120 tons  
Total yearly value of ash from 4 coal-fed furnaces at 15s. per unit K<sub>2</sub>O .. £3900 approx.  
Total yearly value of ash from 4 coal-fed furnaces at 4s. per unit K<sub>2</sub>O .. £1040 approx.

*Spent liquor.*—Fifteen works supplied samples. Two samples were taken from each plant at an interval of one week. The method of sampling consisted in taking a ladle-full—about  $\frac{1}{2}$  gallon—every hour during the 24 hours and, from the composite sample, a further one gallon was drawn for analysis. The specific gravity, total solids, ash and water-soluble potash in the ash were determined in each sample.

Owing to the presence of the tarry matter evaporation was greatly retarded. The Cottrell process has been employed for precipitating coal tar products<sup>10, 11</sup>. If the temperature was kept above the vaporising point of the light oils, etc., the heavy tarry matter might be precipitated out with all the dust and thus produce a partial cleansing of the gas. This pitch might then be burnt.

The potassium was present mainly as sulphate and chloride except in one or two cases where a fair percentage of carbonate was found.

The ash was usually white or grey. The duplicate analyses varied considerably in many cases but on the whole the agreement was considered satisfactory. The highest percentage of K<sub>2</sub>O found was 55.7% from firm No. 6 and the lowest 20.6% from No. 8, the average being 33.5%. The potash content of the ash was high, but the yield of ash was disappointing. The highest yield estimated for any one plant was 318 tons at No. 12 and the lowest 17 tons at No. 8, while the total yield of ash was 1696 tons.

The factors which would account for the variation in yield comprise the number of furnaces

water or the ash, without further treatment, might be used as a manure. The value of the ash is given in Table V. No. 1 plant yields 257 tons containing 31.4% K<sub>2</sub>O soluble in water and at the present market price of potash, namely, 15s. per unit, this quantity is valued at £6052 or at £1614 on the pre-war basis of 4s. per unit K<sub>2</sub>O. No. 12 plant yields 318 tons containing 22.3% K<sub>2</sub>O soluble in water and valued at 15s. per unit of potash represents £5286 or £1418 on a 4s. per unit basis. The total value of £1696 tons containing an average of 33.5% water-soluble potash (K<sub>2</sub>O) is £44,312 on a 15s. per unit basis and £11,500 on a 4s. per unit basis. These data refer to 15 plants using coal fuel and having a total of 72 furnaces in blast. The question of the advisability of evaporating the spent liquor must be decided by each firm for itself. When the yield of ash reaches 100 tons and its value between £2,500 and £3,000 and when spare heat and exhaust steam are available, the matter is worthy of serious consideration. It is, however, necessary to remember that the prices per unit of K<sub>2</sub>O in pre-war days was about 4s. and it may again fall to that figure after the war. The erection of evaporating plants at most of the works would be necessary and, at the present time, the cost of so doing would be high. Preliminary concentration of the liquors, where exhaust steam is available, is recommended and, owing to the scum of tarry matter which forms on the surface, this must be facilitated by mechanical stirring and splashing with furnaces of the Porion type or by other means. According to an



TABLE V.  
SPENT LIQUOR.

Firm No.	Sp. gr. average.	One gallon contains :			Ash yield in tons : yearly average.	Water soluble potash : average % in ash.	Yearly value of potash at 15s. per unit.	Yearly value of potash at 4s. per unit.
		Dry matter.	Ash.	Potash K <sub>2</sub> O.				
		grms.	grms.	grms.			£	£
1	1.019	9.443	4.721	1.525	257	31.4	6052.4	1614.0
2	1.039	120.58	84.525	30.753	273	36.4	7452.9	1987.4
3	1.320	—	434.952	156.583	125	36.0	3375.0	900.0
4	1.035	9.824	6.773	2.452	58	36.2	1574.7	419.9
5	1.020	7.640	4.843	1.822	148	43.1	4719.5	1275.8
6	1.025	9.198	4.431	2.429	66	55.7	2757.2	735.2
7	1.017	6.065	3.990	0.826	50	20.6	772.5	206.0
8	1.027	4.558	1.587	0.321	17	20.6	262.7	70.0
9	1.013	8.622	5.257	2.029	95	38.2	2837.0	725.8
10	1.018	7.318	2.065	0.573	45	27.2	918.0	244.8
11	1.019	5.663	3.787	1.329	36	33.6	907.2	241.9
12	1.019	18.296	12.712	2.833	318	22.3	5286.8	1418.3
13	1.023	5.401	2.260	0.667	41	28.8	885.6	236.2
14	1.035	9.797	6.405	3.036	69	47.4	3833.0	654.1
15	1.026	10.460	5.448	2.179	98	40.0	2940.0	784.0

Average % water-soluble potash ..... 33.5  
 Total yearly yield of ash from 15 coal-fed furnaces ..... 1696 tons  
 Total yearly value of ash at 15s. per unit K<sub>2</sub>O ..... £44,575 approx.  
 Total yearly value of ash at 4s. per unit K<sub>2</sub>O ..... £11,513 approx.

estimate supplied to us by Messrs. Gillespie and Son, Consulting Engineers, Glasgow, an evaporating plant of this kind to deal with 20,000 gallons daily, would cost £4000 and to deal with 100,000 gallons daily would cost £14,000. If spare heat is available then the capital outlay may be wiped off in a few years. Even if the value of the potash salts recovered is sufficient only to pay the running expenses of the plant, then evaporation of the spent liquor is perhaps the best way for its disposal.

**Sources of loss.**—Considering that such relatively small quantities of potash are recoverable in the dust and spent liquor, the question arises as to what is the weight of potassium charged into the furnaces and what are the sources of loss. The principal losses would be in the pitch and slag.

**"Pitch"** from blast furnaces contains much more mineral matter than that from coke and coal gas plants. According to the conditions of deposition and recovery of the tarry matter, the percentage of the amount of ash in the pitch will vary in different works. Figures which have been supplied to us, indicate that the ash content varies from 10% to 25%, the average being about 15%. Devices such as filtration, centrifuging and so forth are in use to remove the ash. It is also possible that the percentage of potash in the ash from any one plant may vary. An analysis of the pitch from No. 1 plant shows it to contain 17% of ash and the ash 8.8% of water-soluble potash. The yield of pitch per ton of coal burnt varies from 98 lb. to 140 lb. at different plants, the average being about 112 lb. The pitch left, after the recovery of the light oils, etc., from the tar, also varies at the different works, as, for example, at No. 1 plant it was 70% of the tar while Gray and Mellanby<sup>12</sup> put the figure at 65%.

When the ash content is 15% and 8% of this is water-soluble potash it would, with pitch at 12s. per ton, pay to burn the latter as fuel and collect the ash. On the basis of these figures and ignoring the insoluble potash 1 ton of ash could be obtained from 6.6 tons of pitch and, with the market price of potash at 15s. per unit (K<sub>2</sub>O), would be worth £6 without taking into account the fuel value, while the initial cost of the pitch at 12s. per ton would be approximately £4. At the pre-war rate of 4s. per unit the ash would only be worth £1 12s. The burning of the pitch might produce difficulties but these doubtless could be overcome.

The coal consumed in blast furnaces in Scotland

in 1915 amounted to 1,478,924 tons, so that on an average production of 112 lb. pitch per ton of coal, then the total yield of pitch would be about 74,000 tons. If all the pitch were burned, 11,092 tons of ash containing 8% K<sub>2</sub>O would be obtained and at present prices would be worth £41,596 and at pre-war prices £11,100.

**"Slag."** The quantity of slag produced per ton of pig iron varies according to the purity of the iron ore and limestone and the weight of slag also varies from 9 cwt. to 23 cwt. per ton of pig iron produced at the different plants. The slag contains potassium, 0.24%. K<sub>2</sub>O being found in slag from Firm No. 1, but at present it does not seem feasible to recover this.

**Balance Sheet.**—The amounts of potash in the raw materials—coal, coke, iron ore, and limestone, and in the slag and other by-products from No. 1 plant have been determined and are as follows: Spent coal 0.040% K<sub>2</sub>O, coke 0.121%, iron ore 0.128%, limestone 0.136% K<sub>2</sub>O. A balance-sheet was drawn up with the following result:—

Total potash charged into furnace—7.6 lb. per ton of pig iron.

Potash recovered—	
In spent liquor .....	1.4 lb. per ton of pig iron.
In flue and stove dust .....	0.04 " " "
In pitch .....	1.7 " " "
In tube cleanings .....	0.2 " " "
In slag .....	2.7 " " "
Total .....	6.04 " " "

Unaccounted ..... 1.6 " " "

The deficiency is due to other sources of loss in the furnace and in the running off of the slag, pig iron, etc.

The iron ore was the principal source of the potash, while the coal did not supply much more than the limestone. In contradistinction to these results Sir Lothian Bell states in his work on "Chemical Phenomena of Iron Smelting" that the potash is derived from the coal.<sup>13</sup>

The balance-sheet is in great contrast to that obtained by Wyssor. Too much stress, however, cannot be laid on the fact, that our figures are only applicable to the raw materials and to the plant in question.

Although it has not been possible in the time at our disposal to obtain similar figures from other works, these figures may be extended generally to those plants using Welsh limestone and a mixture of Spanish ores. Thus taking the production of pig iron in Scotland for 1916 at 1,144,756 tons and the potash charged into the furnaces at 7.6 lb. per ton of pig iron, the total weight of

potassium would amount to 3884 tons, more than one half of which is recoverable.

In Scotland 12 companies having 17 works, a total of 102 blast furnaces, produce potash-bearing by-products.

The total quantity of potash recoverable from these by-products is as follows:—

	Tons K <sub>2</sub> O.
Spent liquor 1694 tons containing 35.5% wat. sol. potash	567.4
Flue and stove dust about 100 tons do. 8% do.	8.0
Pitch ash about 11,092 tons do. 8% do.	885.8
Tube cleaning dust about 8,250 tons do. 2.5% do.	206.0
Total production	1667.2

The figure is, relatively, a very small one, but it represents water-soluble potash. It is hardly possible with the data we have to estimate approximately what the total amount of insoluble potash would be. The insoluble potash might be rendered soluble by a process similar to that of Rhodin<sup>14</sup>. The heat from burning the pitch and the tube cleanings could be utilised for evaporating the spent liquor.

As the total requirements of potash for the British Isles is estimated to be 32,000 tons per annum, there is good reason to believe that if the different sources of potassium in this country (see Bibliography) were developed to the fullest extent, the country's requirements in potash salts would be more than met.

In any scheme of development of this nature, after war conditions have passed away, it must always be borne in mind that if the Alsatian deposits come into the occupation of their rightful owners, as they must, there will be available an almost unlimited supply irrespective of the deposits in Germany.<sup>15</sup>

In conclusion we wish to thank the different companies for their very sympathetic co-operation in the work and in particular Mr. A. K. McCosh for his help and advice at every stage of the investigation.

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## THE DISTILLATION OF WOOD.

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The distillation or carbonisation of wood is a subject which is becoming increasingly prominent amongst the world's industrial activities. We have heard a great deal in the past three years about so-called "key industries," and their importance to the industrial and commercial development of the country. The wood distillation industry may, I think, be classed among the minor key industries, particularly in view of the ever-increasing range of uses of the prime and derived products.

The art in its elementary form dates back to the prehistoric ages, for we read of charcoal burners and their activities in the historical tales of every century. As to when and where the first commercial step forward from the mere carbonising of wood for the production of charcoal to the condensation of the gases was taken, who can say?



In Britain among the earliest factories was that of Messrs. Shirley Aldred and Co., established in 1796, long before the advent of railways. Their works was established near Rotherham for the production of charcoal to supply the metal trades at Sheffield, and to supply the chemical trades at Manchester with acetates and acetic acid. The chemicals were carted over the roads fifty miles to their destination. It is interesting to note that of some seven plants erected subsequently in that district there are only two remaining, of which the above is one, the lack of wood and the loss of the demand for charcoal due to discontinuing manufacture of charcoal iron being among the principal reasons for their abolition. There were also several works established in Scotland at an early date, some of which are still in operation, and operating to-day on lines very similar to those originally used. It is a remarkable fact, although not so remarkable when the fact that Scotsmen were the entrepreneurs is recalled, that from one of these Scottish works sprang the whole immense industry of wood distillation in the United States of America. About 1849 Messrs. Turnbull of Glasgow built and operated the first complete American wood distillation works at Millburn, N.Y. Messrs. Turnbull sent over their own equipment and workmen, and the works was consequently known, and is known to-day, as the "Scotch Works." The original workmen gradually dispersed into other new factories in the United States, so that from this one factory the industry grew very gradually to its present proportions.

Continental development may be said to be similar to British, but on a larger scale. The United States produces about 50% of the world's requirements, Canada and Europe an equal amount, of which about 80% is produced in the Central Empires. The amount of wood distilled throughout the world for by-products is approximately three million tons annually, or about two million cords of 128 cubic feet each.

The ramifications of the industry to-day are evident in an almost inconceivable number of activities. Manufacturing works of many kinds are using the immediate products or derived products from the distillation of wood. Originally, as has been noted, charcoal was the only substance obtained. This was followed at an early date by crude acetic acid and the various acetates. Later a demand for wood spirit or alcohol and tar grew up and these materials were consequently collected. At present the four prime products obtained at the majority of works are as follows:—

1. Charcoal.
2. Acetate of lime (grey).
3. Wood alcohol.
4. Tar.

More recently, owing to the increasing costs of raw material (wood), fuel, and labour, and to increasing competition and consequent lowering of prices, the larger works first, followed eventually by the smaller, were forced to methods of increasing income by increasing their efficiency and making use of what were formerly considered waste products. This gradually resulted in the saving and production of light oil, wood oils, first runnings from alcohol distillation known as methyl acetone or alcohol-acetone, sifted charcoal, fertiliser "filler," etc.

Following on this came the manufacture of derived products from the prime products, such as acetic acid, esters, and acetone from acetates, formaldehyde from methyl alcohol, pure drugs such as creosote, guaiacol, etc., from the tar, and from these products a whole series of organic chemicals, dyes, and drugs required in the arts and industries to-day. These latter developments

are being proceeded with owing largely to the very successful beginning made in combining the production of refined acetic acid with the crude plants. As by far the largest portion of the acetate made is used in normal times for producing acetic acid, naturally the greatest development came in this line. Acetic acid plants are in operation in connection with wood distillation plants producing continuously a redistilled pure glacial acetic acid and showing yields of from 91—95% of the theoretical.

In connection with the principal products of the industry used in this country it may be of interest to note the amounts imported and used here in the past few years (average per annum 1913, 1914, and 1915), calculated from figures given in Bull. Imp. Inst., Vol. 14, No. 4:—

Acetate of lime .....	4709 tons.
Acetic acid .....	4257 tons.
Acetone .....	3430 tons.
Wood alcohol .....	589,148 galls.
Wood tar .....	12,148 tons.

Of this about 20% is imported from Canada. The amount of acetate of lime imported is sufficient only for the production of half the acetic acid import, supposing none were used for other purposes, thus leaving an amount to be made up necessary to produce acetone and other products imported equal to over 20,000 tons.

To produce the wood alcohol imported would require from 60,000 to 80,000 cords of wood per annum, which would, in addition, produce roughly 8000 tons of acetate. The wood available in Great Britain under present conditions of forestry and lumbering would not support such an increase in the industry, but the remedy lies in Colonial development, as will be mentioned later.

In discussing the distillation of wood, it must be pointed out that the industry itself is broadly divided into two major divisions, both of which are subdivided into several minor subdivisions, each having its distinct type of plant and methods of operation. The major divisions are known as soft wood or coniferous wood distillation, and hardwood or deciduous tree distillation.

Softwood distillation deals with woods containing resinous substances and oils in addition to the cellulose substance, but only a very small proportion of ligno-cellulose bodies. Distillation of such wood produces products of an entirely different nature from those obtained in treating hardwood, which is primarily composed of cellulose and lignous matter, and contains no resins or oils.

Softwoods, such as pine, fir, spruce, etc., produce large quantities of crude oils and tar, but very little acid and alcohol, whereas hardwoods, such as oak, birch, beech, maple, elm, etc., produce very small quantities of oils and tar, but large amounts of acid and alcohol.

The softwood plants are subdivided into the following types, which derive their names from the method of treatment:—

1. Destructive process, in which the wood is heated as in hardwood distillation, the oils being driven off, and the resins decomposed.

2. Solvent extraction process, in which the wood is treated with either a volatile solvent or molten resin which is recovered and re-used.

3. Alkali extraction, in which the wood is treated as for the manufacture of paper pulp, the volatile oils being recovered by condensation.

4. Steam distillation process, in which the wood is treated by live steam under pressure, the oils being condensed with the steam.

Hardwood distillation, on the other hand, is made up of much simpler subdivisions, the process being almost entirely one of destructive distillation, but carried out in the following types of plants:—

1. Retort plants, in which the wood is treated in small retorts holding from 1 to 5 tons.

2. Oven, or large retort plants, in which the wood is handled in trucks, each oven treating from 10—15 tons per charge.

3. Kiln plants, being of the beehive type, holding 30—75 cords at a time.

4. Mechanical plants, dealing with chippings and shavings only, and operating continuously.

The detailed description of the processes and types of plants used in the softwood industry must be omitted from this paper owing to the limits of time and space, only a few important facts and comparisons being admissible, since the principal activity and interest in this country is in hardwoods.

Referring, therefore, to the types of hardwood plants, we may consider, for purposes of description, the small and large retort plants as one type. These plants make use of cord wood, slabs and edgings, blocks or small wood, tops, branches, etc., ranging in thickness from 1 in. or less to 6 ins. and 8 ins., but preferably about 4 ins. diameter. They are the most usual type of installation where timber is grown or in connection with large saw-mills—in other words, where wood is fairly cheap and of uniform quality. They are installed where the industry is primarily one for the production of chemicals, and may vary in size from 5 to 150 tons of wood per day.

The kiln type of plant is gradually being eliminated, as the yields of products are only about half those obtained from retorts. Their greatest use has been in connection with the production of immense quantities of charcoal for use in iron smelting, the recovery of the by-products being a secondary consideration. They use cord wood, slabs, etc., and the distillation is effected by the partial combustion of some of the wood itself, so that in consequence, part of the gases is decomposed. They are an uneconomical and inefficient type and are very unwieldy in operation. Consequently they are rapidly being replaced by oven or large retort plants.

The mechanical retort plants which use chips and shavings are naturally few in number, due to the fact that they deal with only a very special class of wood, to the various mechanical difficulties encountered in the mechanical operation of moving parts at such high temperatures, and also due to the difficulty in controlling temperatures and obtaining suitable yields of certain of the products from wood in such finely-divided condition where a large surface is exposed to the heat.

Before proceeding to a detailed consideration of the hardwood distillation plants, it may be of interest to interpolate some data and comparisons in connection with softwood distillation by the distillation process.

The woods sufficiently rich in resin to permit of profitable distillation by the distillation process are pitch pine, or long leaf yellow pine, Norway pine and fir. Small retorts are usually used, sometimes with oil jackets, as it is very important that the temperature be under accurate control. The wood is loaded in cordwood sizes and heated gently. The temperature is kept below 200° C., until all the turpentine and low boiling oils are driven over and condensed so that no charring of the wood takes place. If the temperature be allowed to rise above this point the spirit is contaminated by decomposition products of resin, etc., from which it is extremely difficult to free it. After the light oils are removed the temperature is raised to the usual degree for carbonisation, and the acid liquor and heavy oils are collected in a separate receiver. The time required for these woods is from 24—30 hours.

The immediate crude products are charcoal residue, non-condensable gas, tar, oils, and acid liquor. The tar is either shipped as such—Stock-

holm tar of commerce—or may be redistilled by fire or superheated steam, yielding light oils, creosote oils, pitch, and acid water. The crude oils are fractionated with steam several times and yield:—

Rosin spirits distilling between .....	80° and 150° C.
Wood turpentine .....	150°—180° C.
Pine oils .....	175°—250° C.
Rosin oil .....	250°—400° C.

The acid liquor is neutralised with lime, distilled to remove wood spirit, and evaporated to dryness to produce acetate of lime.

The yields of products in softwood destructive distillation plants vary between extremely wide limits, due almost entirely to the "richness" or "leanness" in resin of the wood used. The species differ so greatly one from another in the amount of resin and oils contained that no definite statement can be made regarding yields from softwoods as can be done with a greater degree of accuracy in connection with hardwoods. The subjoined results of tests on two charges of four cords each illustrate the great differences observed. One charge was made with a very rich pitch pine wood, and the other with a lean batch of pitch pine. The products obtained from a 4-cord charge of hardwood are appended for comparison.

TABLE I.

Products.	4 cords rich wood, wt. 16,600 lb.	4 cords lean wood, wt. 16,000 lb.	4 cords hard wood, wt. 14,900 lb.
Charcoal .....	4284 lb.	4400 lb.	3600 lb.
Refined turpentine ..	41.5 galls.	20.75 galls.	—
Pine oil .....	11.6 "	4.2 "	—
Rosin spirit .....	21.5 "	12.0 "	—
Rosin oil .....	141.0 "	64.75 "	—
Creosote oil .....	46.5 "	21 "	20 galls.
Acetate of lime 80% ..	300 lb.	350 lb.	852 lb.
Wood alcohol (spirit) ..	6 galls.	9.6 galls.	37.5 galls.
Pitch .....	1100 lb.	625 lb.	720 lb.

The refining and separating of the crude oils calls for apparatus of a particular type, since these oils are composed of a heterogeneous mixture of rosin spirits, turpentine, pine oils, and rosin oils with boiling points respectively varying from 80° to 400° C.

Innumerable distillations in ordinary pot type stills are required to obtain any degree of separation, so that column stills of a high degree of rectifying efficiency are necessary. Fortunately the lighter oils such as rosin spirit and turpentine are easily distilled in live steam, while the pine oil and rosin oil are more difficult to carry over in this medium. Even with columnar stills having the usual single column, however, it requires several distillations and the redistillation of various fractions to obtain a perfect separation which would produce a pure refined turpentine free from the other products. Using single column stills, the first operation removes the rosin spirit, and then a small quantity of intermediate product containing some turps. This is added to the first crude oil distilled. The residue from the first run is re-run and a refined turps obtained, also a second intermediate product, which latter is re-run along with the following charge of second-run oils.

The residue from the second run is nearly all pine oil and rosin oil, which are then re-run alone and the pine oil collected, the residue being the rosin oil, which may be fire-distilled to remove carbonaceous matter.

It will be seen that the running of a pure turpentine and a pure pine oil was rather a tedious process, requiring careful operation and considerable quantities of fuel. Within recent years the demand for pine oils, rosin oils, creosote, etc., has greatly increased due to the sudden demand for these oils for mineral separation from ores by the



flotation process. It became necessary to deal with large quantities of oils, so that the single column method was considered too cumbersome and expensive.

Our present practice is to combine two, three, or four stills in one, and operate them continuously, fractionating the several products in successive portions of the same still. It was determined experimentally by Veitch and Donk (U.S. Dept. Agric. Bureau of Chem., Bulletin 144) that refined turpentine was being distilled when the proportion of oil in the mixture of oil and water condensed was 55%, and similarly pine oil was produced when the proportion of oil to water in distillate was less than 35%. Working from these figures a scheme of distillation and rectification was drawn up. Each column has a known number of plates and sufficient rectifying surface to produce the mixture of oil and water necessary for the particular product to be condensed. In this way the various intermediate products remain continually in the system, rising and falling over a few plates, but never reaching the outlet, as they are continually being enriched in one component through the introduction of fresh liquor, and alternately being impoverished by the withdrawal of another component to the higher rectifier.

While the mechanical process, as has been stated, is confined to a comparatively small proportion of the total wood used, it is interesting to compare and discuss the manner in which it differs from the cordwood or retort type of plant. It will be readily realised that the main difference lies in the retort itself. The products obtained are similar in kind to those obtained from cordwood retort plants, differing only in quantities, hence the refining equipment will be described in connection with the cordwood retort process.

The continuous or mechanical retorts now in operation are of two distinct types—the horizontal, which seems to be the most used, and the vertical. The horizontal mechanical retort which is most in use consists simply of an oblong metal box, divided horizontally across the middle by a plate; chain drums at each end, operated by gearing, serve to carry endless chains with a series of scrapers over the horizontal plate the entire length of the retort, returning along the bottom plate to the point of starting. The chips are fed into the retort at the same end as discharged, hence they travel twice the length before being completely carbonised and ready for removal. The rate of travel and supply of wood are regulated so that carbonisation is complete in the cycle. The gases are usually removed at the top of the retort through pipes of ample area and pass to condensers in the usual manner.

Another horizontal mechanical retort makes use of the spiral conveyor, the chips, etc., being fed in at one end and charcoal discharged at the other.

Another type in successful operation is composed of a large chamber over the floor of which travels an endless chain guided by rollers and carrying vertical scrapers. The chain passes back and forward across the floor of the chambers several times, turning the chips with the scrapers until the carbonisation is complete.

The only successful vertical mechanical retort within the author's knowledge is Ross and Corner's patent type; this consists of a vertical metal cylinder, which slopes off in a conical shoot at the bottom. Inside the cylinder are plates placed horizontally, one above the other, and spaced equally. The plates are made with a slot extending from the centre to the circumference, and also with a circular hole cut out of the centre. A shaft passes down the centre of the cylinder, and arms attached to the shaft extend over the plates. Each arm is set to clear the plate at a greater distance than the others, and on rotating the shaft these arms cause the chips to travel circum-

ferentially round the plate until the slot is reached, when the wood falls to the next lower plate, and so on. The length of travel and speed are maintained at the correct rate to carbonise the wood thoroughly. The charcoal is collected in air-tight receptacles at the shoot. The gases pass up the annular central passage around the shaft (the coolest portion inside the retort) and out at the top of the condenser.

Heat is applied to all these different retorts by means of fuel burned in a brick-work furnace setting, the hot furnace gases passing round the retorts by means of various flue arrangements, each individual installation seemingly having been erected according to a different idea of heating. This is rather unfortunate in its results, as the author is aware from commercial operating tests and laboratory experiments that temperature control and the resultant regulation of the decomposition of the wood by controlling temperatures in the retort has a material effect on the yields of products obtained. Many operators have carried out experiments with this end in view, but probably the most important and convincing are the result of commercial tests carried out for the U.S. Forest Service by R. C. Palmer ("The effect of temperature control on the destructive distillation of hardwood"; see this J., 1915, 898). In his experiments Palmer showed conclusively that by keeping the temperature of the retort as low as possible at the point at which exothermic reaction takes place (275° C.), this reaction could be spread out over a longer time and, therefore, less decomposition of the prime products would take place. The greatest increase in yield was shown to be that of wood alcohol, the resulting increase being 30% over that obtained in uncontrolled operations. The increase in acetic acid (or acetate of lime) was only about 14% under the best conditions.

Any exothermic chemical reaction is naturally more or less difficult to control, the more so when it takes place at a relatively high initial temperature. Another well-known chemical phenomenon bearing on the matter is the tendency of materials in a finely-divided condition to respond to treatment or to react more readily than similar materials in homogeneous masses. Applying these two facts to the distillation of hardwood chips in continuous mechanical retorts, we are immediately faced in practice with conditions which are exactly the reverse of what might be desired for obtaining the most satisfactory results. In the first place the critical temperature, or the point at which the exothermic reaction takes place, is about 275° C., and until this temperature and the resultant decomposition reaction are reached and completed, only 25–30% of the total acetic acid and alcohol will be produced. In the second place there is, instead of a comparatively large and solid piece of wood exposing, say, a unit of surface to the reaction, an extremely large number of small pieces exposing many times the unit surface to the reaction, and consequently reacting individually almost spontaneously. As a result of such uncontrolled distillation mechanical retorts would be expected to give yields considerably lower in alcohol and slightly lower in acetic acid than are obtained in retorts using cordwood or similar material. This is exactly what happens in commercial practice, as the yields in even the most efficiently operated of the mechanical retort plants are as regards wood alcohol nearly 50% less, and as regards acetic acid slightly more than 10% less than those obtained in cordwood retort factories.

In the completion of the destructive distillation of wood there is formed a certain amount of non-condensable gas. Some of this gas is the natural result of the decomposition of the wood, but in addition some is formed by the decomposition of the condensable products formed, due to superheating, etc. If a charge be overheated



at the critical point it sometimes gets out of control, and there occurs what is known as a "blow" of gas. Under these conditions the exothermic reaction proceeds very rapidly, causing a rapid rise in temperature and giving off the products in an excessive amount per given unit of time. The retort and the material inside it being overheated, a large amount of decomposition of the usually condensable products takes place, and in consequence an excessive amount of non-condensable gas is formed. As it is the practice in some plants to burn the non-condensable gas in the fire box for assisting in heating the retort, it can be seen that under certain conditions a vicious circle of excess heat—excess gas—more excess heat may be formed. In mechanical retorts this critical temperature is being reached in certain parts continuously, hence there is a continuous supply of gas for burning. This fact led to an incident in my experience some months ago, when I was investigating conditions at a factory, with a view to a reorganisation and bringing the equipment up to date. I found that the yields of wood alcohol and acetic acid, the former, particularly, were extremely low. The reason for this proved to be that a very small amount of fuel was used for heating, the gas from the retort doing most of the firing, so that they were working at greatly excessive temperatures, overheating the retort and decomposing the alcohol and acid into firing gases, which when burned caused more excessive heating. In other words, they were operating what might be called a gas-producer instead of a retort for low temperature distillation.

The cordwood retort process has had a slow but positive development dating from the period of commencement of more extensive by-product production. The retorts themselves were originally cast-iron cylinders, about 3 ft. 6 in. diam., by 8 or 9 ft., long, with a door bolted at one end and the vapour outlet at the dead end. Working at the temperatures required, and with the rapid heating and cooling which took place in the system, these naturally had a very precarious existence, and great trouble was experienced with cracks, resulting sometimes in disastrous explosions. Gradually the cast iron retorts were replaced by steel plate cylinders, having cast iron doors, and these were set up so that they could be revolved if the side in contact with the fire became burned, thus exposing a fresh surface and giving considerable extension of life. From this type, the next step was to the rectangular steel oven of large capacity, into which the wood was charged on the trucks. This practice gradually became almost universal where comparatively large quantities of wood were used, as the labour and time saved in charging and discharging were enormous. At first these large retorts were merely oblong boxes resting on brick piers, with rollers to allow for expansion. Present-day developments have altered the contour of the retorts to prevent the escape of the vapours more easily, and these retorts are now suspended in the furnaces, expansion being allowed for by the play of the hangers.

The arrangement of furnace flues has also been the subject of many improvements. The original retort was merely set over a fire, the flames playing on and around it. Later a baffle was interposed between the flame and the retort, and gradually the present practice developed of entirely removing fire from the vicinity of the metal retort by means of arches, with positive circulation of the furnace gases around the retort in a manner best suited to the efficient utilisation of their heat.

German and Austrian practice is distinct in that nearly all the retorts, both of small capacity and large, are cylindrical in shape, and are fitted

with an extremely complicated system of flues. The majority of the German and Austrian retorts are of small capacity (not exceeding 2 cords each). Swedish retorts, on the other hand, while showing the result of German origin, are of large size (10—15 tons each) and present several interesting departures in design. The principal one is known as the Gröndal retort, and is of large capacity, but is built in three sections, each isolated from the other and from the air by doors. The first section is the drying and preheating chamber, where the wood is dried; after remaining in this chamber a certain time the truck is removed into the central chamber and is replaced in the drying chamber by a fresh truck of wood. In the central chamber distillation is completed, after a certain number of hours, and the truck moves into the final chamber where the charcoal is cooled, finally being replaced by the following truck on its discharge into the open. The difficulties met in operating this type of retort are mainly mechanical, owing to the trouble in obtaining complete and perfect isolation of the interior chambers.

The present American practice is almost entirely confined to large retorts, having capacities of from 12—20 tons per charge. Most of the plants have daily capacities of not less than 24 tons of wood, the average probably being about 48 cords of 30 cwt. to 2 tons each. A few plants distil as much as 150 tons per day. In recent years many efforts have been made by German engineers and plant manufacturers to introduce into American works, types of apparatus and retort systems which had been developed in German works. One of the large operating companies was induced to make an installation of German equipment, and after spending more than £20,000 on it, the process proved entirely unsuitable to American conditions, besides being in some of its principal features scientifically unsound.

The tendency in Great Britain, on the other hand, has been almost entirely along the lines of small retorts and small capacity, most plants here treating not more than 6 to 10 tons per day in  $1\frac{1}{2}$ -ton retorts. This tendency is, of course, due mainly to the limited supply of wood available in any particular district, and also to the limited attention given to the disposal of products, each producer being apparently satisfied to sell his production in a more or less local market. The first step in the installation of the large retort plants was taken by the Government, who put down a plant of the Continental type, to deal with the coppice and cuttings from the Forest of Dean, controlled by the Office of Woods and Forests. The retort is of the cylindrical type, and treats about 10 cords of wood at a charge, and is fitted with Meyer's so-called "tar separator" and the usual condensing system for the acid vapours.

With the advent of the war, the increasing demand for the products of wood distillation in Great Britain, coupled with the cessation of the Continental supply, caused a considerable shortage. Consequently new installations began to appear and old works were enlarged. One or two idle works were rebuilt and enlarged to meet the demands of the moment, and the industry as a whole took a new lease of life. Very naturally the new installations, particularly as they were mostly on a larger scale than the average plant heretofore operated here, were installed along the lines of American and Canadian experience; the smaller plants, recognising the tendency, also adopted the larger scale plan equipment modified to suit their requirements, so that the present condition of the industry shows two distinct types of retort equipment. On the one hand there is a considerable number of plants using the small retorts, while in the larger plants are installed the ultra-modern type of oven retorts.



The present-day equipment in Great Britain is thus a result not only of the slow evolution of the original retort in this country alone, but is also the result of the development which has taken place in other countries in which the industry is and has been a thriving one.

The retort, being the point at which the products are formed, is without doubt the most vital part of the equipment. Unfortunately the development and improvements in refining equipment and methods have progressed far in advance of those in connection with retorts. Possibly this has been due to the fact that the need of improved refining methods was so obvious; nevertheless the fact still remains that the yields of products from the wood in the retorts to-day lack a great deal of reaching the figures which might be expected from a theoretical consideration of the chemical composition and structure of wood substance.

I am soon starting some large-scale tests along certain lines, after having carried out small-scale experiments the results of which indicate that it is possible nearly to double the output from a given weight of wood in commercial plants.

The type of condenser attached to the original retorts was in nearly all cases a series of large straight copper pipes, passing through troughs or boxes and surrounded with water. No particular method of calculation or design seems to have been adopted—each works copying the original. At various times some operator would add a length of pipe and find that he condensed more products, after which this lengthened size was copied by the others. After the introduction of large retorts a more compact system was necessary, and the result was the construction of the tubular surface condenser, in which the vapour passed through the tubes and was cooled by water circulated in a wooden or iron shell. The small retort plants later saw the advantages of this condenser, and it is now rapidly replacing the older pipe types in this country.

The original products sought in wood distillation were the acetates and acetic acid, little or no demand for wood alcohol existing for many years. The original refining equipment, therefore, consisted of a simple copper still heated by fire, with a coil condenser. The resulting liquor was either shipped as made, or certain metals or salts were dissolved in it to produce the required acetates, which were sometimes evaporated before shipment. These acetates, if dried, were dried in open pans over direct fire.

Later, when the demand for alcohol grew, the liquid that came over first in the distillation was collected separately and redistilled several times to obtain spirit, great difficulty being met in freeing the final product from esters, acetone, etc., due to the acid distillation. In more recent years the once-distilled liquid has been neutralised and then distilled for alcohol, but even this process required four, five, and sometimes six distinct distillations in order to obtain a refined product. Until about 1895 very few plants made use of any kind of rectifying head or column, one or two using a series of water-cooled or air-cooled pots in the final distillation through which the vapours were passed. In every case, however, the acetate liquor was boiled down in open pans, and all distillation was by direct fire.

From this time advancement became rapid. Column stills, first of a crude type, came into use, steam heating coils were introduced for all distilling purposes, thus eliminating many dangers and reducing decomposition, more efficient means of testing the products were adopted, and gradually the refinery came under chemical control. At this period, when by reason of more perfect refining of the crude liquors, a better and

more uniform product was obtained, certain substances were eliminated from the products. These substances became known and used, so that new products began to be recovered, forming an additional source of revenue to the plant from materials formerly considered detrimental waste.

After that period we arrive at the present-day procedure, and find the plants either equipped or equipping themselves with apparatus which enables them to make thorough separation of any or all components of the crude liquors and to produce these components as refined products. During the past ten years the tendency, aside from purely chemical development, has been to alter the equipment in such manner as would economise in fuel and labour in order to meet modern industrial conditions.

The greatest scope for increasing efficiency, both chemical and mechanical, naturally lies at the door of the larger works; and it is in these large works that the most improved equipment is found. The small works of to-day, as of yesterday, present inherent difficulties in the way of efficient operation, owing to the bulk of raw material and comparatively small quantity of product. However, it is pleasing to note that many of the small plants have realised that their path to efficiency lies in the line of that type of equipment for the production of the highest-priced products with the minimum of labour and fuel. The large plants, in replying to the call for industrial efficiency, show wonderful results. When a plant treats many tons of wood per day, even an increased production of a fraction of a gallon per ton, the saving of a few pounds of coal per ton, or the development of a new product, means the addition of no mean sum on the right side of the balance-sheet of a year's operation. Present-day refining equipment has, in fine, to mention only a few of the many points of advancement, added some half dozen prime commercial products to the output, elevated the quality of all the products made as compared with those of a score of years ago, and at the same time reduced the fuel requirements in some cases more than two-thirds.

The purposes of the refinery are roughly, first, to separate easily volatile substances from the tarry matter in solution, second, to separate the acids from the alcohols and oils, and finally to eliminate the water from all. When it is remembered that the crude liquor as supplied to the refinery is about 80% water, it is obvious that the principal requirement is economical evaporation. A multiplicity of secondary or subsidiary actions must also take place, and modern design of apparatus has combined many of these subsidiary steps and eliminated others, using the heat developed in one distillation as a source of heat for a subsequent step, and making use of gravity where possible, so that now the entire process of refining is made continuous wherever possible, and the liquors receive the minimum number of distinct "handlings" before being finished. With every separate distillation, agitation, or "handling" of the liquid some small amount of the product is liable to be lost or destroyed, the quantity varying with the type of equipment. To indicate the high degree of perfection of the latest equipment, I have operating records which show a daily production efficiency of 98—99½% of the theoretical. Another and a very marked instance of the effect of installing efficient equipment was the recent reconstruction of a refinery. This refinery dealt with approximately 3000 gallons per day. Substituting one continuous type of refining still with preheater for the old 3-still periodic system, and a triple-effect evaporator instead of open pans for acetate liquor, produced a more reliable product and reduced the steam

consumption from 38,200 lb. to 10,700 lb. for this particular department.

The yields of what I will call prime products, viz., charcoal, 80% acetate of lime, 95% refined wood naphtha (including esters and ketones), and dry tar, present a fruitful field of research, and have been the basis of a great many experiments. Unfortunately commercial yields obtained from different countries or even different plants have such widely different bases of computation that they cannot be used for comparative purposes. "Yields per cord" or "Yields per ton of wood as charged" mean nothing whatever, as a cord may be anything from 64 cub. ft. stacked to 256 cub. ft., depending on the country and district referred to, and cords of wood of 128 cub. ft. may vary in actual weight more than 100% and still contain very nearly the same weight of water-free wood. I have carefully correlated in Table II. some records of yields obtained in various plants which have come under my experience, and in addition have tabulated yields obtained in American practice, and in experimental operations as collected personally and by other investigators. In each case the actual yields of commercial products have been calculated to production per ton (2240 lb.) of water-free wood carbonised.

wood in mechanical retorts lies, as has been mentioned, in the production of gas at the expense of products, as well as in the fact that the chippings are usually in a very dry condition.

While on the subject of yields it is necessary to point out some of the factors that have a strong influence on the yields obtained in practice. The first point that naturally arises is that of species. All hardwoods do not give exactly similar yields of products, although taken weight for weight there are not many striking differences. The principal differences make themselves evident in the alcohol and tar yields, which may be considered to be due to the composition of the wood itself.

The following results are based on small scale experiments only, but all were carried out under similar conditions, so that they are comparable.

The methods of determining the various products were as follows:—

*Acetic acid.* The crude liquor was distilled until the residue in the flask became heavy, followed by steam distillation until the resultant distillate tested only 1% acid on titration of 5 c.c. The whole was well mixed and 5 c.c. taken for titration with  $N/2$  NaOH.

*Wood alcohol.* The above distillate was fractionated three times over lime and the sp. gr. of

TABLE II.

Plant.	Charcoal.	Acetate of lime, 80%.	Wood alcohol. Imp. gals. 95%.	Boiled tar. Imp. gals.	Coal used for fuel.	Remarks.
	lb.	lb.			lb.	
British cordwood type . . .	795	164 ‡	4.13	12	235	Wood mostly oak, about 30% moisture and 1—2½ in. thick.
Mechanical retort . . . . .	729	137 §	2.26	18	122	Very dry wood chips, mostly birch. Acid strength tests 10—12% in crude liquor.
American cordwood type	815	158	5.54	11	178	Mostly maple 4 in.—8 in. diameter, containing about 15% moisture. Maple predominating.
Commercial experimental†	826	162	6.56	—	—	—
Laboratory experimental	—	197	6.7	—	—	—

NOTES.—\* Includes all low-boiling ketones, esters, and aldehydes as usually found in this 95% commercial product.

† Hawley and Palmer, Proc. 8th Int. Cong. App. Chem., 1912, Vol. 6, p. 133; this J., 1912, 865.

‡ The actual production tests 84—86% acetate.

§ The actual production tests 75—78% acetate.

Even this tabulation should not be taken as indicating the comparative overall efficiencies of the several types of plants, owing to the variation in moisture content, size of wood, and species used in the average materials of the several countries and plants. Broadly considered, however, these figures serve to bear out my remarks on the effect of control of reaction and the size and condition of wood. The reasons for the marked difference in fuel consumption per ton of actual dry wood carbonised in British practice as compared with American, may be found in the fact that the average moisture content of wood in Great Britain is nearly double that of average American wood, and also in the fact that a cord of British wood as charged is made up of pieces of smaller diameter, so that the weight per cord as charged is only about 1½ tons as against nearly 2 tons. Under the practical conditions of distillation, there is a large amount of excess heat supplied to a retort, consequently in distilling two charges of dry wood in equal sized retorts, one occupying considerably less bulk than the other, and of consequent different weights, only slightly different amounts of coal will be used, the excess being necessary for heating the brickwork, radiation losses, etc. It will readily be understood, therefore, why in plants which are favoured with heavy, dry wood, the fuel requirement per ton of dry wood carbonised is less than in similar plants where more bulky wood is used. The reason for the low fuel consumption per ton of

the final distillate determined on a Westphal balance and calculated to methyl alcohol.

*Tar.* The residue from the first distillation of crude liquor was weighed. Several tests of this tar indicated that it contained about 0.5% acetic acid, but this quantity was neglected in the calculation of acid produced.

The figures in Table III. indicate broadly that the best wood for producing acetate of lime is the birch, which yields about 20% more than the average of the six species. On the other hand, for the best production of alcohol, we must turn to the maple, which is again some 20% better than the average.

Another factor which, in commercial operation, particularly, may have an important bearing on the yields, is the moisture content of the wood. The mere fact that water is present in the wood does not of itself necessarily reduce the yield per ton of dry wood present. If a wet charge be heated carefully, and due precautions taken that the drying operation is complete before decomposition begins, then no serious losses need result. If, on the other hand, the charge is fired carelessly and the exothermic point reached before the moisture is eliminated, a condition exists which offers a very favourable opportunity for the setting up of side reactions. In other words, there is a high temperature, possibly slight pressure, and products in their nascent state in the presence of steam. The decomposing effect of steam on many nascent substances is well



TABLE III.

Species.	Charcoal.	Acetic acid.	Wood alcohol.	Tar.	Remarks.
Oak (English) .....	36.1	5.40	1.58	14.7	Air-dry samples 2-in. dia., without bark.
Oak (American) .....	35.0	5.12	1.33	6.3	Air-dry samples 2-in. dia., without bark.
Maple .....	36.8	4.92	2.02	12.6	Selected from Hawley and Palmer's figures for wood without bark.*
Beech .....	38.4	5.22	1.95	9.9	
Birch .....	39.1	6.48	1.45	10.4	
Wattle (mimosa) .....	37.5	5.68	1.59	12.8	
Soft pine (white wood) .....	38.8	2.47	1.01	20.2	Air-dry sample 2-in. dia., without bark.
Average .....	—	5.47	1.65	—	Air-dry sample 2-in. dia., without bark.

\* Hawley and Palmer (*loc. cit.*).

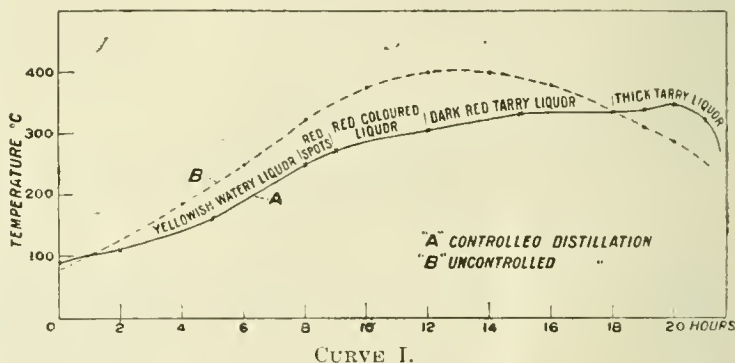
known, although superheated steam is not quite so active under all conditions as saturated steam. However, that saturated steam is present must be admitted, as the moisture at the instant it is formed into steam in the wood is saturated, before it can become superheated. This theory I have never specifically proved by experiment, but it is the result of observation in commercial experience. In distilling wet wood I have many times noticed that if the exothermic point is reached too quickly, a greatly increased amount of gas extending over a much longer period is generated, with a correspondingly decreased flow of liquor. If, on the other hand, the moisture is eliminated before the reaction takes place, the decomposition may be easily regulated and the gas production proceeds normally.

It is in this connection that British operating conditions are more difficult than American, as well as in the fact that the majority of wood used in this country is smaller in diameter than the average American cordwood, and consequently more difficult to control during decomposition.

Another factor which has a very considerable bearing on the fluctuation in the yields is the variation in heating, and to a lesser extent the actual temperature itself. The attaining of high temperatures during a distillation does not necessarily mean that extremely low yields will result, although in those industrial plants where the attaining of high temperatures is a common practice, it has been noticed that the yields have been rather lower than the average. The reason for this is that where excessive temperatures are the rule, there is little attempt made to control the period at which high temperatures occur, and this time factor is most important in connection with temperature. For instance, a retort charge may be fired so slowly during the first portion of the distillation (that is, during the time when water is being driven off) that in order to complete the charge in the 24-hour cycle, it is necessary to rush the last portion. This entails excessive firing during the exothermic reaction and after it, which, of course, means decomposition or "cracking" of a portion of the vapours as formed. Another charge, uncontrolled, may be fired too heavily at the start and reach the exothermic point before the operator is aware of the fact. The heavy fire causes the decomposition to proceed too violently, and again overheating results, with consequent decomposition of products. On the other hand, if a charge be fired carefully at the point of reaction, and for some time afterwards, permitting only a gradual rise of temperature at the exothermic

point, it may be "finished" to a very high temperature without undue decomposition because the "spontaneous" decomposing point is passed and the balance of the reaction may be termed endothermic. The proper method of heating may be quite accurately plotted. A sample time and temperature curve (fig. 1) for normal distillation is shown herewith (curve A) and alongside it is plotted a sample "uncontrolled" run (curve B). A comparison of these curves will illustrate clearly the above remarks. The uncontrolled curve, B, indicates a continual firing, with consequent rapid rise to the exothermic point, but instead of reducing the external heat at this point the temperature continues to a marked maximum and declines rapidly, indicating that the reaction has

Time v. temperature.



taken place in a minimum time and with consequent increased intensity. In the charge which was fired carefully (curve A), on the other hand, the exothermic point was anticipated by easing off the fire, and the reaction spread over several hours at a minimum temperature. When the reaction had practically ceased, one or two hours' additional hard firing completed the distillation and finished the charcoal to hard and lustrous black. Of two similar charges of wood distilled, as indicated in the respective curves, the "controlled" distillation produced 1580 gallons of crude liquor, equivalent to 1785 lb. of 80% acetate of lime, while the "uncontrolled" charge produced 1300 gallons of liquor, equivalent to 1429 lb. of 80% acetate of lime. Another curious occurrence noticed in connection with high temperature during exothermic reaction, is that the content of ketones in the spirit is considerably increased. This might naturally be expected owing to the decomposition of acetic acid. The so-called "acetone" content (ketones determined by the iodoform test) of the first runnings from the rectifying stills has been varied from as low as 17% to as high as 23.5% by variation of the heating of the retorts.

### Operating methods and equipment.

With regard to the retorts, the mechanical type is operated at a disadvantage due, to the fact that all the steps of removing moisture, decomposition, etc., are taking place simultaneously and continually. There is certainly, however, some fixed temperature or condition in the retort at which it is most advantageous to work in order to obtain the largest yields of both acid and alcohol. What this condition or temperature is I am unable from experience to say, but from a theoretical standpoint it would seem advantageous to have the operation in two distinct and separate stages, each stage being conducted in a separate retort or portion of retort, with separate means of firing and control. The practical objections to such a method may be overwhelming, but the idea is advanced as a suggestion for future experiments.

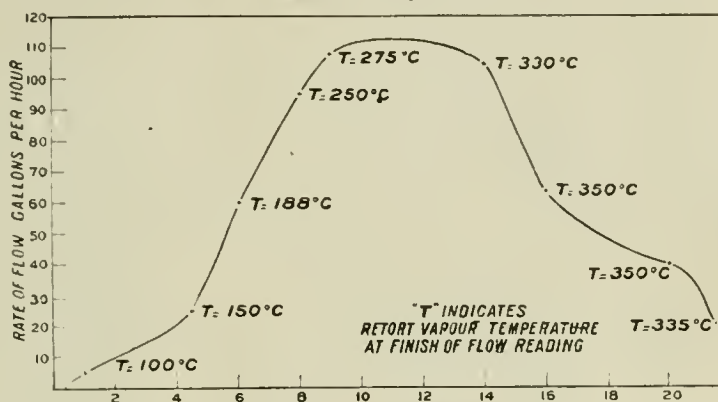
The cordwood retort is not only very easily operated, but produces its own indicator in the liquor, which tells plainly every change which is taking place in the interior. Although many retorts are fitted with pyrometers for the guidance of the operators, after a few months the men become expert at reading the liquor colours, and can actually anticipate the reactions as accurately as with the pyrometers. The recording pyrometer is to be preferred to other types, as a permanent record is automatically made which cannot be evaded, whereas workmen have been known to disregard liquor changes with results detrimental to the plant as well as process.

The operation of retorts is or may be governed largely by the condensers for the vapours attached to them. Condensers may be built of maximum or minimum capacity for a retort. I think it a distinct advantage from the operating point of view to have them of minimum size. A condenser made of maximum capacity for a given retort is made in reality large enough to care for the greatest volume of vapours that such a retort may ever produce. This means, in other words, that no matter how careless the operators may be, no matter how much overheating occurs, with consequent decomposition and excess gas formation, there will never be a "blow" of gas, that is, the condensers will account for it all, and no external evidence will be available to call the attention of the man in charge to what is going on in the retort. If, on the other hand, the condenser be of minimum size, that is, large enough thoroughly to condense and cool all the condensable products from the retort under most efficient conditions of working, then, if through carelessness or for whatever reason, excessive gas is formed, it will immediately become noticeable to anyone in the factory, and can be corrected within half an hour or so. Instead of per-

mitting careless or inefficient operation, the minimum sized condenser makes it imperative that the retort be carefully attended.

A certain amount of the reduction of yields from what may be called the "theoretical" is undoubtedly due to decomposition of vapours where they are in contact with the retort itself and the hot wood, in their passage to the condenser. To obviate this in some degree, retorts have been fitted with two, three, and sometimes four condensers attached at various points. This lessens the length of travel for the average particle of vapour from its point of formation to the outlet, and certainly increase in yields has followed the adoption of this practice. The question is principally one of economies, or, in other words, does the increase in efficiency pay for the increased capital expenditure and attendant multiplication of cleaning troubles? Our experience tends to the opinion that the most economical installation in the nominal 12-ton capacity retort embodies two condensers. One difficulty which has become evident occasionally in retorts fitted with two condensers is that of persuading the vapours to pass to both of the condensers in approximately

Time v. rate of flow.



Time in hours; retort charge—12 tons.  
CURVE II.

equal amounts. There have been times when, without any apparent reason, the entire volume of vapours from a retort has passed through one of the condensers while no condensate has been obtained from the other one. With no alteration or cleaning, the next charge behaved normally, the condensers taking approximately equal portions of vapour.

The purpose of the condenser equipment is to condense the vapours to liquids, and to cool these liquids to as low a temperature as possible. The presence of volatile products renders it vital to successful recovery that the liquor be cooled as nearly as possible to atmospheric temperature. It is during the time of the exothermic reaction, with its consequent heavy flow of liquor and non-condensable gas, that the concentration of the acid, alcohol, and oils in the liquor is

TABLE IV.

Liquor. temp.		Acetic acid per 100 c.c. condensate.	Alcohol per 100 c.c. condensate.	Acid lost per 100 c.c. condensate.	Alcohol lost per 100 c.c. condensate.	% of lost acid recovered in scrubber.	% of lost alcohol recovered in scrubber.
°C.	°F.	grms.	grms.	grms.	grms.	%	%
15	60	12	5	—	0.3	—	98.5
21	70	12	4.7	—	1.4	98.5	94
27	80	11.9	3.6	0.1	2.2	98	90
32	90	11.7	2.4	0.3	2.4	97.8	86.3
38	100	11.5	2.68	0.5	2.43	97.7	82.5
43	110	11.1	2.57	0.9	2.45	97.6	80
49	120	10.7	2.55	1.3	2.48	97.6	77.9
54.5	130	9.9	2.52	2.1	—	—	—



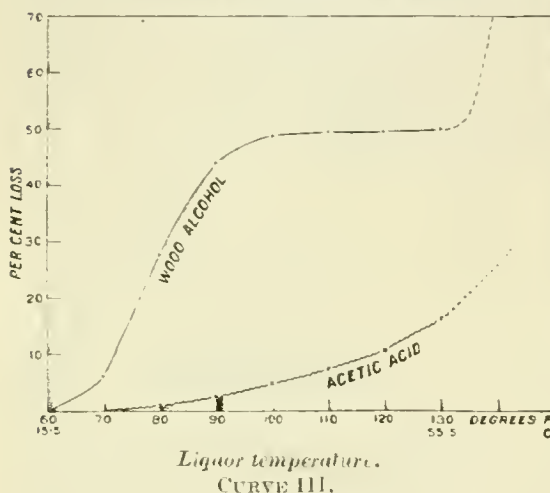
highest, so that any increase in liquor temperature at this point gives full opportunity for the greatest loss of product.

Table IV, indicates the total loss of acetic acid and alcohol that takes place where the gas is not passed through a scrubber after leaving the condensers, if the liquor temperature is allowed to reach the respective figures.

In addition I have appended to the table the proportion of the products (acid and alcohol) recovered by scrubbing the non-condensable gases after leaving the condenser at these temperatures.

Fig. 3 shows the percentage losses of alcohol (A) and acetic acid (B) plotted against the temperatures of liquor. It will be noticed that the loss of alcohol does not increase directly with the increase in temperature, so that the curve A is a very unusual one. There appears to be a period between 38° and 55° C., after a rapid initial loss of about 40%, at which the percentage loss remains fairly stationary; then as the boiling point of the alcohol is approached the percentage

Per cent. loss v. Liquor temperature.



Liquor temperature.  
CURVE III.

loss rapidly approaches 100. It is possible that the acid curve proceeds in the same manner, but owing to the practical difficulties encountered in operation of commercial units the temperature could not be permitted to reach more than 60°C. It will be seen that suitable scrubbing arrangements are almost a *sine quâ non* in connection with condensing equipment, as even with excessive liquor temperatures a considerable quantity of product is recovered that would otherwise be used as fuel. Since between 60 and 80% of the total production of acetic acid and alcohol is produced during the exothermic reaction, the importance of low temperature of condensate and auxiliary scrubbing apparatus is apparent.

The products of carbonisation must be condensed from a temperature somewhere about 350° C., or higher, to atmospheric temperature to obtain crude liquor. As the subsequent refining involves distillation, evaporation, etc., it has been the aim of many designers and investigators to eliminate the first condensation, and utilise the heat present at this stage to perform certain of these secondary operations. On standing, a large portion of the heavy tars separates out from the crude liquor, leaving a supernatant layer of acid and alcohol in water. This watery layer, being acid, naturally carries a certain amount of tarry matter in solution. In order to produce a grey acetate of lime and a colourless

wood alcohol, it is essential that this dissolved tarry matter be eliminated. This has usually been effected by simple distillation, the water, acid, and alcohol passing over as vapour and the tar remaining as residue. The principal systems for eliminating this distillation of liquor were developed in Germany and are known as Meyer's and Jørgensen's systems respectively. Both operate along similar lines, and both have met with a certain amount of commercial success. However they are both basically wrong in conception, and have met with practically no success in operation outside of the Central Empires. The system is based on the idea that by requiring the hot vapours as they leave the retort to bubble through several layers of hot tars, the tarry matter will be collected and the resultant vapours, after condensing, will be watery distillate of acid and alcohol freed from the dissolved tarry substances. Viewed superficially this would appear quite a satisfactory arrangement, and it must be admitted that in practice some such actual result is obtained, that is, the tar is separated and the distillate is condensed in such condition that grey acetate of lime can be produced from it without further distillation of the acid. When that is said, then all is said for the process, for it is impossible to produce colourless alcohol from the liquor by the usual methods, and in addition the oils and resinous matter present cause an excessive amount of lime sludge to be formed after neutralising.

Where the idea is fundamentally erroneous is in the fact that by causing the hot gases to bubble through hot, liquid tarry matter, every opportunity is provided for the interaction of the substances present, with the consequent formation of condensation products, such as phenol-aldehyde resins, acetyl compounds, and methyl-phenol compounds. These formations naturally result in decreased yields of prime products, which is in itself to be deplored. Then, too, the introduction of the necessary apparatus causes a back pressure to be put on the retort, and an exhaust fan is necessary. This in turn tends to increase the speed of travel of the gases, with increased entrainment losses. The most promising process lies along the lines of fractional or even differential condensation of the heavier substances. The result of experiments on this line has been the production of a system of separation which shows every promise of meeting the requirements of present-day operation, without the difficulties present in the above-mentioned system. In this new system, which is being protected by the necessary patent, the vapours are passed through a two-stage cooler in which the heavy tars are condensed differentially and removed immediately from contact with the gases. The remaining vapours, consisting of water, acid, alcohols, and some light tar oils, then pass to a final separator in the form of a rectifying column, in which the tar oils by reason of their higher boiling points are eliminated at temperatures just above the boiling point of water, the final condensate being a clear liquor slightly tinged with yellow, which differs in practically no respect from the white acid obtained by ordinary distillation of the crude liquors. It is quite possible to make use of the remaining heat of the vapours before condensing to separate the alcohols, but we have not found this to be profitable on a small scale, in view of the highly efficient rectifying plant available under present conditions.

The detailed consideration of refinery practice and equipment for producing the prime products as well as the derived and secondary products must be left to another paper.

Great Britain's pre-war requirements of wood distillation products have been summarised, and mention made of the impossibility of increasing

the native industry sufficiently to account for the necessary quantities. The magnitude of any future development depends on the degree of acceptance by the Government or by private owners of a suitable and complete scheme of afforestation and forestry control. Whenever the laws of scientific forestry have been allowed to control timber lands, such lands have become a source of profit to the State or the owner, directly as well as indirectly.

Possibly the most serious difficulty to be met, as has been the experience of most other countries, will be the suitable and profitable disposal of the charcoal. Every effort should be made by the associated producers to develop new markets or uses for this material.

The inauguration of a scheme of forestry on a national scale requires time, so that to satisfy immediate requirements, the timber resources of the Colonies must be called into use. In this respect the Empire resources may be said to be inexhaustible. Canada already has her industry established. South Africa has available an unmeasured quantity of wattle wood (mimosa), of which a great amount is at present waste material from tannin extract factories, for the bark only is used for this purpose, the wood being left or burned. Australia, with her present timber industry, has available enormous quantities of lumber and sawmill waste suitable for distillation. Here, also, the demand for wood oils, for flotation of zinc and other ores, brings an active market at the distillers' door. India, the Federated Malay States, Borneo, etc., all have unlimited quantities of wood, and have markets locally for many of the products, such as charcoal, acetic acid for rubber coagulation, tars for disinfectants, etc. Japan extends an open market for the various products in which these Colonies and countries would have a great shipping advantage. It may be said, therefore, that the Colonies offer an exceptional field for the immediate development of the industry, and as only very crude labour is necessary for most of the departments, Colonial conditions present distinct advantages for successful commercial operation. We may look, therefore, to these countries supplying our markets in the near future with the 50% production that previously came from Germany and Austria.

#### DISCUSSION.

Mr. W. D. A. BOST said that, with reference to the yields obtained in mechanical retort plants, the figures presented did not agree with those given by Haig (this Journal, 1890, 1120). With regard to the differences in products from pine woods and hardwoods, he pointed out that in the former the oils and resins were present in the wood and were merely driven off by the heat, while in the latter the products were formed from the decomposition of the wood substance itself.

Prior to the war, some 2000 tons of charcoal had been imported annually, but now the home production had been increased by about 15,000 tons, so that the post-war disposal of this excess would be a serious matter.

Professor T. GRAY enquired as to the point at which the vapour temperatures of the retorts were taken, and pointed out that the apparent temperature might be higher or lower than the actual temperature, due to radiation from the brickwork, etc., which raised the question as to whether 275° C. was the actual temperature of exothermic decomposition. He enquired if it were not practicable to use several retort outlets, connecting them to one condenser, thus overcoming the tendency for the gases to travel unequally to the two.

The CHAIRMAN enquired as to the effect of injecting steam in wood distillation.

Mr. LAWRENCE, in reply, said that the figures

given for yields covered several types of mechanical plants, not the worst nor the best being taken for the comparative purpose. The temperatures were in nearly all cases taken in the vapour outlet from the retort, before the gases reached the condenser. The vapour outlet was very short, but as the distillation was carried out at a comparatively low temperature the brickwork should not materially affect the readings, as the pyrometer couple was entirely immersed in the flowing gas. The temperature inside the retort itself was probably higher than that shown by taking the temperature of the vapours at the outlet, but knowing the indications of the exothermic point, as shown in the colour of liquor, any arbitrary temperature curve would serve the purpose of control. The temperature of the flue gases at the chimney was usually about 100° C. higher than the retort gas temperatures. The exothermic point, 275° C., had been determined experimentally by many investigators, notably P. Klason and others. The reason for not connecting several outlets to one condenser was the trouble experienced in choking of pipes when exposed to the air for any distance before reaching the condenser. He had seen a 30-in. bore pipe completely choked in a very few weeks where it was only about 6 ft. long between retort and condenser. The use of steam in the retorts during wood distillation had been tried with a view to clearing the gases out more rapidly, but the result had been adverse, due to decomposition and to the dilution of the liquors. Probably the increased yields when steam was used with coal, were attributable to the high temperature at which the distillation was carried out. In wood distillation the products were nearly all soluble in water and the water must be evaporated. That was not so much the case in coal distillation products.

#### Newcastle Section.

*Meeting held at Newcastle on Wednesday, December 12th, 1917.*

MR. H. FEILE IN THE CHAIR.

#### THE MELTING POINT OF COAL ASH.

BY J. T. DUNN, D.S.C.

In 1913 it became necessary to ascertain the melting points of the ash of a number of samples of coal for certain industrial purposes. There proved to be so much variation amongst them that it was decided to investigate the matter further. The ashes were accordingly analysed, and gave the figures tabulated on page 16*r*, columns 2 to 17. As a matter of interest, the figures in columns 1 and 18 have been added. The ashes are arranged in ascending order of melting point, and the letters, N, D, S, indicate respectively Northumberland, Durham, and West of Scotland coals. The temperatures were taken by a thermoelectric pyrometer and also by Seger cones, but are not to be regarded as accurate within 10° or 20° C. Alkali was taken by difference, except where marked "d.," in which cases it was determined.

Further work on these has been stopped by the pressure of more urgent professional work, and the analytical figures are now presented as a matter of interest. Half a century ago, Richters made a very elaborate and interesting pioneer investigation of refractory materials (fireclays), and inquired into the conditions under which their melting points varied. He arrived, empirically, at results some of which are now known to be typical of the general behaviour of mixtures of more or less fusible substances. He found that, if alumina and silica were melted together, the



melting point of the mixture became lower on passing from pure silica to more and more alumina and then rose again until it reached the point of pure alumina. He further examined the effects of different weights of different basic oxides and came to the conclusion that the effect produced upon the melting point was determined by the quantity of the basic oxide which was added and its molecular weight, so that, if one adds to a given

## DISCUSSION.

Prof. HENRY LOUIS suggested that a good deal of progress had been made in such matters since Richters' time, and he did not think they could accept his theories as correct. Lead oxide, for instance, lowered greatly the fusion points of silicates. Furthermore the question of the state of oxidation of the iron in the ash was a vital point. Ferrous silicate was a very easily fusible substance, whereas

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19
		D	N	D	D	N	D	N	N	S <sup>a</sup>	S	S	D	D	S	N	D		
Silica .....	25.90	45.50	36.90	40.80	40.40	37.40	42.10	42.70	45.40	41.20	46.93	49.30	43.30	47.10	48.50	51.10	51.80	51.15	51.89
Alumina .....	17.46	25.69	25.82	31.36	29.34	28.21	30.67	35.05	35.53	33.45	33.23	35.40	38.88	37.39	36.93	35.93	38.25	44.70	44.59
Ferric oxide ..	16.86	19.00	25.00	24.00	15.21	22.14	21.71	8.79	12.57	17.14	8.14	7.29	11.71	11.14	7.36	4.86	5.64	1.25	0.98
Manganese oxide ..	trace	0.20	0.30	0.20	0.30	0.25	0.30	trace	none	0.40	0.20	0.30	0.90	0.40	0.10	trace	trace	trace	none
Lime .....	22.80	2.09	3.71	1.19	4.61	4.65	0.99	5.84	3.54	3.79	5.24	2.96	1.36	0.82	3.33	2.78	0.45	0.15	0.38
Magnesia .....	3.22	1.16	2.10	0.36	1.96	1.67	0.76	1.30	1.23	1.78	0.95	1.34	0.43	0.65	1.08	0.80	0.74	0.36	0.38
Alkali .....	2.70	2.68	4.79	1.15	7.23	4.23	2.55	2.09	0.83	0.83	2.82	1.90	2.12	1.38	1.54	3.32	1.71	1.20	1.77
Titanium oxide ..	0.74	1.31	1.38	0.94	0.95	1.45	0.92	1.16	0.90	1.41	2.49	1.51	1.31	1.12	1.16	1.21	1.41	1.15	0.15
Sulphuric anhydride	8.91	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Phosphoric anhydride	1.55	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Quicklime .....	5.95	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Baryta .....	—	2.37	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Melting-point, ° C...	1020	1150	1150	1200	1230	1330	1340	1380	1380	1390	1415	1460	1460	1460	1480	1480	1500	—	—

mixture of alumina and silica a quantity of another metallic oxide, the amount by which the melting point is lowered is almost independent of the nature of the material, provided that its quantity is proportionate to its molecular weight, and that, if a number of different oxides are mixed together with the alumina and silica mixture at the same time, the reduction of the melting point depends upon the number of molecules of the oxides which have been added to the given number of molecules of alumina and silica.

Calculating the above analyses to molecules, and taking the number of molecules of alumina and of other basic oxides associated with 1000 molecules of silica, the following figures are obtained:—

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
Mol. of alumina .....	382	314	391	426	402	420	405	455	435	453	394	400	499	443	422	389	410	485
Mol. of other bases ..	—	307	526	286	456	471	291	288	240	351	256	194	199	155	184	158	136	38

It will be seen from them that, whilst by no means always accordant with Richters' rule, they do indicate in general that the melting point rises as the relative proportion of basic oxides diminishes.

Attention may be drawn to Nos. 1 and 18. No. 1 is the ash of a Natal coal, and of the total lime (22.80%), 6.24% is present as sulphate, 2.0% as phosphate, and nearly 6% as quicklime (due of course to carbonate in the original coal). The silicates thus form only about 75% of the total ash. No. 18 is the ash of a Spitzbergen coal, perfectly white, and almost identical in composition with a china clay (No. 19 is the analysis, for comparison, of an ignited Cornish china clay). I have no doubt, although I have not had an opportunity of testing it, that this ash would show the same refractoriness as a china clay. The peculiar ash of a Kent coal, which contained only 4% of silica and 80% of ferric oxide—a rich iron ore—is also noteworthy.

These determinations of melting point were made for a briquetting plant for iron ore, where the briquettes are taken in wagons through the furnaces; in the course of this operation considerable trouble resulted when a change of coal was necessary because of the failure of the usual supply, from the ash of the coal with which the furnaces were fired being deposited on the wagons until they clogged up and jammed in the furnace.

ferric silicate (the existence of which was very debatable) would be fusible with difficulty. It was known that magnesia rendered silicates far more infusible than a good many other substances of both higher and lower molecular weight, so that the problem could not be worked on the lines suggested by Dr. Dunn. He thought that the modern view was that each silicate had its own melting point, and in the case of complex silicates silicates formed eutectics. The very elaborate work of Prof. Vogt should be consulted. The whole subject was exceedingly complex and could not be disposed of by the simple theory of molecular weight.

Mr. C. H. RIDSDALE said that the fusibility of ash had a very important practical bearing on the use of coal. Dr. Dunn had mentioned one

way in which the coal acted detrimentally, namely, by the ash becoming deposited upon the bogies which carried the briquettes through the furnaces. Another instance was that in raising steam by means of coal that had a very fusible ash, it would soon be found that the grate bars silted up or the air space became reduced and the amount and evaporative power of the fuel that could be burnt would be very much reduced.

Mr. S. H. COLLINS stated that the Board of Agriculture particularly wanted information with reference to the potash content of ashes, and he would be very glad if anyone who knew of any large quantities of coal ash likely to contain potash, even in very small amounts, would inform him. It did not depend upon the percentage of potash in the coal ash but upon the quantity which could be washed out with water. The expense of so washing it out was very small.

Mr. H. DUNFORD SMITH stated that he believed that some of the Continental railway companies had a specification for the melting point of coal ash and that they would not accept a coal with a melting point below 1400° C. for use on their express trains.

Mr. WEYMAN asked whether the temperatures given were those at which the first signs of softening appeared, or those of complete fusion.

Dr. DUNN, in reply, said he entirely agreed with Prof. Louis as to the advances made since Richters' day, although he had, perhaps, given them the impression that nothing had been done since. He had only referred to him as a pioneer. It was quite true that subsequent research had proved that his rule did not hold in every case. The coal-ash cones he used were always allowed to melt completely and the temperature was taken when they had sunk down entirely. It was very noticeable that different coal ashes behaved in very different ways. Some of them melted quickly—like ice, so to speak—whereas others went through a long period of gradually decreasing viscosity and the temperature at which they began to show signs of motion was many degrees below that at which they finally liquefied in a drop or blob at the bottom. Most of these ashes contained anything from 1 to 3% of potash.

The CHAIRMAN remarked that there was no doubt that it was a subject of great importance, especially as regarded the quality of coal used. He did not think it was at all customary for buyers to insist upon a particular melting point of the ash, although it was very important.

## New York Section.

*Meeting held at Chemists' Club on Friday, November 23rd, 1917.*

MR. JEROME ALEXANDER IN THE CHAIR.

### RECOVERY OF POTASH FROM WOOL-SCOURING WASTE.

BY ROBERT SPURR WESTON.

For many years the author has been interested professionally in the engineering and scientific problems connected with the cleaning of rivers, and has devoted a great deal of attention to the disposal of factory wastes, among them wool-scouring waste. Wool-scouring waste is one of the most befouling and greasy liquids which streams receive. If discharged unpurified, it fouls the banks and bottom, and causes the growth of dense masses of aquatic vegetation, which subsequently becomes covered with wool fat, dies, disintegrates, becomes detached from the banks and bottom of the stream, and drifts down to damage the property of riparian owners.

In one bleachery, said to be the largest of its kind in the country, grease spots on the finished cloth caused damages at the rate of several thousand dollars a month, until the wool-scouring waste discharged by three large mills above was purified. In another case, the discharge of wool waste prevented the dyeing of delicate fabrics. In still another, the waste fouled a stream so much as to cause a down-stream mill to shut down a part of its plant until the water could be filtered before use.

The condition of such rivers as the Merrimack, the Blackstone, and the Passaic is too well known to comment upon. Notwithstanding that they are large streams, they are so polluted as to be useless for many industrial purposes unless the water be first purified.

Even when wool-scouring waste is discharged into a sewer connected with municipal disposal works, it will, unless greatly diluted, cause many types of disposal beds, especially intermittent sand filters, to clog rapidly, and to become useless. Hence the difficulty of the sanitary engineering problem of wool-scouring waste disposal. The waste fouls the rivers unless treated, and works designed for the purification of domestic sewage may be unable to dispose of it.

Until the great war, the only practical method of treating wool-scouring waste was to precipitate the solids with sulphuric acid or acid sodium sulphate. The precipitated sludge was then dried on sand beds and the grease recovered from it by pressing in steam-jacketed hydraulic presses. This method produces an acid wool grease or so-called acid dégras; it is now worth 95c. a pound and may be produced for less than 4c. The potash in the supernatant liquor is in the form of sulphate, but the liquor contains much other mineral matter and free acid, and no method has yet been devised to recover the potash economically. The liquor can be discharged into a stream, although filtration may be necessary if the stream be small.

Since the war, the old-fashioned acid or "cracking" process just described has been modified, in this country, chiefly through the efforts of the Du Pont de Nemours Co. The improvement consists first in allowing the waste to flow over solid nitre cake, a cheap and abundant waste product. Then, instead of precipitating the greasy sludge to the bottom of the treating tanks, the waste is so treated in a continuous subsiding basin that the grease rises to the surface in the form of scum. This is skimmed off, the acid neutralised with sodium carbonate, and the whole heated to boiling. The use of soda causes the dirt to separate from the grease and avoids the necessity for presses. The grease and soap are then skimmed off, re-acidified with nitre cake, and the acid grease is clarified by allowing it to stand over acidified water in the usual way. This process produces a very light-coloured grease, and the liquor is of nearly as good character as produced by the old-fashioned process. This continuous acid process, like the older one described above, produces a waste from which potash cannot be recovered economically, unless some method be devised for evaporating the acid liquor. There are other methods of treating wool-scouring waste, however, and many have been known for a long time, but have been considered impracticable on account of the low price of potash. Among the many considered, three main types may be described.

1. *Precipitation process.* This consists in first allowing the waste to settle to remove sand and other settling solids and afterwards passing it through a centrifugal machine to remove the free fat and the finely-divided suspended matter. The solids in the machine effluent are then precipitated by chemicals and the sludge so formed is dried, using filter-presses, or, if necessary, hot-air driers. A low-grade grease is extracted from the sludge and the latter is ground and dried for use as a fertiliser base. The filter-press effluent is then evaporated, the residue calcined in a rotary furnace, and the potash recovered by lixiviation.

2. *Cardem process.* This is a process which has been successfully employed to reduce sulphite pulp liquor and tanning extracts to a powder. The apparatus consists of a series of chambers through which hot flue gases are passed and into which the settled and centrifuged wool-scouring waste is sprayed through special nozzles. The process is carried out in two stages, known as the wet chamber and the dry chamber respectively. If a dry product is to be secured, the concentrated waste from the wet chamber must be degreased by solvents. The dried product, quite hygroscopic, may be used without further treatment or may be calcined and lixiviated to recover the potash.

3. The settled and degreased liquors may be concentrated to a liquid containing 65% of solids, using an evaporator especially designed to prevent foaming and scaling, and the concentrated liquor coked in an open furnace, after which the potash may be removed from the coke by lixiviation. This process is facilitated and made more profitable



by degreasing the concentrated liquor before coking or by washing the wool in naphtha.

It may be noted that in all of the processes just outlined, the first steps are the subsidence of the waste to remove the settling solids, and its passage through a centrifugal machine to remove the free fat and coarser colloidal suspended matter. The degreasing process by itself is profitable. The grease recovered by the machines sells for an average of 10 c. a pound, and the better grease may be used for the manufacture of lanoline, which is now worth 41c. to 46c. a pound. In the author's opinion, it is the centrifugal machine which makes the recovery of potash possible. Former attempts to recover potash from wool-scouring waste failed because of the formation of an impure, vitreous potassium silicate during calcination. The potash in this vitreous silicate is insoluble in water and its recovery from the ash is as difficult and the cost as prohibitive as from a natural silicate. During the last year, cokes the ashes of which contain 95% of soluble matter, consisting almost entirely of potash salts and sodium carbonate, have been produced from settled and centrifuged wastes. During the war, the "potash" produced will contain sodium as an impurity because of the decrease in the use of potash soaps in the wool-scouring process.

Another essential improvement has been made in the mill scouring rooms, where a great saving in water and consequently a greatly increased concentration of both grease and potash has been effected. Except in two cases where the wool is degreased by the naphtha process and subsequently washed in water without soap, the American mills wash the wool in so-called trains of from three to five bowls. Formerly nearly all of the mills used clean water to refill the bowls, but where recovery methods are practised, it is essential that the washing process be conducted on the counter-current principle, that is, the first bowl only should be discharged to waste, and it should be refilled as far as possible from the second bowl, the second from the third, the third from the fourth, etc. Thus the wool and the water will pass through the train of bowls in opposite directions and the minimum of water will be used.

Where potash recovery is practised abroad, the so-called desuinting process is used, that is, the wool is first soaked in water which dissolves the potash, and then the solution is evaporated and calcined. In America, however, this process is seldom used, largely on account of the extra machinery and attention required. Usually the wool and soap with other detergents are introduced into the first scouring bowl, while water alone is used in the remaining bowls. In most mills the contents of the first bowl are used until they become so dirty that they must be changed.

Instead of using alkali and soap in the first bowl, one mill omits the soap and adds it to the second bowl, thus producing a first bowl discharge much more suitable for the recovery of grease and potash than if soap were used. The discharge from the second bowl may be treated by the acid process if the unpurified discharge would injure a stream; otherwise, it would hardly pay to treat it even in war times. Another mill has tried the plan of using over again the settled and centrifuged waste discharged from the first bowl, and has been able to do this three times and still produce a properly scoured wool. The water saved in the first bowl is passed through the end of the train, thus cleaning the wool without diluting the waste. In this way the waste can be concentrated to contain several per cent. of solids. In a few mills water and soap solution are passed continuously through the train of bowls. This would seem to be the ideal method of using water if by-products are to be recovered.

For the recovery of by-products, the best method of washing wool is the naphtha process. Wool which has been washed with naphtha is subsequently washed in water and the waste produced is free from grease and easily concentrated. It can be evaporated to dryness without difficulty and calcined to produce a coke containing as high as 45% of potash.

The various processes outlined have all been tried in practice. The precipitation process is too expensive on account of the large amount of chemicals required to precipitate the suspended matter, and too complicated on account of the fifteen or more distinct operations necessary.

The Cardem process is very promising, and we have tested it quite thoroughly, using the greaseless waste from wool which had been washed with naphtha. Undoubtedly where waste flue gases are available, their use enables the waste to be concentrated more cheaply than by any other process and has the further advantage of causing little loss by entrainment. Unless degreased, the waste cannot be evaporated to a dry, friable mass in this evaporator, and even if the waste from the wet chamber be degreased before its final concentration in the dry chamber, the product is very hygroscopic; consequently it must be coked before it can be used directly as a fertiliser or before it can be lixiviated for the recovery of the carbonate. Where waste heat is available, the wet chamber of the Cardem apparatus can take the place of the multiple-effect evaporator, but if the dry chamber be used the final product ought to be coked, and it may prove best to perform the final evaporation and coking in the same apparatus, usually an open pan, heated by fuel oil.

The process which is usually recommended is to evaporate the machine effluent in a triple-effect of proper design, or in a Cardem wet chamber, degrease the concentrate with an immiscible solvent, distil off the solvent from both aqueous and solvent layers, and then coke the aqueous concentrate in an open pan heated by fuel oil. The concentrated mass takes fire during coking and incinerates to a porous, gray, non-hygroscopic solid, which may be ground and lixiviated for the production of a nearly colourless solution of potassium and sodium carbonates, which is nearly free from silica and from which the potassium salt may be recovered by fractional crystallisation. At present the coke finds a ready market in the fertiliser industry. It usually contains 20% or more of potash as  $K_2O$ .

There were many practical difficulties which had to be overcome before the waste products could be recovered economically. The centrifugal machine eliminated the silica, but the evaporation of a soapy, alkaline liquor in an evaporator was a real problem, and for this reason the Cardem apparatus promises a satisfactory solution, although the first cost of the plant plus royalty is higher than the first cost of an evaporator.

The first and second evaporators tried were of the ordinary, tubular type in which the vapour spaces were about twice the usual volume, that is, the effects were nearly twice the usual height. One of these evaporators, having each effect 4.5 feet in diameter, had vapour spaces 9.5 feet high. Notwithstanding these liberal allowances, the liquor foamed so much in the first two effects that it filled the vapour spaces with a mass of bubbles, many of them 6 inches in diameter; this foaming was attended with loss of liquor and low efficiency. The first evaporator tried was about 55% efficient. The second was ordered amply large to make allowance for foaming. It was then learned that the bubbles can only be broken up by high vapour velocities, and at present two evaporators embodying this principle are being tried. One of them is reported to be work-

ing efficiently and economically; the other has not yet been tested.

The evaporation problem has been solved satisfactorily. With proper apparatus, triple-effect or wet chamber, degreased waste may be concentrated to 65% of solids. It is not usually practicable to obtain this concentration in a triple-effect. It is necessary to use a small finishing pan or open kettle for the final concentration, because the boiling point of the waste rises rapidly above concentrations of 55%. In the Cardem process the liquor is sprayed into the chamber, over and over again, until concentrated sufficiently.

The difficulties encountered in degreasing by immiscible solvents are neither unusual nor unfamiliar and are easily overcome. A large ratio of solvent to waste must be used, otherwise the solvent will not separate from the aqueous liquid. No untried apparatus is necessary for the recovery of the grease and the warm product from the evaporator in which the aqueous portion is treated is fluid even at high concentrations.

As mentioned previously, the degreased liquor may be concentrated to 65% of solids. One sample of evaporator concentrate which had not been degreased had the following composition: sp. gr., 1.158; b. pt., 104.3° C.; total solids, 37.1%; ash, 15.5%; fats (by ether), 5.9%; potash ( $K_2O$ ), 7.0%. This analysis represents the minimum degree of concentration which is practicable. The process of degreasing reduces the water content and the residual potash liquor may then have the following composition: sp. gr., 1.38; b. pt., 120° C.; total solids, 65%; ash, 29%; potash ( $K_2O$ ), 13.4%; fats (by ether), 0.7%. The concentrated liquor readily cokes to a porous solid containing from 40 to 46% of potash as  $K_2O$ . One sample of coke produced from waste from wool scoured with naphtha contained 46.3% of potash, and its ash had the following composition: Soluble in water, 95.2%; potassium carbonate, 77.3%; equivalent  $K_2O$ , 50.0%.

The calcined coke from ordinary wool waste using soda soap (potash soaps have scarcely been obtainable since the war began) had the following composition: Soluble in water, 88.5%; silica, 3.5%; potassium carbonate, 44.9%; sodium carbonate, 40.0%.

To recover the grease, the waste, first heated to 60° C. or above, is allowed to subside for several hours in a tank, and is then reheated to 72° C. and passed through centrifugal machines, where from 40 to 70% of the ether-soluble fats are removed. The hot effluent should then pass directly to the evaporator to avoid loss of heat.

The grease from the machines contains from 5 to 20% of water and some suspended colloidal matter. It is re-emulsified and re-machined to produce a grease containing less than 2% of water and practically no suspended matter. The quality of the centrifugal grease varies greatly, depending upon the grade of wool washed and the care exercised to prevent fermentation of the waste during sedimentation and degreasing. The best grease is produced from Australian wool and the worst from the dirty Territory and domestic wools. U.S.P. lanolin can be made from the best grease, while the cheaper greases are used for stuffing leather, lubrication, veterinary ointments, etc. The grease recovered by the solvent process is dark coloured and contains many organic impurities. Its odour is pronounced and it is used chiefly for stuffing leather, for cheap lubricants, and in general for those purposes for which acid wool grease is employed.

**Cost.** The average wool-scouring plant probably has two trains of bowls discharging 12,000 gallons or 200,000 lb. of waste daily. The cost of a plant for the disposal of this waste will vary

greatly with conditions, but will rarely, if ever, exceed \$10,000, even at the present high prices. The 200,000 lb. of waste will, on the average, contain 4000 lb. of grease and 2500 lb. of potash, and there may be recovered therefrom 2000 lb. of centrifugal grease, 1750 lb. of extracted grease, and 1600 lb. of potash. The cost of operation is approximately as follows:—

	Per diem.
Cost of centrifugal process .....	\$45
Cost of evaporation .....	48
Cost of degreasing .....	35
Cost of coking .....	17
Management .....	20
<b>Total .....</b>	<b>\$165</b>

#### Value of products.

2000 lb. of centrifugal grease at 10c. ....	\$200
1750 lb. of extracted grease at 8c. ....	140
1600 lb. of potash at 17.5c. ....	280
<b>Total .....</b>	<b>\$620</b>
Daily profit .....	\$455

This profit may seem large, but one mill operating three trains of bowls is at present making a profit of \$500 a day on centrifugal grease alone, and it is believed that at present prices a plant may be built and amortized before the end of the war. At pre-war prices, the cost of operation would be slightly less and the value of the products \$184, thus leaving a small profit for a plant of this size. In the case of a small, single train plant, the pre-war profit would have been a nominal one but the stream would have been kept clean and valuable products would have been reclaimed from a waste otherwise a nuisance. It must be borne in mind that when potash is recovered no waste is discharged into the stream, and even the degreasing plant alone greatly improves the character of the polluted stream.

The waste has another set of possible by-products, namely, those produced by the destructive distillation of the coke—among them some valuable waxes of high melting point, and possibly other commercially valuable substances. However, these possibilities have not yet been sufficiently studied for one to give an opinion regarding their value.

This paper deals, of course, with a general problem, but just as wools vary, so do the applications of the process in specific cases. Therefore each case, beginning at the scouring room and ending at the stream into which the waste is discharged, must be studied with care before decision can be made regarding the practicability of recovering valuable by-products from the waste in question. Every plant should make this study so that the dearth of wool grease and potash, formerly imported, may be in a measure relieved.

## Communications.

### THE MANUFACTURE OF PORTLAND CEMENT.

BY EDWARD D. BOYER.

[ABSTRACT.]

The history of the commercialising of Portland cement is a history of inventions and improvements in its manufacture. Perhaps the greatest among these is the substitution of the rotary kiln for the old stationary dome kiln. In the early days the mixture of the raw materials, usually in the form of moulded brick, was placed in these dome kilns with alternate layers of coal or coke. The output of these kilns seldom exceeded 100 barrels per day. This process of burning was continued until about 24 years ago, when the Atlas Portland Cement Company began experimenting with and rapidly developed the rotary kiln. This type of kiln is to-day being used for calcining Portland cement



in every mill in the United States, and was rapidly adopted in Germany and England. These rotary kilns produce from 500 to 3000 barrels per day according to their size, which varies from 60 ft. long by 5 ft. diam. to 240 ft. by 12 ft., and they alone have been largely instrumental in reducing the cost of manufacture to such an extent as to make Portland cement an economical building material.

Portland cement clinker is one of the most refractory materials and the machinery used for its reduction to a fine powder—78% through a 200-mesh screen—wears out rapidly and constant repair and renewal of parts are required, making the cost of grinding very high. But undoubtedly the most serious loss is that of the heat and dust passing out of the stacks from the rotary kilns. Some few of the plants use the heat from the stacks for generating steam under their boilers, and since the outbreak of war, particular attention has been given by a number of the manufacturers to the recovery of potash from the dust of the kilns.

Portland cement consists mainly of lime and silica, with a certain amount of alumina and small amounts of free salts of potash, soda, and magnesia. The raw materials may contain as much as 2½% of potassium oxide, but normally 0.8 to 1.25%. Efforts are now being made to recover this potash, and it is claimed that a large plant in California has succeeded in doing so. Two Eastern cement plants have also adopted the system and claim success. It is estimated that the Portland cement mills of the United States as at present operated are capable of producing about 100,000 tons of potash per year, or about 33% of our normal requirement.

The process used is the Cottrell electrical precipitation method, the purpose of which is the removal of suspended particles from gases by the aid of electrical discharges. The gases carrying the suspended finely-divided particles of dust are passed between two systems of electrodes, one of which carries a negative electrical charge, while the other carries a positive charge. In ordinary practice the negative electrodes are small in size, such as iron wire or chain, and the positive electrodes are large, such as iron plates or pipes. The gases are divided into several channels and passed through the space between the wires and the plates or pipes; in the latter case each pipe has a wire placed along its longitudinal axis. The electrodes are charged by being connected with a source of high voltage electricity, consisting ordinarily of a high voltage transformer for increasing the electric potential up to the working voltage of 70,000 to 80,000 volts, a rectifier for changing alternating current into direct current, and a switchboard provided with the necessary standard control equipment. The suspended particles while passing between the electrodes become electrically charged and are then driven to the plates or the inner surface of the pipes by the forces of the electric field.

Each precipitator consists of a lower header and an upper header, with twenty vertical tubular electrodes fastened between. The lower header is connected by means of a damper inlet with the main distributing flue, and the upper header is connected with a small separate stack mounted directly over the unit, this stack also being supplied with a damper at the outlet end. The bottom of the lower header is provided with a hopper to receive the collected material, which is delivered by feed spouts to cars on the ground floor. The tubular or "collecting" electrodes are 13 inches outside diameter and 15 feet long, and consist of standard well casing. They are fastened securely to both the upper and lower headers by means of flanged ends on the pipes, these being riveted to the header plates. The inner or "dis-

charge" electrodes consist of No. 9 copper wire, this diameter wire corresponding to the conductivity of the gases here under treatment. These discharge electrodes are supported upon bus bars which, in turn, are supported upon insulators, all in accordance with standard design.

In the operation of these treaters, the finely-divided dust carried out of the kilns by the gases is precipitated upon the tubular electrodes, as the gases pass up through the precipitator. The precipitated dust clings tightly to these electrodes, which are given a mechanical jar from time to time, and the dust, thereby shaken loose from the electrodes, falls into the hoppers underneath the lower header. Each hammering member has six loosely hung hammers mounted upon it, each inner hammer serving to jar four pipes, but each pipe is hammered on two opposite sides. The hammers impinge upon lugs welded to the sides of the pipes; adjacent lugs are fastened together by means of small bolted plates. The hammering system is operated manually. This cleaning system is very effective and simple and the cleaning of the pipes of the complete installation requires but a few minutes, and is carried out about once every two hours.

The potash is volatilised in the "burning zone" of the kiln, the amount volatilised depending entirely upon the factors of time and temperature. The emission of potash apparently follows the ordinary vapour pressure laws, being quite independent of complex chemical considerations. Potash is probably volatilised from the mix in the form of oxide, which immediately reacts with various constituents in the gases to form carbonate or other salts. In the ordinary operation of cement kilns the potash combines with the sulphur dioxide, resulting from the sulphur in the fuel, and the sulphite is then oxidised immediately to potassium sulphate, which has a considerably lower vapour pressure than any of the other potassium salts which could be formed by reacting with the various constituents of the gases present in the kiln.

The resulting fume of potassium sulphate, together with the dust mechanically blown out of the kiln, is carried by the kiln gases into the electrical precipitators, in which a fractionation of the material is accomplished, as outlined above. The commercial result at the California factory has been that a material containing 10%  $K_2O$  is being collected in the two outer hoppers of each division of the main precipitator system, as well as in the multiple pipe treater, and this material has found a ready market in the fertiliser industry of California. It has served to give a profit to the California factory upon the entire precipitation plant investment. The average potash content of the raw mix is about 0.5%, and the volatilisation in the kilns, as ordinarily operated, does not exceed 60% of even this small amount of potash present. The California installation cost \$180,000 and their recoveries per day are 65 tons of dust and 20 tons of potash material (average 10%  $K_2O$ ).

There are still wonderful possibilities for research in the manufacture of Portland cement, including improved grinding machinery, burning of the raw material (which perhaps some day will be done electrically), collection of dust, recovery of potash and other by-products, and the practical control and use of the waste heat from the kilns.

The powdering or pulverising of the raw materials is one of the most important steps in the manufacture of cement. Decided advances have been made in the last few years in grinding and pulverising machines, and types found to be most advantageous in the cement industry are quite novel. Grinding machines nearly all work on the principle of striking or pounding the material between a hammer in some form and a solid metal



mass. The ball mill, for example, is a horizontal iron cylinder, 6 to 8 feet in diameter and 4 to 6 feet long, revolving about its axis 23 to 25 revolutions per minute. This mill is partly filled with steel balls and is lined with steel plates fastened inside the cylinder and arranged in steps. In rolling around the balls fall from these steps on the material, which is fed in at one end, until it passes annular screens fastened to the outer side of the cylinder and revolving with it. The particles too coarse to pass the screen are returned to the grinding chamber through the openings beneath the stepped grinding plates. The output of these machines ranges from 15 to 24 barrels per hour.

Another type of grinding machine is the Griffin mill, which consists essentially of a steel ring or die against the inside of which a heavy steel crushing roll, mounted on a pendulum suspended by a universal joint, is made to roll by centrifugal force. The pendulum is rotated by a pulley and the grinding zone is between the steel outside stationary ring and the revolving pendulum. The output of these mills is about 8 barrels of raw material per hour, to a fineness of 80% through a 200-mesh screen.

A new use for Portland cement, brought about by the war, is at present being rapidly developed with every reason for success, and that is the construction of ships and barges of concrete. Ships of this character have been successfully built recently in Norway, and a 5000-ton ship is at present being constructed in San Francisco, while on November 21st last, a 250-ton ship was successfully launched at Montreal. The American Concrete Institute and the Portland Cement Association have organized Committees which are studying the construction of vessels of this type, and these Committees have designed a reinforced concrete barge of 2000-ton carrying capacity, and have every reason to believe that it will be successful.

The author also gave a brief description of the process of manufacturing Portland cement.

## DISTILLATION TEST OF PETROL.

BY N. A. ANFILOGOFF.

Distillation tests are carried out either for purposes of analysis, for trade purposes, or for manufacturing purposes.

For the purpose of purely analytical work, to enable the chemist to determine the maximum quantities existing within certain predetermined ranges of boiling temperatures, and/or to determine if the petrol consists of naturally consecutive hydrocarbons, or a mixture of volatile and heavy grades of distillate, one cannot do better than follow the methods advised by Professor S. Young, in his treatise on "Fractional distillation," and by employing one or other of Young's dephlegmator columns, accurate results can be obtained. In such analyses of petroleum spirit, I have not found it necessary to use the Young and Thomas evaporator; any of the less severe still-heads, like either the "pear" or "rod and disc" dephlegmators, was found adequate. For accurate work of this description, it would be necessary to introduce corrections of the boiling points for barometric pressure and for the length of the thermometer stem which is not covered by the vapour.

For trade purposes the distillation test is employed to denote the "trade" quality or grade of the petrol. For this purpose the Redwood test is the recognised test employed, but a number of other tests are also used, and as the multiplication of apparatus and methods leads to great confusion and disparity of results, a standard method and apparatus should be devised and employed.

From the manufacturer's point of view, to enable him to decide what grade of petrol and what quantities he can obtain from the crude material, any method would serve, as long as there is some known relation between the apparatus and test employed in his laboratory, and the maximum efficiency of his large scale plant on the works.

It is obvious that the most rational method of testing the petrol for both trade and manufacturing purposes, would be the one which most nearly approaches the best method employed by the analytical chemist, and which would, at a glance, give both the manufacturer and the merchant or the consumer a comprehensive idea of how much distillate it will yield below certain defined temperatures of boiling. Such a method I claim mine to be.

During the years 1902, 1903, and 1904, when petrol commenced to be imported in bulk and the motor industry was being developed at a very rapid rate, heavier grades of petrol were gradually introduced on the market to replace the quondam "680" spirit, which was the only grade supplied for internal combustion engines. Being at that time chemist in charge of the only refinery in this country distilling petrol from crude oil, and being also connected with the landing station where the whole of the petrol destined for this country was landed, I modified the Redwood method of testing to enable the distillation to be carried through more quickly; notwithstanding the fact that my method of testing only took some twenty minutes as against forty to fifty minutes by the Redwood method, the results agreed practically within the limit of experimental error.

At that time the Sumatra spirit, with a sp. gr. of 0.702 yielded some 80% boiling below 100° C., the Rumanian imported spirit with a sp. gr. of 0.700 yielded about 85% boiling below 100°, with a "final boiling" point of 135°, and the Rumanian spirit manufactured at Thames Haven, with a sp. gr. of 0.712, was put on the market to compete with imported lighter grades having 70% boiling below 100° C., with a final boiling point of 145°. Within the years mentioned even the very heavy Borneo spirit imported, having a sp. gr. of 0.774, yielded about 45% boiling below 100°, and had a final boiling point of 165° C. The initial boiling point was not insisted on in years gone by.

The results obtained by my method only commenced to disagree slightly with those of the Redwood method when heavier grades had been introduced and spirit was put on the market which contained less than 20% distilling below 100°, and I maintain that this is due to the initial position of the Redwood thermometer for ascertaining the initial boiling point, which causes, in that method, masking of the presence of the lighter hydrocarbons.

A glance at the appended table will show that, except for the initial boiling point and the percentages below 100°, our tests agree, and where the percentages yielded by the petrol below 100° are more than 15%, the percentages at the trade temperature requirement of 100° and over, agree with those indicated by Redwood's test.

The method described below has been employed consistently at Thames Haven, during the past 15 or 16 years, and has proved particularly advantageous since 1908, when the Thames Haven Petrol Distillery was installed. At this distillery to-day nearly 2,000,000 gallons are distilled per week, and not infrequently 30 or 40 control tests have to be carried out in a day.

My method of testing consists in the use of the regulation Engler flask of 150 c.c. capacity in which 100 c.c. is distilled. The condenser employed is a 24-inch glass tube with an 18-inch water jacket over it, the inner tube preferably being straight, without an enlarged inlet, so as to



avoid having the small pocket of liquid where the narrow tube is sealed on to the short wider tube. I have found a condenser of this length desirable, as though the vapour will be condensed in a shorter condenser, it does not always follow that it will become cooled to room temperature. The thermometer, which is preferably not calibrated for the first four inches above the mercury bulb, is placed in such a position that the top of the mercury bulb is half an inch below the outlet of the side tube. The fact that the calibration does not start at the commencement of the mercury column, obviates the reading of temperatures inside the cork, and therefore obviates moving the thermometer up and down. The moving of the thermometer down nearer the boiling liquid during the progress of the distillation, exposes a certain amount of unheated thermometer stem, on which the vapours condense, lowering the temperature immediately.

When the distillation flask and condenser are connected in the usual way, and the thermometer is placed in the position specified, the flask is heated by the Bunsen burner and the initial boiling point noted as the temperature at which the first drop falls from the thermometer back into the flask due to condensation of vapour. Distillation then continues uninterruptedly at the rate of two drops per second till the bottom of the flask is dry. In this way the whole distillation only occupies 20 minutes, and the position of my thermometer prevents the masking of the presence of lighter hydrocarbons, which undoubtedly happens in the Redwood method.

A slow rate of distillation, as specified in the Redwood method, must obviously give incorrect results when the petrol to be distilled either contains hydrocarbons of very low boiling point or is a mixture of petrol and condensed casing-head gas, which has a notoriously low boiling point. In such a case a considerable percentage of the condensed vapour will evaporate from the receiving measuring cylinder and will therefore give inaccurate readings. It is, of course, impracticable to enclose the receiving cylinder in an ice-chest, to prevent evaporation, as in that case, too, the reading will be inaccurate, since the distillate should be at the same temperature as the liquid before it was placed in the still for testing.

My method thus differs from the Redwood specification, firstly in the initial boiling point, and secondly in the speed of distillation. It is very difficult to define the initial boiling point of a petrol, seeing that the liquid consists of a series

of hydrocarbons of different boiling points, but if the boiling point of a liquid is identical with the condensing point of its vapour, under the same pressure, the initial boiling point of such a liquid as petrol should be the temperature at which the first vapours given off are condensed. It would be impossible to observe this point with the Redwood method where the thermometer is immersed in the boiling liquid, and is only raised to its vapour position with the ascending vapours, in which short time the thermometer has no opportunity to cool sufficiently to register the condensing point of the first vapours passing over.

It is to be noted that, with the exception of the position of the thermometer, the method devised by Ubbelohde and Holde resembles my method and has been adopted by the Deutsche Verband für die Materialprüfung der Technik, for all trade, technical, and customs purposes. In their case the thermometer is placed in such a position that the top of the mercury bulb is just below the side outlet of the Engler flask, and their initial boiling point is the temperature registered by the thermometer when the first drop falls from the tail-end of the condenser. I do not agree with their position of the thermometer, because the slightest variation in the flame, or the slightest draught on the flask, will create a slight vacuum, and the cold air drawn in from the condenser is bound to affect the thermometer readings. This is not so marked in my method, because of the position of the mercury bulb below the outlet. I also do not agree that their initial boiling point temperature is the correct one, because by the time the first drop of condensed liquids falls from the tail-end of the condenser a considerable amount of further vapour has entered the condenser, and the temperature registered is that of the then ascending vapour and not of the vapour which had already condensed.

My method of testing the boiling points of petrol by distillation, like the Redwood method, is quite simple, and requires no special apparatus, and, with a little practice, any intelligent person can get concordant results.

One other claim my method has for consideration by the petroleum trade as a whole is that, except for the very lightest hydrocarbons, results obtained by it are not very divergent from distillation results obtained by the use of an "eight-pear Young's still-head," and also I have habitually controlled my stills, and was able at the refinery to obtain products at least in the same percentages as those indicated in my preliminary distillation.

#### Comparative tests of various trade grades of petrol.

Y = Distillation with Young's 8-pear dephlegmator }  
A = Distillation by Anfilogoff method } % given by volume.  
R = Distillation by Redwood method }

Boiling points.	No. 1 war spirit. Sp. gr. at 60° F. 0.725.			No. 2 war spirit. Sp. gr. at 60° F. 0.730.			No. 3 war spirit. Sp. gr. at 60° F. 0.741.			Bus spirit. Sp. gr. at 60° F. 0.768.			Bus spirit. Sp. gr. at 60° F. 0.765.		
	Y	A	R	Y	A	R	Y	A	R	Y	A	R	Y	A	R
Initial .....	35° C.	40° C.	60° C.	35° C.	40° C.	62° C.	40° C.	45° C.	72° C.	44° C.	60° C.	82° C.	70° C.	75° C.	94° C.*
Below 50° C.	1	—	—	1	—	—	1	—	—	—	—	—	—	—	—
" 60° C.	6	1½	—	6	1	—	3	1	—	—	—	—	—	—	—
" 70° C.	11	4	3	9	3	4	6½	3	—	2	—	—	—	—	—
" 80° C.	17½	15½	14	15½	11	10½	10	6½	6	4	2	—	—	—	—
" 90° C.	30	27½	26	25	20½	20	16	13	12	6½	4	1	3	—	—
" 100° C.	42	41	40½	34	31	31	23	21	21	11½	10	7	9	6	3½
" 110° C.	56	55	54½	45	43	43	33½	30½	31	19½	18	17	33	30	25
" 120° C.	67	67	67	55	56	56	45	41	41	34	34	33½	53	50	51
" 130° C.	75	77	76½	65	67	67	58	53	54	51	54	54	68½	71½	71
" 140° C.	83	84	83½	74½	75	75	68½	65	65	68	73	72	81	84½	85
" 150° C.	88	89	91	81	82½	83	78	76	76	80	86	86	86	92	92
" 160° C.	—	93	93	87	80	80	—	84	84	89	93	93	—	96	96
" 170° C.	—	96	—	—	92½	92	—	89½	89½	—	98	98	—	—	—
" 180° C.	—	—	—	—	94	94	—	93	93	—	—	—	—	—	—
Final .....	—	179° C	179° C.	—	189° C.	189° C.	—	190° C.	190° C.	—	178° C.	178° C.	—	170° C.	170° C.

\* First drop at 94° C., then thermometer fell to 85° before again commencing to rise.

## Bristol and South Wales Section.

Meeting held at Bristol University, on Tuesday,  
November 20th, 1917.

### MODERN PRACTICES IN COAL TAR DISTILLATION.

BY T. HOWARD BUTLER, M.D., M.S.C.

(Abstract.)

The composition of coal tar is so varied that it is impossible to give any general analysis that would be representative; it is dependent on such factors as temperature of carbonisation of the coal, nature of coal used, the shape of retorts, etc. Generally speaking these factors combine together to form a tar that contains a preponderance of paraffins with a low percentage of pitch, or one that contains nearly all ring compounds with a higher amount of pitch and free carbon. At any rate, tar can be looked upon as a very complex mixture, having a sp. gr. of approximately 1.1 to 1.2.

The apparatus used for the distillation of tar varies amongst different distillers, but practically only in detail, such as, size, type of still, materials used in construction, etc. Generally speaking, the still is of a vertical type having a dish bottom, which insures longer life and greater heating surface. The stills are as a rule arranged in a row and are generally of 10—20 tons capacity.

The modern practice in economising heat in distillation is to attach to the still a pre-heater, in which the vapours from the still are condensed by raw tar, which is afterwards used as the still charge on the next time of working.

Many forms of continuous stills have been tried, but have so far not been very freely adopted for the distillation of tar, except in cases where only dehydration of tar is required. One might mention such plants as Hird's, Wilton's, and Lennard's stills, the latter being interesting in so far that it is worked on the principle of fractional condensation and not fractional distillation.

The fractions obtained from the distillation of coal tar are by no means constant, as they depend upon the plant used, and the plant available for working up the products produced.

For the sake of these remarks the fractions may be divided up as follows:—

- |  |              |
|--|--------------|
| (1) Ammonia liquor that distills approximately up to             | 150° C.      |
| (2) Light oil that distills approximately at.....                | 150°—220° C. |
| (3) Creosote oil that distills approximately at.....             | 220°—280° C. |
| (4) Anthracene or yellow oil that distills approximately at..... | 280°—350° C. |
| (5) Pitch.   |              |

*Pitch* from the still at the end of distillation is run into coolers, where it is allowed to remain until cool enough to run into bays or beds. The handling of pitch is a problem of great importance to the tar distiller, as, being approximately 60% of the crude material, the quantity is large; also the danger to workmen in working it is great, the dust therefrom causing temporary blindness. Again men working in contact with pitch for some years have a tendency to a terrible disease known as "pitch cancer."

One of the most successful modern methods of dealing with pitch, is to run it while hot into pans holding about  $\frac{1}{2}$  ton. The pitch is allowed to cool, after which the pans are swung over a railway truck by means of a crane and tipped, the whole block of pitch falling into the truck. By this method the pitch is not handled by men at all; the only objection being a large capital outlay required in pans, light railways, cranes, etc.

*Anthracene oil* is allowed to cool in boilers having some form of slow agitation, when anthracene is deposited. This is filter-pressed, passed through a centrifugal machine, or hot-pressed, when a dark green powder containing about 40% of anthracene is obtained. This can be further purified by means of various solvents, one of the most valuable being pyridine. The purified anthracene finds a valuable use at the present day as the parent substance of alizarin dyes.

*Creosote oil* on being cooled deposits naphthalene in a crystalline form, and on being drained off is ready for the market for use as wood preservative, liquid fuel, etc. The naphthalene so obtained is drained or centrifuged. This product can be purified by washing while melted with sulphuric acid and caustic soda, and is then either sublimed or re-distilled and crystallised.

*Light oil.* This fraction is in many ways the most interesting, especially at the present day, as it contains the tar acids, the most important of which is phenol, from which are prepared picric acid and all the other explosive picrates. Also in this fraction is found benzene, from which synthetic phenol is prepared, and toluene, which is nitrated to the familiar trinitrotoluene (T.N.T.).

It is first necessary to extract from the light oil the tar acids, and the process most freely adopted is the one known as the  $\text{CO}_2$  process. In this the light oils are washed with caustic soda, and the sodium salts of the tar acids are admitted to a cylinder through which carbon dioxide is passed. On being neutralised the free tar acids float to the top, and the resulting sodium carbonate is used for manufacturing a further quantity of caustic soda, by boiling with the lime obtained from a lime kiln, which supplies the carbon dioxide. This process, which works in a cycle, is very efficient and the loss in working is not high. The acid-free light oil is then submitted to washing with concentrated sulphuric acid and caustic soda, and on fractional distillation benzene, toluene, xylene, and heavy naphtha are obtained.

The uses of the various products obtained from the distillation of coal tar were mentioned. Also some of the effects brought about in the industry through War conditions were discussed.

## Edinburgh Section.

Meeting held at Edinburgh on Tuesday, December  
11th, 1917.

MR. D. B. DOTT IN THE CHAIR.

### ANTISEPTICS.

A SHORT ACCOUNT OF WORK DONE FOR THE  
MEDICAL RESEARCH COMMITTEE IN THE  
PATHOLOGICAL DEPARTMENT, EDINBURGH  
UNIVERSITY.

BY THEODORE RETTIE, D.S.C.

In January, 1915, the National Insurance Medical Research Committee, at the request of the War Office, issued an appeal to the various medical schools to institute research on several problems which had been encountered in the medical service at the Front. One of the most urgent requirements was a reliable antiseptic for the treatment of heavily-infected wounds, with special



attention to spore-bearing organisms. Professors Lorrain Smith and Ritchie at once organised several sets of workers to investigate the various problems, under their own direction. The work on antiseptics was carried out by the Professors. Dr. A. Murray Drennan, now Professor of Clinical Pathology in Otago, Dr. W. Campbell, now a Captain in the R.A.M.C., stationed at Alexandria, and myself.<sup>1</sup>

It may seem strange that over 40 years after the introduction of antiseptic surgery by Sir Joseph Lister no ideal antiseptic has been devised for such emergency treatment: the explanation, I think, is to be found in the tendency of modern surgery to leave as much to the recuperative power of the patient's own body as possible. In preparing for operation the surroundings of the patient are rendered as sterile as possible. Instruments, swabs, dressings, everything that will come in contact with him, are sterilised by heat, in fact nothing that is not sterile is allowed to touch him. Under such conditions the only antiseptic necessary is some iodine solution to sterilise the patient's skin, and lysol or such preparation in which to place instruments after use. In accident cases where the wound is already infected, the injured tissues are carefully dissected out under chloroform and the wound cleansed with some strong antiseptic, most often with 5% aqueous carbolic acid, Lister's original antiseptic, or, by some of the ultra-septic surgeons, with large douches of sterile saline (0.85% salt solution)—this is known as normal saline as its osmotic pressure is equal to the pressure of the plasma of the body). Such procedure, though successful with a limited number of patients, obviously cannot be applied in a casualty dressing station; the wounds are always contaminated: they are deep, often ramifying, and may contain pieces of clothing or splinters, all presumably carrying infection. To meet such conditions powerful remedies are necessary, but they must be discriminatingly powerful; microbes which are vegetable cells must be killed, both vegetative forms and spores, which are much more resistant. At the same time the animal cells of the tissues must be damaged as little as possible by the antiseptic; free drainage for all discharges from the wound must also be maintained.

In opposition to the antiseptic method there is what has been called the physiological method of wound treatment, introduced by Sir Almroth Wright. The wound is treated with a hypertonic saline solution, i.e., a solution having an osmotic pressure higher than that of the plasma. As a result of this, fluid is rapidly poured out by the tissues, the idea being that the microbes are thus washed out of the wound and at the same time destroyed by the bactericidal properties of the lymph.

In support of this method the argument was advanced that any antiseptic damages the tissue to which it is applied to such an extent that the value of it as a destroyer of bacteria is lost, and the dead cells and coagulated albumin which the antiseptic leaves in the wound are a fertile source of further trouble. By hypertonic saline treatment this difficulty is avoided. With these two principles in mind, our object was to find an antiseptic agent thoroughly efficient as a killer of bacteria and spores, and at the same time harmless from the point of view of the wound tissues.

Our first step was to test the comparative efficiency of all the antiseptics in general use. It is obvious that it is impossible to make a definite statement as to the value of an antiseptic for wound treatment from its behaviour under laboratory conditions. Most elaborate experimental methods have been devised in the endeavour to fix a standard by which antiseptics may be tested,

but further research has shown that so many factors enter into the efficiency problem that test tube methods have come to be regarded more or less as a compromise. For instance Chick and Martin<sup>2</sup> have shown that what might be called mass action, i.e., the actual number of bacteria exposed to the action of the antiseptic in the test tube, has a very important bearing on the efficiency. The presence of organic matter other than the bacteria has naturally a protective action in favour of the bacteria: various mixtures of bacteria with blood serum, whole blood, pus, muscle extract, etc., have been employed to reproduce as far as possible conditions likely to be met with in a wound. For our tests we decided to use pieces of heavily-infected tissues which were exposed for definite periods to a large volume of the antiseptic solution. The tissue after treatment was washed free of the antiseptic with successive quantities of sterile water, and in cases where it was deemed necessary, any residual antiseptic was neutralised by appropriate chemical methods. The tissue was then put into a tube of sterile broth and incubated: readings were taken at 24, 48, and 72 hours.

The antiseptics tested were those in use at the military hospitals in Edinburgh and others which have been more or less in general use: they were phenol, acrosyl, kymol, chinosol (oxyquinoline potassium sulphate), hydrogen peroxide, mercuric iodide, tincture of iodine, potassium permanganate 4%, methylated spirit, turpentine, salicylic acid, sodium salicylate, methyl salicylate (oil of wintergreen), glycerin, bleaching powder, bleaching powder and hydrogen peroxide (for nascent oxygen), eau de Javelle, boric acid. Our test proved very drastic: of the above only bleaching powder, 10% solution, eau de Javelle (10% sodium hypochlorite), 5% phenol, and the mixture of bleaching powder and hydrogen peroxide had any effect in delaying or inhibiting growth, and the first two were decidedly ahead of the others. The hypochlorites were thus proved, as has often happened before, to be the strongest antiseptics in general use. There are other points decidedly in their favour. Bleaching powder is cheap, easily procured anywhere, and above all it cannot be classed as a dangerous poison. On the other hand pure *Liquor calcis chlorinata* and eau de Javelle are very drastic remedies and on account of their strong alkalinity and high chlorine content (about 3% available chlorine) the tissues will not stand their continued application: for this reason they have never come into general use, though they have both been used with great success on occasion. For instance in 1846 Semelweis, an Austrian physician, stamped out an epidemic of sepsis in his hospital in Vienna by using bleaching powder. Pasteur used *Liquor calcis chlorinata*, and I am informed by Sir James Russell that when he was a medical student in this University Professor Spence constantly used it. Our problem thus reduced itself to getting bleaching powder into a solution or powder that could be applied to open wounds without damaging the tissues unduly.

Following our original method, i.e., with infected tissues, and using mixtures of varying proportions of boric acid and bleaching powder with small quantities of water, we found that with equal quantities of each we got a most pungent smelling paste which had no difficulty in sterilising the tissue and on the other hand, apart from bleaching, did not seem to damage it as much as was expected. We also found that the gas (hypochlorous acid) given off by this mixture was capable of sterilising highly infected tissue, provided it was allowed to act long enough: in some of the experiments two hours was sufficient. This mixture therefore gave promise of high value as a wound dressing, and I shall have occasion to refer to it later. Solutions



prepared from this mixture were next tested and very interesting results were brought out; the bactericidal efficiency of the solution was greatly increased; instead of 3.5% available chlorine, as in *Liquor calcis chlorinatae*, one-tenth of that amount gave very satisfactory results, the free hypochlorous acid proving a much better germicide than the calcium salt.

Further test experiments were carried out with anthrax spores, one of the most virulent and resistant of pathogenic organisms; we were gratified to find that our solution in a strength of 0.35% available chlorine killed the spores in one minute.

Turning next to the action on living tissues, working first on rabbits, then on ourselves, then on patients in local hospitals, we were soon convinced that hypochlorous acid in a solution such as ours could be applied to tissues in a strength hitherto unsuspected. Large quantities of the solution can be applied to extensive wound surfaces, and may be freely introduced into the peritoneum or pleural cavity without producing any toxic effect; indeed the mixed powders may be introduced into wounds and even into the peritoneal cavity without damaging the tissues; such treatment has been found very effective in grossly infected wounds.

Pure hypochlorous acid in aqueous solution always contains free hydrochloric and chloric acids due to spontaneous decomposition, one molecule of hypochlorous acid being oxidised to chloric acid at the expense of other two molecules which are reduced to hydrochloric acid. Both are very strong acids and therefore pure hypochlorous acid is not suitable for wound treatment.

By adding boric acid to chloride of lime in the proportions above stated we have produced a solution containing calcium biborate, an acid salt of extremely low hydrogen ion concentration; on mixing such a solution with one containing hydrochloric acid, the acidity is reduced as the free  $H^+$  ions are taken up by the boric ions forming  $H_3BO_3$ ; the acidity of the solution therefore cannot rise above the dissociation constant for boric acid, which is very low.

By this adjustment various advantages have been secured: the alkalinity of the chloride of lime has been reduced, the full effect of the free hypochlorous acid has been secured, and the solution cannot become unduly acid. In virtue of this balance it follows that the solution can be applied freely to the tissues of the body, and that a considerable quantity can be injected into the circulating blood without harmful effect.

The high germicidal value of pure hypochlorous acid solutions was demonstrated in 1903 by Andrews and Orton.<sup>3</sup> In test-tube experiments they found that very weak solutions of hypochlorous acid, 1 part in 100,000, would kill pathogenic organisms in one minute, but when applied to solutions containing organic matter as well as bacteria they found the hypochlorous acid so rapidly destroyed that they did not evolve any method of applying the solution as a practical antiseptic.

Putting all the above observations together we fixed on the following as safe antiseptics:—

The powder, equal weights of chloride of lime and boric acid. The solution, prepared by shaking up 25 grms. of the above mixture in one litre of water and filtering off the sediment; this solution contains about 0.26% hypochlorous acid. The powder we named Eupad and the solution Eusol—words derived from the initial letters of Edinburgh University Pathology Department.

A simple and convenient method of preparing small quantities of Eusol is to make it up from *Liquor calcis chlorinatae*, that is 10% chloride of lime; this solution, contrary to the statement in the Pharmacopœia, keeps very well if stored in a

cool dark cupboard. I have kept it for months in the laboratory with a very small loss of chlorine. This solution may be made in quantity, say two litres, filtered clear, and the chlorine content determined; the amount necessary for one litre of Eusol is easily calculated; with a good chloride of lime this should be about 125 c.c., which is diluted to one litre, and shaken with 10 grms. of boric acid; the solution remains clear.

For testing we recommend N/10 sodium arsenite solution; this solution keeps better than sodium thiosulphate; it is also better for testing bleaching powder, as chlorates do not interfere with the result, as they do in the hydrochloric acid and potassium iodide method.

Another hypochlorite solution has also been introduced as an antiseptic. It is known as Dakin's<sup>4,7</sup> solution and contains sodium hypochlorite and sodium bicarbonate; its action is much the same as that of Eusol but it is decidedly alkaline.

As Eusol is a most powerful oxidising agent it is evident that its value as an antiseptic will soon be reduced in contact with organic fluids such as are encountered in an open wound; therefore in order to bring any effective solution into contact with the organisms hidden in the depths of the wound, large quantities must be used and the solution got down to the lowest recesses and pockets. To accomplish this, surgeons have various appliances with rubber tubes branching from a common source of supply; this method was applied most successfully with Eusol by Captain Miles<sup>6</sup> in Edinburgh and also by Captain John Fraser<sup>6</sup> in France. A similar method has been advocated by Carrel, who worked with Dakin's solution at Compiègne, and constitutes the Carrel-Dakin<sup>7</sup> method of wound treatment.

We have further observed in open wounds a distinct outpouring of lymph on the application of Eusol, thus combining the advantages of the hypertonic saline treatment with the killing power of the hypochlorous acid; here also the non-toxicity of the solution tells strongly in its favour; there are no toxic by-products at all. Carbolic acid is an excellent antiseptic, but if applied to a wound in unlimited quantity it very soon produces necrosis and may even produce symptoms of general poisoning. The cresols and emulsions containing them precipitate a sticky film of resinous matter in the wound, clogging it and preventing free drainage.

In the course of our preliminary experiments on the effect of Eusol on live tissues we found that large quantities, as much as 40 to 50 c.c., could be injected into the blood stream of rabbits without injuring the animal; following up this line of investigation, with a view to attacking sepsis in the blood, we have met with a considerable measure of success. We applied this method of treatment in the first instance to a case of puerperal septicaemia in the Maternity Hospital in this city.<sup>8</sup> The patient was suffering from an extremely grave form of blood poisoning; the treatment was completely successful.

Following on this, Captain John Fraser, R.A.M.C.,<sup>9</sup> applied the same treatment to soldiers suffering from the acute toxæmia arising from wounds infected with the gas-producing organisms—*Bacillus Welchii*, *B. sporogenes*, etc. These organisms, which cause what is known as gas gangrene, owing to the fact that they produce large quantities of gas inside the tissues, are the scourge of the casualty clearing stations; they are spore-bearers and therefore difficult to kill and the spores are present everywhere.

In certain types of gas gangrene toxæmia, Captain Fraser found intravenous Eusol as strikingly successful as in our first case, but in others it did not seem to have any effect; and this has been the experience of all workers who have



employed the method. Sir Herbert Waterhouse<sup>10</sup> in his report from Anglo-Russian Hospitals, says: "We entertain the highest opinion of its value as a life-saving method in many apparently hopeless cases of septicæmia and pyæmia." This treatment was the subject of much investigation under our own care at the Sick Children's Hospital in Edinburgh. A paper on the subject was published in the "Edinburgh Medical Journal"<sup>11</sup> this summer. As a result, we found that evidence of benefit was recorded in cases of lung infection, such as broncho-pneumonia, empyema, abscess of lung; in toxæmia from appendicitis, and in one case of toxic diarrhoea, also in cases of chronic meningitis. No benefit accrued in cases of rheumatism or in tuberculosis.

The problem we are now engaged on is to find out why in certain bacterial infections we can help the recuperative powers of the body and in others we cannot. We may be acting on a toxin produced by the bacteria and circulating in the blood, or we may destroy some toxic agent formed by the blood itself, or may merely stimulate a protective reaction in the body fluids. Many theories have been advanced as to the conditions found in acute toxæmia. A toxin of protein origin has been held accountable. Again, an increase in the acidity of the blood, due to the production of butyric and kindred acids by the bacteria. A later suggestion by Wright is that the antitryptic power of the blood is reduced; this allows the trypsin to prepare a suitable medium for the growth of the bacteria in the blood itself and the patient is overwhelmed by an acute invasion of the actual organisms. The subject is much too large to enter into in a paper like this. I merely indicate it to show the sort of problems that the chemist is asked to solve in pathological or physiological chemistry.

Take the question of toxins. In a rabbit of 2 kilos, weight there is, say, 100 c.c. of blood. Such a rabbit can stand without inconvenience, say, for a very safe estimate, 20 c.c. Eusol intravenously; this contains 0.05 gm. HClO; obviously 0.05 gm. of hypochlorous acid in 100 c.c. of blood can have no possible action as a direct antiseptic. On the other hand, ricin, a vegetable protein poison extracted from castor oil beans, very closely resembling the bacterial toxins, when administered intravenously to a 2-kilogram rabbit in a dose of 0.001 mgrm., kills the animal. A very small amount of hypochlorous acid would suffice to neutralise this dose, if it could reach it.

Hitherto the treatment of conditions due to organic toxins has been based on the conception of a specific antidote; for example, take diphtheria. The method of treating the disease is to inject into the patient the serum of an animal which has been rendered highly immune to the diphtheria toxin. This serum has the power of neutralising the toxin, but it is a specific power; it cannot neutralise the toxin produced by other organisms, e.g., tetanus. The interest of the method of treatment by intravenous injection of hypochlorous acid lies in the fact that we are able to introduce into the blood a considerable quantity of a strong chemical reagent which will act in a general and not a specific manner.

The chemical reaction between hypochlorous acid and blood is naturally very complex. When hypochlorous acid or hypochlorites act on proteins, the first products are chloramines. In these compounds, which have been studied by Chattaway, Langheld, and later by Dakin, chlorine displaces the hydrogen attached to the nitrogen, giving compounds containing the group NCl. These substances give the reactions for free chlorine and are themselves antiseptics of considerable value. As they are formed in the wound or in the blood stream in intravenous injection, and may continue to exist as such for some time,

they may prove to have an important bearing on the reactions of the body. They ultimately break down to aldehydes, nitriles, carbon dioxide, ammonia, etc. Work on these compounds as antiseptics has been carried out by H. D. Dakin<sup>12</sup> and others working in Professor Cohen's laboratory at Leeds, and two chloramine antiseptics have been prepared and are now in extensive use. They are known as chloramine T, which is *p*-toluene sodium sulphochloramide,  $\text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{NaCl}$ , and dichloramine T, toluene-*p*-sulphodichloramine,  $\text{CH}_3\text{C}_6\text{H}_4\text{SO}_2\text{NCl}_2$ . The former is soluble in water and is used in a strength of 0.5%. The dichloramine is insoluble but dissolves in eucalyptol, which is then diluted with paraffin oil.

In the exigencies of war surgery a large variety of antiseptics, including several synthetic dyes, have been tested, but the general conclusion seems to be that hypochlorous acid, one of the oldest antiseptics, still remains the most reliable for general wound treatment.

In Eusol the full value of hypochlorous acid is available without the drawbacks inherent in the earlier solutions containing this potent reagent.

<sup>10</sup>Lorrain Smith, Drennan, Rettie, and Campbell. B.M.J., 1915, 2, 129.

<sup>11</sup>Chick and Martin. Journal of Hygiene, 1908, 8, 654.

<sup>12</sup>F. W. Andrewes and K. J. P. Orton. Cent. f. Bakt., 1903—4, 35, Abt. 1, pp. 645 and 811.

<sup>13</sup>Dakin. B.M.J., 1915, 2, 318.

<sup>14</sup>Miles. Edin. Med. J., Feb., 1916.

<sup>15</sup>Fraser. B.M.J., Oct. 9th, 1915. Ed. Med. Journal, Mar. 1916.

<sup>16</sup>Sherman. Hypochlorite Solutions for Wound Treatment. B.M.J., 1916, 2, 621.

<sup>17</sup>Lorrain Smith, Ritchie, and Rettie. B.M.J. 1915, 2, p. 716.

<sup>18</sup>Fraser and Bates. B.M.J., 1916, 1, 83.

<sup>19</sup>Sir H. Waterhouse and others. B.M.J. 1917, 2, 441.

<sup>20</sup>Lorrain Smith, Ritchie, and Rettie. Ed. Med. J., Sept., 1917.

<sup>21</sup>Dakin and others. Proc. Roy. Soc., 1916, B, 89, 232, and B.M.J., 1917, 1, 865.

For clinical trials with Eusol see Lancet., Feb. 5 & 12, 1916.

## London Section.

Meeting held at Burlington House on Monday,  
January 7th, 1918.

DR. CHARLES A. KEANE IN THE CHAIR.

## THE TOXICITY OF METHYL ALCOHOL IN RELATION TO ITS INDUSTRIAL USES: A REVIEW OF THE PUBLISHED DATA.

BY THOMAS D. MORSON.

### Introduction.

In the course of an investigation into the question of the use of methyl alcohol and methylating agents generally for various industrial purposes, the author found it necessary to have a complete search of the literature carried out. It soon became evident that a mass of information was available as to the toxic effects of methyl alcohol. The references are distributed through a wide range of scientific publications, and these have been tabulated and in some cases extracted. The value of the compilation will be appreciated mostly by those who have previously attempted to supply evidence that methyl alcohol is a poison. The work has been carried out with a definite object, concerning which it is sufficient to say here that pure methyl alcohol is classified by the Customs and Excise authorities as "plain spirits" and has been subjected to the same rates of duty as pure ethyl alcohol.

The origin of the classification of pure methyl alcohol as "plain spirits" is not clear, but it doubtless arose from the assumption that this article was or might be regarded as potable and capable of substitution for purposes for which

pure ethyl alcohol was used. It is reasonable to suppose that if it can be proved definitely that this "plain spirit" is highly toxic the main argument for taxation on the grounds of potability falls to the ground. If, in addition, it can be shown that pure methyl alcohol is the starting point for many manufactures of the greatest national importance, one may hope that unnecessary restrictions will no longer be allowed to stand in the way of the development of British chemical industry.

These are the broad lines upon which action may be taken. I have only to add that to-day British chemical manufacturers, who are interested in this question, are both united and determined. Restrictions which have proved an unfair and unjustifiable burden on the British manufacturer, must be removed, or modified, to enable us to compete successfully with foreign rivals in the great industrial struggle that lies before us.

#### *Production of methyl alcohol.*

Supplies of methyl alcohol before the war were obtained from the United States, Canada, and France, but chiefly from Germany and Austria. It is obviously desirable that the British Empire should be self-supporting in this respect. The following are the views of a prominent British wood-distiller:—

"Before British chemical manufacturers can hope to compete successfully with the foreigner in the manufacture of dyes and fine chemicals used in the arts and for medicinal purposes, it is essential that they be able not only to produce these substances cheaply and in a high state of purity, but also that they should be independent of the foreign manufacturer for the chemicals required in their production—otherwise it will be in the power of the foreign manufacturer to limit the extent of the expansion of the British industry. In this connection, the products of wood-distillation are second only in importance to the products of what might be termed the sister industry—coal tar distillation.

"There is immediate necessity for encouraging the expansion of the wood-distillation industry, the present decadent condition of which is due largely to the fact that wood distillers, unlike their foreign competitors, could find no market for their methyl alcohol owing to the heavy restrictions imposed upon it by the Government. These restrictions not only hinder the expansion of the wood-distillation industry by, and to, the limited demand for wood spirit as such, for denaturing purposes and varnish making, but also the greater industry of dyes and fine chemicals, upon which the wood-distillation industry depends for an outlet for its products. The result is that methyl alcohol (pure) is not produced in this country to any extent—if at all—while of the other necessary products of wood distillation only a mere fraction of this country's pre-war requirements of acetone, acetic acid, and the acetates, was produced at home."

Another manufacturer makes what is, in my opinion, a very valuable suggestion. He proposes to replace the denaturant of "methylated" and "industrial" alcohol by a cheaper and equally efficacious substance. By so doing, the whole of the crude methyl alcohol produced in this country could be rectified to pure methyl alcohol for essential industrial purposes.

The importance of conserving supplies of methyl alcohol cannot be over-stated. To-day it is little short of criminal folly to waste (I use the word deliberately) wood alcohol for denaturing purposes.

#### *The importance of pure methyl alcohol in industry.*

In support of the claim that methyl alcohol is,

in effect, a "key" chemical, it will suffice to give a very brief survey of its main uses. In this connection I have to acknowledge with grateful thanks the assistance of a number of my fellow-manufacturers, who have sent me useful information and practicable suggestions. Much information is available from the Minutes of Evidence taken before the Departmental Committee on Industrial Alcohol and still more from the papers by T. Tyrer read before this Society in March, 1903, and March, 1904.

The position has materially altered since then owing to the outbreak of War, and it is mainly from this point that I review the question under the above heading.

#### *Industrial uses of methyl alcohol (pure).*

In further support of the claim that methyl alcohol must be regarded as a "key" chemical it will suffice to summarise as follows:—

*Manufacture of dyestuffs.*—Methyl alcohol (pure) is absolutely essential for the production of a vast range of dyes containing methyl groups, and is necessary also as a solvent.

Dyestuffs and intermediate products can be imported into Great Britain free of duty and the methyl alcohol which they contain (as methyl groups) pays no tax.

*Manufacture of medicinal chemicals.*—The necessity for using a methyl alcohol as pure as possible in this class of manufacture is vital both from the therapeutic and economic standpoint. The purposes for which it can be used may be divided into:—

(1) Where the alcohol is essential for the process in question.

(2) Where it possesses advantages over industrial methylated spirits or other duty-free solvents.

The replacement of hydrogen atoms by methyl groups, which is an essential feature of the preparation of a number of drugs and fine chemicals, usually necessitates the use of the methyl ester of an inorganic acid. In the preparation of methyl salicylate, for example, the methyl alcohol and salicylic acid are made to react in presence of a mineral acid and, whilst absolute purity of the methyl alcohol is not essential, the purer the alcohol employed the less trouble will be experienced in the purification of the final product. In cases where a hydroxyl group has to be converted into a methoxyl group, or methyl added to tertiary nitrogen (as in the preparation of phenazone) the use of a methyl ester of a strong (generally mineral) acid is unavoidable. For purposes of this character the following methyl esters are available:—Chloride, bromide, iodide, nitrate, toluene, sulphonates, picrate, sulphate, and occasionally ester-salts such as sodium methyl-sulphate.

In preparing any of these compounds impurities in the methyl alcohol are disadvantageous but do not greatly hinder the preparation of the chloride, bromide, or iodide. These three compounds are, however, of little use for other reasons.

The most convenient methylating agent known is methyl sulphate. Its great reactivity and high boiling point (188°C.) generally render the use of autoclaves unnecessary. In the preparation of methyl sulphate, methylsulphuric acid is first produced and subjected to distillation. Unless methyl alcohol of high purity is employed, the methylsulphuric acid is dark in colour, contains tarry substances, and chars badly on distillation. Both the yield and purity of the methyl sulphate are affected adversely, and whilst a certain amount of the impurity can be removed by distillation, complete removal of the condensation products derived from the impurities in the methyl alcohol is almost impossible. In the case of salts of methylsulphuric acid, unless pure



methyl alcohol is used the impurities cling to the salts and their purification by repeated crystallisation is impracticable owing to the ease with which they decompose in solution. The use of impure methyl alcohol in preparing methyl sulphate or its salts is very wasteful.

The manufacture of many medicinal chemicals in this country is dependent on the use of pure methyl alcohol free from revenue restrictions.

*Manufacture of formaldehyde and its derivatives.*—Formaldehyde forms the starting point of a number of syntheses. The products are largely used in medicine (e.g., hexamine) and the arts (e.g., synthetic resins). The manufacture must be carried out with pure methyl alcohol.

*Manufacture of photographic chemicals.*—Only pure methyl alcohol is suitable for this class of preparation. Previous to the war large quantities of these were made in Germany and at such prices that it could only have been possible with unrestricted duty-free methyl alcohol. It will be necessary that the use of pure methyl alcohol, duty free, shall be made easy, subject to certain unhampering conditions of liability and returns as to manufacture, if these photographic chemicals are to be produced in this country in quantity and at a price to compete with the German productions.

The foregoing statements will have sufficed to emphasise two facts:—(1) The importance of pure methyl alcohol in chemical industry. (2) The economic difficulties which hinder the development of manufactures requiring its use.

Before passing to the main heading of this paper I would like to quote the following extract from the Report of the Departmental Committee on Industrial Alcohol (1905):—

*"Methylic alcohol does not fall within the charge to spirit duty in Germany and may be used freely for industrial purposes without control by the revenue authorities."*

#### *The toxicity of methyl alcohol.*

So far as I am aware the toxicity of methyl alcohol has remained up to the present a disputed question in this country.

In addition to searching the literature I have endeavoured to obtain first-hand evidence from cases actually occurring here. Owing to the comparatively restricted use of pure methyl alcohol in this country, for reasons already referred to, experiences are not easily obtainable. One manufacturer, however, reports as follows:—

"I have never experimented with it personally and do not intend to do so, as I have been aware of its deadly poisonous action from experiences gained in foreign aniline colour works years ago, where workmen, known dipsomaniacs, have taken it as a potable spirit with deplorable results, when taken in only minute quantities. I have, however, had experiences in my own works of the influence of its vapours and have found a complete derangement of the mental faculties, together with a loss of vision, which, I believe, would have produced a state of coma if the surrounding conditions had been prolonged. Some of my workmen suffered in a similar manner, some more acutely than others, dependent, no doubt, upon the density of the vapours. But all were affected similarly.

"For a concrete case of poisoning which occurred at the Docks of the Manchester Ship Canal last year, when three men lost their lives through taking some methyl alcohol drained from empty drums and adding same to their tea, I must refer you to the report of the inquest before the Salford Coroner. The evidence was conclusive enough and corroboration can be found in foreign publications dealing exhaustively with the impossibility of regarding methyl alcohol otherwise than as a deadly poison."

In America the poisonous properties of methyl alcohol have been very thoroughly investigated. The results are collected in a pamphlet entitled "Blindness from Wood Alcohol," by Dr. Casey A. Wood, published in 1914 by the American Medical Association. The conclusions arrived at may be summarised to this, that methyl alcohol is a deadly poison and it should only be available to the general public under the restrictions usually applied to poisonous substances. The report is so complete and probably so little known in this country that I may be pardoned for quoting extracts somewhat extensively.

"In the years 1903—1904 the late Dr. Frank Buller of Montreal and myself set about collecting the histories of cases of poisoning to that date. Doubtless many instances of wood-alcohol poisoning escaped this inquiry, but we found and reported 275 instances of death or blindness (sometimes both) directly traceable to drinking, or inhaling the fumes of "Columbian Spirits" or some other form of "deodorized" wood alcohol. At that time we drew the following conclusions:—

1. Methyl or wood alcohol, in any of its forms, and all methylated preparations as well, are dangerous poisons, menacing both life and eyesight.

2. It is used as an adulterant of, and substitute for, grain alcohol in cheap whisky and other alcoholic beverages, not to mention Jamaica ginger, lemon extract and many other essences and flavouring fluids.

3. Methyl alcohol is largely used in the preparation of many proprietary and patent medicines, witch hazel, domestic liniments, as well as hay rum, Cologne water, Florida water and other perfumes.

4. The injury to the eyesight consists chiefly of a destructive inflammation of the optic nerve or of the retina (or both) followed by their atrophy.

5. The symptoms of acute poisoning are disturbances of the stomach more or less severe, accompanied by abdominal pain, general weakness, nausea, vomiting, dizziness, headache, dilated pupils and blindness. If recovery does not occur, there is marked depression of the heart's action, sighing respiration, cold sweats, delirium, unconsciousness, coma and death.

6. The blindness affects both eyes and may set in a few hours after the contact with the poison, or it may be delayed for several days. It is generally complete, with a subsequent improvement and, finally, a relapse into permanent blindness.

7. The diagnosis can hardly be mistaken. Methyl alcohol poisoning presents a picture unlike that of any other intoxication. Acute abdominal distress, followed by blindness, should always awake suspicion of methyl alcohol poisoning.

8. The prevention of poisoning by this insidious drug can only be brought about by prohibiting (or rendering unprofitable) the sale of "deodorized" wood alcohol in all its forms. The number of deaths may, meantime, be limited by putting all preparations containing wood alcohol on the list of poisons and prosecuting all persons adulterating foods and drinks with it. Labelling it with the notice, "This fluid taken internally is likely to produce blindness," will certainly have a deterrent effect.

9. Wood alcohol intoxication is an example of idiosyncrasy. As in the case of several other poisons, some persons are largely immune so far as permanent damage to the body is concerned. If ten persons drink, say 4 ounces of Columbian spirits within three hours, all will have marked abdominal distress and probably four will die, two of them becoming blind before death. Six will eventually recover, of whom two will be permanently blind. With still larger doses, the proportion of death and blindness will be greater.

Commenting on the death of four men and the blindness of one man in Whitestown, Indiana, about August 16th, 1911, Dr. P. B. Little of that town writes me:—

"It seems that these men purchased and made a mixture of one gallon of wood alcohol and 3 gallons of grain alcohol and all drank freely of it. The symptoms that followed soon after were chiefly headache, nausea, vomiting, extreme weakness, clammy sweats, weak pulse, blindness, dilated pupils, cyanosis, sighing, respiration, convulsions and death. The blindness came on six to eight hours before death. The survivor of this debauch was the first to fall sick and the first to become blind."

**References.**—Journal of the American Medical Association, December 7th, 1912, page 2075; May 10th, 1913, page 1479; March 18th, 1916, page 913.

We may revert now to my claim earlier in this paper that methyl alcohol is highly toxic and for that reason the Revenue Authorities should have no fear that it can be consumed as "potable spirit." *Then why tax it as "plain spirits"?* Why penalise British chemical industry by taxation of a "key" chemical? One may suppose that if methyl alcohol had been called by some other less alcoholic name it would never have been classified as "spirits" at all.

During the inquiry of the Departmental Committee on Industrial Alcohol (1905) the question of the toxicity of methyl alcohol never seems to have been raised. There is, moreover, a curious omission in the Report of the Sub-Committee that visited Germany. They make no comment on their own statement that methyl alcohol in Germany is free from all Revenue control.

Yet to the British Revenue Authorities methyl alcohol is "spirit." Why could Germany afford to ignore methyl alcohol as being "potential whisky?" This same Sub-Committee enthused over the "pre-eminence long enjoyed by Germany in the manufacture of pharmaceutical products—fine chemicals."

Does the Sub-Committee enlighten us on the use the German fine chemical manufacturer made of the "spirit" which was for him really duty-free? Did they ask if the German-made alkaloids were crystallised from pure methyl alcohol over which there was no Revenue control or from ethyl alcohol over which the supervision was strict and costly? If they did the reply is not printed.

I suggest that henceforward pure methyl alcohol should be known by its scientific name of "Methanol" and that it be labelled "Poison."

As a further safeguard let the Customs Board be authorised to sanction its use under the same conditions that apply to-day to the use of industrial alcohol and free from all further taxation and Revenue control.

With these recommendations and the following references to the literature on the toxicity of methyl alcohol I close my case.

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#### ABSTRACTS OF LITERATURE REFERENCES.

POINCARE. (Compt. rend., **87**, 682.) *Danger of employing methyl alcohol in certain industries.* Some animals which had remained from 8 to 16 months in an atmosphere continually renewed, but charged with a certain quantity of methyl alcohol vapour, were afflicted with great increase in size of the abdomen, together with other serious symptoms. On examination after death, there was found to be considerable hypertrophy of the liver, which filled the greater part of the abdominal cavity: a fatty degeneration of this organ to an extent that could be scarcely exaggerated: an alteration of the constitution of the muscular fibres of the heart, of the epithelial cells, of the uriniferous tubes, and of a large number of the cells of the lungs.

POHL. (Chem. Centr., 1893, ii., 380—381; from Arch. Expt. Path. Pharm., **31**, 281—302.) *Oxidation of methyl and ethyl alcohols in the organism.* Ethyl alcohol administered to a dog produced protracted sleep, and when this passes off, the dog awakes in a normal condition. Methyl alcohol, on the other hand, produces restlessness, giddiness, and then broken sleep; and the effects of a dose last for three or four days. A dog may be dosed without harm for a year with ethyl, isobutyl, or amyl alcohol; but with methyl alcohol death ensues in a few weeks. In the case of poisoning by methyl alcohol, formic acid appears in the urine, and reaches a maximum on the third or fourth day.

BOKORNY. (Pflüger's Archiv, 1897, **66**, 114—144.) *Carbon compounds as food material for bacteria.* Methyl alcohol appears to be a fairly good source from which bacteria can obtain their carbon: ethyl and amyl alcohols can also be used.

MULLER. (Z. angew. Chem., **23**, 351—5.) *Methyl alcohol poisoning.* A lecture delivered before chemists.

LEWIN. (Chem. Zentr., 1911, i., 672; from Apoth. Zeit., 1911, **26**, 54—5.) *The toxic action of methyl alcohol.* The author gives many instances of the extraordinary toxicity of methyl alcohol which follows both inhalation and ingestion through the stomach. Of sixty-three hens' eggs, after injection of ethyl alcohol, nine did not develop at all; twenty-two developed monstrosities; after injection of methyl alcohol, nine did not develop, whereas forty-one gave rise to monstrosities.

FORSTER. (Bied. Zentr., 1911, **40**, 431; from Z. Spiritusind., 1910, **33**, 2.) *Action of methyl alcohol in the organism.* The stronger physiological action of methyl alcohol as compared with ethyl alcohol is perhaps due to impurities hitherto not detected.

SABBATANI. (Pharm. Inst. Parma. Arch. fisiol. (Fano's Festschrift.), **7**, 49—80; through Zentr. Biochem. Biophys., **10**, 621—2.) *In what manner do ethyl and methyl alcohol influence the molecular concentration of the blood and the organs?* The value of the ratio of the change in  $\Delta$  of the blood after the intravenous injection of EtOH, MeOH and NaCl to the minimal dose was found to be 6.4, 6.2 and 7.3 respectively. The cryoscopic value varies from normal in the organs, being very evident in the brain, evident in the kidney, and less pronounced in the heart. The muscles gave only a very slightly higher value, while that of the liver was lowered.



The electrical conductivity was lowered by alcoholic injections and increased by NaCl. A physico-chemical discussion of the results is given.

AXON. (Pharm. Zentralhalle, **52**, 335—6.) *Toxicity of methyl alcohol.* In view of the fact that 8 grms. of MeOH was found to affect the eyesight seriously and 15—30 times as much in one dose would kill, the introduction of MeOH into foods should be carefully guarded against.

HORACEWSKI. (Oesterr. Chem. Ztg., **14**, 139—41; through J. Soc. Chem. Ind., 1911, 826.) *Injurious effect of alcohol denatured with wood spirit on workers' health.* The use of wood spirit should no longer be permitted for denaturing ETOH because the health of those working with such denatured ETOH is liable to be seriously affected by the impurities contained in the wood spirit together with the greater toxicity of the MeOH itself in comparison with ETOH. The use of shellac is advocated as a denaturing agent for alcohol for polishing and lacquering industries.

CORNALBA. (Boll. Chim. farm., **51**, 37—38.) *The toxicity of methyl alcohol.* Warning is issued against possible use of  $\text{CH}_3\text{OH}$  in pharmaceutical products. Cornalba notes authorities who claim that  $\text{CH}_3\text{OH}$  is not toxic, but himself leans to those who assert its poisonous properties. Method of detection of  $\text{CH}_3\text{OH}$  in  $\text{C}_2\text{H}_5\text{OH}$  is given according to Riche and Bardy, modified by Jorissen and Torickanoff, as follows: After formation of methyl-amine iodide, the excess of aniline is eliminated by shaking with ether. Fractional distillation is then employed, the first fractions are collected and dehydrated with dry  $\text{K}_2\text{CO}_3$  and the boiling point detd.  $\text{CH}_3\text{OH}$  boils 13° below the b.pt. of  $\text{C}_2\text{H}_5\text{OH}$ .

FRANCESCHI. (Giorn. farm. chim., **61**, 145—52.) *A supposed poisoning by methyl alcohol and its chemical and toxicological research.* Résumé of poisonings by alcohol—Methyl, butyl, amyl—is given, as well as opinions of various authorities as to relative toxicity. Special notice is given to large numbers of deaths of inmates of a Berlin municipal lodging-house, due to drinking spirits made with wood alcohol. Details of technic are given, together with an outline of work planned to show relative toxicity of ETOH and MeOH on animals. Other chemicals, such as methylene compounds, formic ether, etc., will be tried and reported on later.

SCHMIDEBERG. (Therap. Monatsh., **26**, 329—31.) *Methyl alcohol poisoning.*

THOMAS TYLER AND F. G. GOSLING. (Yr.-bk. of Pharm., 1912, 434.) *Notes on commercial formates and other compounds. Methyl alcohol.* The toxicity of this alcohol is now placed beyond doubt. Attempts to ascribe the toxic quality to other constituents of commercial methyl alcohol and wood naphtha have failed. Undoubtedly methyl alcohol is itself toxic, as well as some other constituents of the commercial methyl alcohol.

HARNACK. (Deut. med. Wochschr., **38**, 358—61.) *The poisonous properties of methyl alcohol.* MeOH, compared with other alcohols, acts less strongly. Its danger lies in the slow oxidation to  $\text{HCOOH}$  which occurs in the nervous tissues having a specific affinity for the alcohol.

MENDEL. (Berl. oph. Ges., Jan. 25th, 1912; Zentr. Biochem. Biophys., **12**, 941.) *Blindness caused by methyl alcohol.* Various cases of blindness are recorded.

VOLTZ AND DIETRICH. (Biochem. Zeitsch., 1912, **40**, 15—28.) *The part played by methyl and ethyl alcohols in the general metabolism of the animal organism.* The amount of methyl alcohol excreted in the expired air and in the urine was estimated. It was found that after administration of 2 c.c. of methyl alcohol per kilo. of the body weight to a dog, 24.3% was excreted in forty-eight hours, of which 21.5% was in the expired air, and 2.8% in the urine. As 36.8% was found in the body, it follows that only 39% was oxidised in the body. If the caloric value of the methyl alcohol oxidised is calculated, it will be found that this represents only 3% of the total metabolism of the body. These results are in marked contrast to those obtained with ethyl alcohol under analogous conditions. In this case 2 to 4% of the quantity administered was found in the expired air and 0.4 to 3.8% in the urine within 10 to 15 hours. After 15 hours only 3 to 12% was found in the body. Of the food-stuffs degraded within 10 hours, the ethyl alcohol (calculated on the caloric values) formed 42% and in 15 hours, 35%.

BUCKENACK, PRAUSE, GRIEBEL, JACOBSEN AND GAZA. (Z. Nahr. Genussm., **24**, 7.) *The Berlin methyl alcohol poisoning.* 70 deaths and 95 cases of sickness in Berlin were shown to have been due to methyl alcohol contained in brandy in the relation of 80 methyl to 20 ethyl alcohol. In the qualitative test for methyl alcohol the method of Fendler and Maunich (Apoth. Ztg., **20**, 569) was used. For the quantitative determination resort was made to the determination of the carbon present, the difference between the carbon content of ethyl and methyl alcohols being 14.68%. The presence of methyl alcohol was also demonstrated in the dead body, and  $\text{HCO}_2\text{H}$  was likewise found.

NICLOUX. (Orig. Com. 8th Intern. Congr. Appl. Chem., **19**, 259—60.) *Determination of methyl alcohol in the blood and tissues.* Method based on oxidation with  $\text{K}_2\text{Cr}_2\text{O}_7$  and determination of the resultant  $\text{CO}_2$ . The ratio  $\text{CO}_2/\text{O}_2$  is specific (0.915 part methyl alcohol). The figure obtained in the experiments coincided with this ratio.

WHITNEY. (Amer. J. Physiol., 1912, **30**, 463—465.) *The relative toxicity of methyl and ethyl alcohols as determined by the rate of reproduction in Hydratina senta.* Methyl alcohol is less toxic than ethyl alcohol, as determined by the rate of reproduction in the rotifer, *Hydratina senta*. Families subjected to 1% methyl alcohol during many (10 to 15) generations recover from its influence in the second generation after the alcohol is removed.

RUHLE. (Münch. med. Wochenschr., **59**, 964—7.) *Findings in the central nervous system of experimental animals after methyl alcohol poisoning.* Two dogs were killed acutely by C.P. methyl alcohol (one animal was given 450 c.c. in 4 hours and died in 38 hours; the second, 250 c.c. in 24 hours and died in 18 hours). All the preparations examined revealed scattered hemorrhages around blood vessels in the pons, the medulla oblongata and the

spinal cord, partly lodged between the medullated fibres and partly between the ganglion cell groups. Preceding these perivascular hemorrhages were seen leucocytic accumulations and loading of the vascular endothelium with lipoids.

TYSON. (Arch. Ophth., 1912; Zentr. Biochem. Biophys., **15**, 205.) *Amblyopia from inhalation of methyl alcohol.* Peculiar anomalies in the field of vision are described.

GRIGNOLO. (Klin. Monatsbl. Augenheilk., **51**, 157; Zentr. Biochem. Biophys., **14**, 889.) *Biochemical changes in the aqueous humor in acute methyl and toxipeptide poisonings.* Osmotic pressure in the aqueous humor increases after methyl alcohol poisoning, while  $\text{H}^+$  remains unchanged and the  $\text{H}^+$  concentration is only slightly modified. In peptone poisoning, osmotic pressure,  $\text{H}^+$  and  $\text{H}^-$  concentration experience no change.

KASASS. (Inaug. Diss., St. Petersburg, 1912; Zentr. Biochem. Biophys., **15**, 205.) *Pathology of methyl alcohol amacrosis.* Methyl alcohol causes blood extravasation in the sheaths of the optic nerve and choroid, thus producing circulatory disturbances; through incomplete combustion of methyl alcohol formic acid collects in the vessels and causes fatty degeneration. These two factors, together with oedema, lead to degeneration of the retina and consequently to rapidly ensuing blindness. If the collateral circulation be restored, oedema disappears and visual power partially returns. Central scotoma depends on axial neuritis while diminution of the field of vision is due to affection of the peripheral end of the optic nerve.

SEGALE. (Accad. med. Genova (session Feb.), 1912; Zentr. Biochem. Biophys., **14**, 446.) *Acute experimental intoxication due to methyl alcohol and differential biochemical diagnosis of acute poisoning of the toxipeptide type.* The author reviews his previous investigations of poisoning by toxipeptides. It is possible from the refractometric data and by means of the determination of the  $\text{H}^+$  ion concentration of serum to differentiate toxic diseases from methyl alcohol poisoning, which has practically the same symptomatology.

NICLOUX AND PLACET. (J. physiol. path. gen., **14**, 916.) *Toxicity of methyl and ethyl alcohols.* Large intravenous doses of methyl alcohol are relatively less toxic than ethyl alcohol. The greatest alcohol retention is found in brain tissue. In repeated small doses every 24 hours, methyl alcohol is more toxic than ethyl alcohol. This appears to be due to the cumulative effect obtained since methyl alcohol is eliminated more slowly and is less readily destroyed in the tissues than ethyl alcohol.

BUTGER. (Chem. Ztg., **36**, 524—5.) *Methyl alcohol poisoning.* A review of the history, chemistry, pharmacology, and toxicology of MeOH, and a discussion of laws relating to its manufacture and sale.

LANGGAARD. (Chem. Zentr., 1912, ii., 1382—3; from Berl. klin. Woch., 1912, **49**, 1704.) *The poisonous nature of ethyl and methyl alcohols.* Methyl alcohol is more poisonous than ethyl alcohol when taken in repeated small quantities, but ethyl alcohol is much more dangerous when taken in a single large dose.

MURA. (Biochem. Zeits., 1913, **49**, 144—151.) *The action of methyl alcohol on the circulating blood.* In 2 out of 5 experiments (on 4 rabbits and 1 dog) injection of methyl alcohol produced anaemia. There was observed a diminution of lymphocytes and an increase in the number of pseudoeosinophile or neutrophile corpuscles and also haemoglobinuria. The methyl alcohol appears to act toxically on the blood-forming apparatus.

FRANCESCHI. (Boll. chim. farm., **51**, 325—9.) *A suspected poisoning by methyl alcohol and the chemical-toxicological investigation of the same.* A detailed discussion of the chemical-toxicological examination made on the body of C. Franchini, a victim of wood alcohol poisoning.

BOESEKEN AND WATERMAN. (Chem. Weekblad, **9**, 694—6.) *Remarks concerning the poisonous properties of methyl alcohol.* Experiments with several varieties of moulds showed that MeOH as a source of carbon is less suitable as a food for the moulds than the corresponding ethyl alcohol derivative. This is not due to physical properties like solubility, but to chemical properties, possibly to the conversion of the methyl alcohol derivative to formaldehyde which is a poison, while the ethyl alcohol may yield only the relatively harmless, easily polymerising acetaldehyde.

HIRSCHBERG. (Berl. klin. Woch., **49**, 245—51.) *Methyl alcohol whisky poisoning.* The author discusses the clinical progress of methyl alcohol poisoning, citing an extensive number of cases, and urges in conclusion a more careful supervision of methyl alcohol whisky dispensation.

SEGALE. (Berl. klin. Wochschr., **50**, 255—6.) *The biochemical differential diagnosis between toxipeptide and methyl alcohol poisoning.* In acute poisoning with toxipeptides the osmotic pressure, the ionic concentration and the refractive index are increased, the electrical conductivity is little changed, and in acute poisoning with methyl alcohol the osmotic pressure becomes high, the concentration of the ions is unchanged,  $\text{H}^+$  is reduced, and the electrical conductivity is little affected.

OLIVARI. (Chem. Zentr., 1913, i., 1780; from Arch. Farmacol. sperim., 1913, **15**, 83—96.) *Poisoning by methyl alcohol.* The toxicity of crude and pure methyl alcohol for frogs, mice, and guinea-pigs was investigated. In the case of the pure product, the minimal lethal doses were: for frogs, 10.2%; for mice, 11.5%; and for guinea-pigs, 9.5% of the body weight; the corresponding numbers for the crude acid product were 10.0, 7.5, and 6%; and for the crude basic product, 8.6, 6.8, and 5.5%. A scheme is given for the forensic examination of drinks and animal organs in which wood-spirit is suspected. The liquid is acidified with phosphoric acid, and the volatile acids, etc., are distilled off, whilst the bases (pyridines, amines, etc.) remain in the residue. The distillate containing the acids is neutralised, and again distilled, the salts of the acids remaining behind, the aldehydes, etc., distilling over. These are fixed by *m*-phenylenediamine hydrochloride and the mixture is distilled, when acetone and methyl alcohol distil over, and are estimated in the distillate. If the



mixture is saponified before these treatments, a conception can be formed as to the quantity of esters present from the amount of alcohol in this distillate. Finally, ethereal oils can be extracted by mineral oils (b. pt.  $140^{\circ}$ – $230^{\circ}$ ), and separated from the higher alcohols by fractional distillation.

NICLOUX AND L'ACET. (Compt. rend. soc. biol., 73, 63–6.) *Comparative toxicity and elimination of methyl alcohol and ethyl alcohol.*

LOWEY AND VON DER HEIDE. (Mochem. Zeitsch., 1914, 65, 230–252.) *The intake of methyl alcohol in inspiration.* Rabbits and dogs were exposed in a respiration chamber to air containing various quantities of methyl alcohol vapours. These amounts could be regulated by passing air through methyl alcohol in various dilutions in water. The air was measured by a meter, and the amount of alcohol was estimated at the beginning and end of the experiment. The toxic effects were noticed, and the alcohol in the animal bodies was estimated at the end of the experiment. In presence of only 0.2% of alcohol in the air, quite appreciable quantities were taken up by the organism. With alcohol of this tension, and up to 0.5%, the saturation of the animal body is reached in 2 hours. With higher concentrations, the times required for saturation are markedly longer, and when the tension reached 2%, the maximal amount is not taken up even after 8 hours. Fat animals under similar conditions take up less alcohol than lean ones. This is due to the small solubility of lipids in methyl alcohol; the coefficient of distribution between oil and water is about 2½:100.

ASON. (Pharm. Notisbl., 1914, 23; Apoth. Ztg., 1914, 639; Pharm. J., 93, 349.) *Determination of methyl alcohol and formaldehyde in urine.* To 10 g. urine add 2 g. pure  $\text{H}_2\text{SO}_4$ , cool, add about 0.7 g.  $\text{KMnO}_4$ , enough to leave a slight rose tint after filtering. To 1 g. of filtrate add 4 g. pure  $\text{H}_2\text{SO}_4$ , cool, then pour on it a solution of 0.05 g. morphine hydrochloride in 3 g.  $\text{H}_2\text{SO}_4$ . A violet-coloured zone will indicate  $\text{MeOH}$  or  $\text{HCHO}$ , up to a dilution of 1:5000  $\text{HClO}$ .

TOERSCHEIMER AND VERZAR. (Arch. Augenheilk., 75, 27; Zentr. Biochem. Biophys., 15, 942.) *Pathogenesis of methyl alcohol and atoxyl amblyopia: experiments on the pathology of the perception of light.* Hens tolerated large amounts of 20–25% methyl alcohol; general symptoms of poisoning with the appearance of inebriation were observed. The retina showed no noteworthy alterations; severe lesions were produced by higher concentrations of methyl alcohol. Either a transitory or a gradually increasing diminution in the sense of light was observed; it was not ascertained whether such modification was due to changes in the retina or to cerebral fatigue. Atoxyl (5%) produced symptoms of poisoning of the central nervous system in hens. The structure of the cells of the inner retinal layers was altered; the sense of light was not affected.

NICLOUX. (Compt. rend. soc. biol., 73, 59–62.) *Determination of small amounts of methyl alcohol in the blood and tissues. Determination of its vapour in the air and means of characterising it.* Methyl alcohol may be determined in blood by adding 6–7 times the vol. of a saturated picric acid solution and distilling in a Schloesing-Aubin apparatus; all the methyl alcohol is found in a vol. of distillate equal to 1/15th the original vol. and may be determined therein by means of the  $\text{K}_2\text{Cr}_2\text{O}_7$  method previously described by the author. In the case of urine or any other liquid of the organism an equal vol. of picric acid solution is taken; in the case of tissues, 10–20 grms. of the latter is added to approximately 40 c.c. of the picric acid solution, and cut into small bits while immersed therein, the distillation and determination being then carried out as above. Methyl alcohol may be determined in air by passing the latter through a series of 6–7 wash bottles containing water at ordinary temperature and then following the procedure described above. In order to differentiate between methyl alcohol and other substances oxidisable by chromic acid, the ratio of  $\text{CO}_2$  produced to O consumed in the reaction is determined; the latter is calculated from the number of c.c. of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution employed, while the amount of  $\text{CO}_2$  produced is ascertained by absorption in  $\text{KOH}$  (the reaction is allowed to take place in a specially designed, hermetically sealed tube and the  $\text{CO}_2$  is then withdrawn by means of a mercury pump).

KROEGER. (Internat. Pharm. Cong.; J. Pharm. Chim., 1913, 8, 371.) *Toxicity of methyl alcohol.* Pure methyl alcohol is not so toxic as is generally represented. Probably the poisonous effects attributed to the alcohol are due to traces of dimethyl sulphate present in it.

KROL. (Arch. exp. Path. Pharm., 72, 444–56.) *Methyl alcohol poisoning.* Ammonia determinations carried out on a dog with const. diet gave a considerable increase after addition of methyl alcohol. Only a small part of the ammonia was neutralised by formic acid; the principal amount was bound to an acid not yet investigated. Animals given 45, 55, and 60 grms. methyl alcohol showed an incomplete narcosis, rapidly disappearing. Blindness was not observed.

FELLENBERG. (Mitt. Lebensm. Hyg., 1915, 6, 24–37.) *The occurrence of methyl alcohol in urine in different kinds of nutrition.* Methyl alcohol in urine was determined by the author's modification of the Denigès method. On a pectin-free diet very small amounts of methyl alcohol occur in the urine (about 0.4–1.0 mg. per 750 gr.), and on a fast it is reduced one-half. On a diet rich in pectin the amount increased to 0.8–2.5 mg. The condition of the pectin, whether raw, containing pectase, or cooked and pectase-free, makes little difference in the methyl alcohol secretion. The use of ethyl alcohol in a pectin-free diet raises the methyl alcohol content slightly; with pectin there is a large increase. Cheap wines containing both methyl alcohol and ethyl alcohol cause a maximum rise. The author discusses briefly the pathological effects of methyl alcohol on the system.

TYSON AND SCHOENBERG. (Sect. Ophth. Am. Med. Assoc., June, 1914; Klin. Monatsbl. Augenheilk., 53, 232 (1914); Zentr. Biochem. Biophys., 17, 663 (1915).) *Poisoning by inhalation of methyl alcohol.* Pronounced manifestations of poisoning were observed in dogs, rabbits, guinea-pigs and monkeys which were

placed in a space in which methyl alcohol was vaporised. The effect on the eyes consisted of irritation of the conjunctiva and cornea, hyperemia and oedema of the papillae with dilatation of the blood vessels. Coma, with moderate myosis and fixed gaze, and pronounced hypotony in severe cases, was observed. The aqueous humor acquired an acid reaction. Blindness ensued in some instances. Degeneration of the retinal ganglionic cell layer occurred.

CHAPANIER AND IBARRA-LORING. (Compt. rend. soc. biol., 1916, 79, 8–9.) *Excretion of methyl and ethyl alcohol by the kidneys.* The concentration of methyl alcohol and ethyl alcohol (determined by the Nicholson method) was practically identical in the blood and urine of subjects who had ingested these compounds. Ethyl alcohol and methyl alcohol were, therefore, simply diffused and not concentrated by the kidneys. Substances excreted by the kidneys may be classified in two groups: (1) those such as urea, glucose, iodides, etc., which have been concentrated by that organ, and (2) those such as methyl alcohol and ethyl alcohol, which are eliminated in the urine in practically the same concentration as they occur in the blood.

RUGGERI. (Liguria med., 7, 137; Zentr. Biochem. Biophys., 18, 461–2.) *Changes in lipid content in acute poisoning by methyl alcohol.* Both methyl and ethyl alcohols modify the lipid content of organs; for equal high concentrations methyl alcohol has the greater effect, while the changes produced at low concentration are practically the same for both alcohols. A considerable increase in the fatty acid and cholesterol content of the blood serum results from acute experimental poisoning with methyl alcohol. The complement action of the serum was diminished somewhat in acute experimental poisoning by ethyl alcohol, but this diminution was not as great as in methyl alcohol poisoning, probably due to greater effect on the lipoids and liver.

I am gratefully indebted to Dr. C. S. Roy and Miss C. Kern for assistance in preparing these references and abstracts.

#### TONICOLOGY OF METHYL ALCOHOL.

Extract from E. Merck's Annual Report, 1912, Vol. 26, p. 86.

In the past year a large number of observers have studied the question of poisoning by methyl alcohol, its various symptoms, its cause, its transient and permanent injuriousness, its fatal termination, and the dangers of methyl alcohol in general. These include, among others: Lewin, Rost, Levy, Strassmann, Foerster, Schenk, Ohlemann, Mendel, Hirschberg, Harnack, Grunow, Voltz, Kefenstein, Kuhn, Burger, Pick, Schmiedeberg, Bockha, Ruhle, Kroeber, Schlichting, Langgaard, Lennhoff, Stadelmann. For all details, reference should be made to the original communications of these authors, as they cannot be considered here. It may, however, be noted that they show that methyl alcohol itself, when habitually used, or when taken in a single large dose, may act as a poison, so that the toxicity is not due to the presence of impurities in the preparation. The fact that some persons are able to take comparatively large doses of methyl alcohol without apparent harm does not militate against the toxicity of methyl alcohol. It is impossible to say beforehand how large a dose will display an injurious action. According to Ruhle the lethal dose varied between 50 and 100 grammes, but the toxic dose is much less, and blindness may ensue after doses of only 7 to 8 grammes.

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Strassmann, Deutsche medizinische Wochenschrift, 1912, No. 3, p. 108.

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I have to acknowledge with thanks the assistance of Mr. P. Alderton for these references.

#### DISCUSSION.

Mr. A. E. BERRY said that the supply of methyl alcohol at the present time was greatly in excess of the demand. He had been closely in touch with the authorities on this question and their view was that there was no duty on crude methyl



alcohol such as was turned out in the Government factories. It was only in the purified form that it came into the same category as ethyl alcohol. He wished to correct one mistake on the part of the author when he said that *pure* methyl alcohol was not produced in this country to any extent. That might have been true a few months ago, but he could assure him that it was being produced now and could be produced in considerable quantities. His own interest in it related to the manufacture of formaldehyde, for which pure methyl alcohol was necessary. For many years formaldehyde had been imported here free of duty, yet the British manufacturer had been handicapped because he could not have sufficiently pure methyl alcohol free of duty, and he was glad attention had been drawn to the matter. He had been working hard at the question for three years, but had not made very much progress with the Customs Authorities. Mr. Morson had said it required an Act of Parliament to change the prevailing condition of affairs, and he (the speaker) suggested that the Society should present a petition to Parliament, pointing out the industrial applications of methyl alcohol and presenting all the evidence as to toxicity in order to get them to free the hands of the Customs Authorities, who, he was sure, would be glad to be relieved of the incubus that was associated with the name "methyl alcohol."

Dr. R. LESSING said that it was not only the duty that was irksome, because in most cases it could be arranged that this should not be paid or it could be refunded. That, however, involved a restriction of the methods of distillation and placed a great handicap upon the chemical engineer, because it was necessary to arrange the plant for the convenience of the Excise officer rather than with a view to the best efficiency of the plant. There were no restrictions as long as the alcohol contained sufficient impurities, for instance, 2% of acetone, but as soon as this figure of purity was reached the authorities stepped in and assumed control of the process from that point. It was then a question of having that portion of the plant under lock and key, which did not permit of that elasticity of working conducive to obtaining the best results.

Mr. W. J. A. BUTTERFIELD referred to Dr. J. Ramboisek's book on "Industrial Poisoning," in the translation of which by Dr. T. M. Legge, the Chief Medical Inspector of Factories, published in 1913, the following passage relating to the toxicity of methyl alcohol occurred:—

"Methyl alcohol plays relatively the greatest part among alcohols as an industrial poison, because it is employed as a means of denaturing spirit. Its poisonous nature is relatively great, being very persistent. Industrial poisoning by methyl alcohol is due to inhalation of the vapour and is rarely of a severe nature. The fumes have a strongly irritant effect upon the mucous membrane, giving rise to throat irritation, cough, hoarseness, and, in severe cases, bronchitis and inflammation of the conjuncture of the eye. In addition, inhalation of methyl alcohol vapour causes headache, giddiness, nausea (inclination to vomit), and occasionally also twitchings and tremor."

Mr. PROCTOR did not think the evidence as to toxicity of methyl alcohol was more than could be obtained with regard to the toxicity of ethyl alcohol, but if the case was proved against methyl alcohol it was probable that the Customs restrictions would be replaced by the more irksome poisons regulations. The question was not a simple one, there being a great many side issues which made the position of the Excise authorities very difficult. Mr. Morson had said that if he could get methyl alcohol duty free as readily as ethyl alcohol, it would meet his case. He (the speaker) did not

think there was any difficulty in this respect. If manufacturers generally made themselves acquainted with the regulations under which both methyl alcohol and ethyl alcohol could be obtained duty free, they would not find the difficulties so great as they imagined. In both cases denaturants were required, but many different denaturants could be used and manufacturers could generally suggest something used in their process which the Excise authorities would be quite ready to accept.

The CHAIRMAN asked whether the author had any evidence of the course of oxidation which methyl alcohol undergoes in the body, as this was a fundamental point in connection with its toxicity. It was known that alcohols acted upon the nervous system as narcotics, but methyl alcohol seemed to be exceptional in its physiological as in its chemical reactions, and it might be that this was due to a partial initial oxidation to formaldehyde or to formic acid. It was to be borne in mind that the term "toxicity" was a total effect and gave little indication of the action of a poison without some knowledge of the metabolic changes involved.

Mr. MORSON, in reply, said he was not able to answer the Chairman's question. The evidence from the literature was certainly in one case that the effect was due to oxidation to formaldehyde, which exercised a powerful chemical influence. In the other case it was due to slow oxidation to formic acid, but beyond that he was equally in the dark as to why it was so toxic. There was, however, the whole mass of evidence from physiologists and American medical men as to the intensely toxic effects, and if Mr. Proctor would look into that evidence carefully he would come to the conclusion that it was strong, especially when he saw the American pamphlets. The Americans had done an enormous amount of work, whereas we had not begun, and he claimed there was overwhelming evidence that methyl alcohol is highly toxic. Mr. Proctor had not answered the question as to why it should be taxed. It could not be used as a substitute for whisky or as a potable spirit at all without fatal results, and in that respect it certainly differed from ethyl alcohol. As to Mr. Berry's remarks, methyl alcohol became dutiable if it contained under 2% of acetone. He was glad to hear from Mr. Berry that there was going to be plenty of methyl alcohol; the statement in the paper on the point was not his but came from a firm of wood distillers. The question of the petition to Parliament need not be taken up yet, because he hoped the action of the manufacturers would go further.

## Nottingham Section.

*Meeting held at Nottingham on Wednesday,  
November 28th, 1917.*

DR. R. M. CAVEN IN THE CHAIR.

## POISONS AND DRUGS OF ANIMAL ORIGIN.

BY GEORGE BARGER.

The protective poisons which occur in many groups of animals have mostly not been isolated in a state of chemical purity. Generally, however,

we have at least some conception of their chemical nature. Thus cobra-venom contains a nerve-poison, which paralyses the respiratory centre, and also a hamolytic principle. The former, ophiotoxin, has, according to Faust,<sup>1</sup> a composition corresponding approximately to the formula  $C_{15}H_{25}O_{10}$  and resembles the saponins in physical properties; anyhow it seems to be nitrogen-free. The hamolytic substance is, according to Manwaring<sup>3</sup>, a lecithinase, an enzyme which splits off one of the fatty acid groups of the lecithin contained in the victim's blood, and this partially hydrolysed lecithin is known to be hamolytic. Previously Kyes<sup>3</sup> had imagined that when snake-venom is shaken with a lecithin solution, a compound soluble in chloroform is formed, in which one of the fatty acid groups of lecithin is replaced by an active constituent of snake-venom, to form a so-called lecithide.

The active principle of a toad has, on the other hand, been obtained crystalline and quite pure. A few years ago Abel and Macht<sup>4</sup> isolated from the so-called parotid skin gland of the large central American toad, *Bufo aqua*, a substance which undoubtedly has the composition  $C_{15}H_{21}O_4$ ; it has a very powerful digitalis-like action on the heart and is also a diuretic. These authors have thus found a scientific basis for the former great reputation of powdered toad skins as a cure for dropsy. Their use for this purpose now only survives in China under the name of "senso," a drug which Shimizu<sup>5</sup> has lately shown also to contain bufagin. The parotid secretion of *Bufo aqua* is further remarkable in containing over 5% of epinephrine (adrenaline) identical in every respect with that obtained from the suprarenal gland of the higher animals.

Whilst bufagin of the toad and apparently also ophiotoxin of the cobra are neutral non-nitrogenous substances, the skin of the newt, *Salamandra maculosa*, contains two alkaloids, which Faust<sup>6</sup> has isolated as crystalline sulphates. They are samandarine,  $C_{26}H_{40}ON_2$ , and samandaridine,  $C_{22}H_{31}ON$ ; both are powerful nerve poisons. From the latter base Faust claims to have obtained isoquinoline by distillation with zinc dust.

The difficulty of obtaining enough material is often a great hindrance in chemical work on animal poisons and is illustrated by Langer's<sup>7</sup> investigation of the poison of bees. This author collected 25,000 bee stings; but since the drop of poison ejected from a sting weighs only 0.2–0.3 mgrm. and consists mostly of water, only a few grams of the crude poison was obtained. The active principle in this case gives strong precipitates with alkaloidal reagents, but does not appear to be a protein.

More important than the protective poisons of the lower animals, of which a few examples have been given above, are the products of the so-called ductless or endocrine glands. These internal secretions, called by Starling hormones ("chemical messengers"), have been studied, especially in the larger mammals, including man, and some of them have of late years come into prominence as very potent and specific drugs. The remedial administration of the whole gland or of an extract has given rise to the name organotherapy. So far the preparation of the pure active principle on an industrial scale has only been accomplished in the case of the suprarenal gland. Two years after the isolation of adrenaline by Takamine<sup>8</sup> in 1901, its synthesis was patented by Meister, Lucius, and Brünig (Ger. Pats. 152,814 and 157,300). Chlor-aceto-catechol is combined with methylamine and the ketone is then reduced to the secondary alcohol. Since, owing to the slight physiological activity of *d*-adrenaline, the synthetic racemic substance was found to have not much more than half the activity of the natural *l*-variety, the Höchst firm had the enterprise to effect a resolution on the industrial scale by means of the bitartrates

(Ger. Pat. 222,451). The dextro-enantiomorph is not allowed to become a waste product, but is again racemised by heating with dilute acids (Ger. Pat. 220,355). Several other syntheses have been patented, chiefly by the same firm, but they do not appear to be of practical value (Ger. Pats. 155,632, 185,598, 189,483, 193,634, 195,814, 209,609, 209,610, 209,962, 212,206, and 216,640), nor have the closely related substances arterenol, homorenol, and epinine found a wide application as substitutes for adrenaline, although their physiological activity is in most respects qualitatively similar. The synthesis, involving a stereochemical resolution, may appear complicated, but it should be remembered that a pair of bullock's suprarenals together contain only 30–40 mgrms. of adrenaline, or about 0.2% (in marked contrast to the 5–6% contained in the parotid secretion of *Bufo aqua*, referred to above). On the other hand the drug is extremely active and it is said that by some physiological reactions as little as 0.000002 mgrm. may be detected.

A second important ductless gland is the thyroid. Its oral administration, introduced in 1891 by Murray, appears to be a complete remedy in those diseases where the natural secretion is defective (myxoedema, cretinism) and constitutes the greatest triumph of organotherapy. The chemical constitution of the active principle has not been established until quite recently. Baumann<sup>9</sup>, who more than 20 years ago discovered the iodine content of the thyroid, obtained from it by boiling with dilute sulphuric acid an amorphous impure substance iodothyron, with 9–10% of iodine. The statements as to the physiological activity of this substance are conflicting. The same may be said of a protein, thyroglobulin, obtained by Oswald.<sup>10</sup> Recently Kendall<sup>11</sup> has employed alkaline hydrolysis, with greater success, and a paper has been announced entitled, "The isolation and identification of the iodine-containing compound which occurs in the thyroid" (J. Ind. Eng. Chem., Oct., 1917, p. 985). Previous papers by the same author<sup>11</sup> have indicated that the free base contains 65% of iodine, its sulphate 60%. The molecular weight of the base is about 586. A photomicrograph of the crystals has also been published. The base is active in cretinism in very small amounts and the limit of tolerance is 2 mgrms. daily. The substance seems to be an indole or tryptophane derivative, and this would explain previous statements in the literature that iodized blood proteins have a physiological action like that of the thyroid gland.

If the constitution of the thyroid active principle has been definitely established, its industrial syntheses will be an important problem. By the use of a synthetic preparation the difficulties of dosage might be overcome, which at present result from the variability of the gland in animals. The standardisation of thyroid preparations has so far only been carried out by physiological means; their iodine content does not afford a sufficient indication of activity.

Reid Hunt<sup>12</sup> has devised a peculiar and very sensitive method for recognising the activity of thyroid preparations; he finds that very small quantities of the active gland increase the resistance of mice to the toxic effects of acetonitrile; as little as 0.1 mgrm. of the gland may double their resistance to this poison. By this means Hunt claims to have proved the presence of the thyroid secretion in the blood of sufferers from Graves' (Basedow's) disease, which is generally regarded as due to an excessive functioning of the gland. A more recent and probably simpler method of testing, which illustrates the effect of the active principle on metabolism, is due to Guder-natch<sup>13</sup> and consists in feeding the thyroid preparation to tadpoles. The latter diminish in size, the tail becomes absorbed, and at the same time



development is accelerated, so that rudiments of the legs appear earlier than in the controls. This method has even been recommended for the standardisation of commercial preparations. (Photographs, illustrating it, will be found, for instance, in a paper by Rogoff and Marine, J. Pharmac. exp. Therap., 1906, 9, 57). By means of the tadpole method Marine and Rogoff<sup>14</sup> have shown that when potassium iodide is injected intravenously into an animal with a defective (iodine-free) thyroid, the iodine is fixed by the gland almost immediately, but it takes about 16 hours before this iodine is converted into the physiologically active substance.

On account of its wide-spread occurrence we may here briefly mention goitre ("Derbyshire neck"), the commonest disease of the thyroid, although it is not connected with a defective secretion of the active principle. The dispute as to the cause of this disease has lately been settled by McCarrison, who attributes it to an infection by an organism from the soil and from drinking water, thus explaining its endemic occurrence in many parts of the globe, while in other regions it is rare or absent. Closely associated with the thyroid gland and often embedded in it are the parathyroids, small organs, which seem to be even more important to life than the thyroid itself. Nothing is known about their active principle. After removal of the parathyroids the urine contains considerable quantities of methylguanidine (Koch<sup>15</sup>).

The pituitary (hypophysis cerebri) is another very important ductless gland. It consists of an anterior nervous, and a posterior infundibular portion. Its situation under the brain makes its experimental extirpation difficult, and attempts in this direction have not thrown much light on the functions of the parts. One of these functions is an influence on growth; the rare disease acromegaly, characterised by an enlargement of some bones (of the lower jaw, hands, feet, etc.), is associated with lesions of the pituitary. Brailsford Robertson<sup>16</sup> has recently extracted a substance from the anterior lobe, for which he claims that it has a definite effect on the growth of mice, making them heavier and sturdier, although not larger, than the controls. This substance, tethelin, is a phosphatide with nitrogen and phosphorus in the ratio 4N : P, but probably not pure.

More than the anterior, the posterior portion of the pituitary has attracted the attention of physiologists. Its extracts have a pronounced action on the blood pressure, on the isolated uterus, on the kidney, on the mammary gland, etc. The pressor action of the pituitary was discovered by Oliver and Schäfer<sup>17</sup> almost simultaneously with that of suprarenal extracts. The rise of blood-pressure is smaller, but more persistent than that due to adrenaline. Whether the diuretic action, discovered later, is due to the same or to a different hormone is not known, and the same may be said of the powerful action of infundibular extracts on the uterus, which was discovered by Dale<sup>18</sup> and has of late been applied therapeutically with great success. Various indications, however, point to there being several active principles. Unfortunately no active substance has been isolated in a state of undoubted purity. Most is known about the properties of the pressor substance; it can be boiled in neutral or slightly acid solution without much loss of activity, but is rapidly destroyed by alkalis. It is also destroyed by trypsin, whence it would seem to be a peptide. It is insoluble in absolute alcohol and other organic solvents and is readily adsorbed by charcoal and by precipitates formed in its solutions and thus it is carried down by phosphotungstic acid. The action of pituitary extracts on the uterus, resembling that of the ergot sub-

stance  $\beta$ -iminazolyethylamine, and the fact that pituitary extracts, like this glyoxaline, give a diazo-reaction, has led to speculations concerning a similarity in the constitution of the two substances. Guggenheim<sup>19</sup> condensed  $\beta$ -iminazolyethylamine with various amino-acids, forming dipeptide-like substances which, however, only had a slight activity. Some years ago Fühner<sup>20</sup> stated that Meister, Lucius, and Brüning had obtained by means of phosphotungstic acid four crystalline sulphates, all physiologically active (compare Ger. Pat. 268,841). Various others have claimed to have isolated an active principle, e.g., Bandonin<sup>21</sup> and Bouin and Ancel<sup>22</sup>, but none of these claims seems to be well founded. It is possible that all the four Höchst bases were contaminated with a highly active substance; it is at least significant that these bases have not been characterised chemically. The isolation and the determination of the constitution of the pituitary active principle is evidently a matter of great difficulty, but would undoubtedly be of great value, particularly if it were followed by synthetical manufacture.

In reviewing drugs of animal origin we must finally draw attention to the immune sera, the production of which forms a peculiar and highly specialised branch of chemical industry. Of these anti-diphtheritic and anti-tetanic serum are by far the most important, the former in times of peace, the latter during the present war. They are obtained by injecting the filtrate from cultures of bacilli into horses. The soluble toxin renders the horses immune and their blood is subsequently found to contain so-called anti-toxin, sufficient to neutralise large quantities of the toxin. By periodic bleeding and separation of the serum, a highly specific and very valuable remedy is obtained. The anti-toxins, like the toxins, are unstable and very complex substances; their isolation seems to lie quite beyond the present bounds of chemical knowledge.

The high hopes raised at first by the success of diphtheria anti-toxin have not been wholly fulfilled, as regards the treatment of other bacterial diseases. In most cases the organism does not secrete a soluble poison into the culture medium, and hence weakened or killed cultures are injected. Then a so-called bacteriolytic serum may result, e.g., with cholera cultures a serum dissolving cholera vibrios. Among bactericidal sera, obtained in a similar way, we may mention those against streptococci, pneumococci, typhoid, and cholera, which are still more or less in an experimental stage; perhaps the anti-meningococcus serum is of most importance in this group. In a few cases immune sera have been employed successfully against non-bacterial toxins. The anti-venin of Calmette is an efficient protection against snake bite, but the difficulty here is that the immune serum against one species of snake does not protect against the bite of another species. A further interesting non-bacterial anti-serum is that from animals immunised against pollen grains; it is used to combat hay-fever.

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## Yorkshire Section.

Meeting held at Queen's Hotel, Leeds, on Monday, December 17th, 1917.

PROF. J. W. COBB IN THE CHAIR.

## APPROXIMATE ESTIMATION OF TAR FOG IN GAS.

BY ALFRED EDWARDS.

The estimation of tar fog in gas presents some difficulty. It is not easily and completely removed by filtration in a bulk sufficient to weigh; evaporation renders the true weight uncertain. Correct samples of the gas and tar fog are not withdrawn from the gas stream as readily as one might expect. A critical examination of the problem indicates that only under specified conditions can the sampling be correct, owing to the non-homogeneous character of the flow and the momentum imparted to the fluid particles by velocity. These conditions require the sampling orifice to face the current at a point of mean velocity, and the flow through the orifice to be the same in speed as that of the main flow.

When I was asked to determine the efficiency of a tar fog extractor, the conditions defined for accurate results seemed so impossible to reproduce that approximate methods were sought. A crude means of indicating the presence of tar fog is to allow the gas, issuing at high velocity, to impinge upon a white surface. Attempts have been made to render a visual comparison of the resulting stains a basis for a roughly quantitative comparison between two similar streams. Visual methods of this character are decidedly preferable in technical work if they can be made quantitative as well as comparative. The variation finally adopted was to draw the gas through ordinary filter paper of stout substance and measure the increased density to light produced by the stain.

If we then know the area of the stain on the filter paper (A), the density of the stain (D), the weight of tar to give unit density on unit area (T), and the volume of the gas ( $V_g$ ), the quantity of tar fog present in the gas is  $A \times D \times T \div V_g$  and may be expressed in grains per cubic foot or grams per cubic metre according to the particular units taken.

T is determined by suspending the tar, collected at the point considered, in solvent naphtha or similar colourless liquid and measuring the density in a glass cell with parallel sides.

If then W is the weight of tar taken, V the volume of solvent in which W is dispersed,  $V_c$  the volume of cell, A the area of side of cell, and D the density of tar and liquid in cell, the weight of tar to give unit density if spread over unit area is :—

$$\frac{W \times V_c}{V \times A \times D} = T.$$

The instrument by which the density to light is measured is one intended for the measurement of silver deposits in photographic negatives. Its use in addition to this specific purpose I have extended to the determination of various substances, where in fact the estimation can be reduced to one of density to light, with such good effect that I have no hesitation in recommending it as an essential part of the equipment of any laboratory. It is sold under the name of the "Density meter" by Sanger Shepherd and Co., and is a comparison photometer of very simple construction. Two apertures are viewed in such a way that they appear disposed side by side. A translucent object placed over one of the holes obstructs the light passing to one half of the field of view, and its intensity can be balanced by means of a neutral tinted pigment wedge deposited on glass placed over the other hole. The wedge is graduated in terms suggested and used by Hurter and Driffield in their classical photographic researches, density = log. opacity, opacity being the ratio the incident light bears to the transmitted light. The density of neutral tinted bodies can be balanced exactly by the instrument. When the body is coloured and transparent, the density to its complementary colour can be measured by viewing the aperture through a transparent screen of the correct colour. The coloured body is, in effect, converted into a neutral tint and can easily be balanced against the neutral wedge. Where an opaque body is present in a transparent coloured fluid, the aperture is viewed through a screen of the same colour as the fluid, and the opaque body balanced by the wedge. The instrument thus becomes a cheap, if crude, substitute for the spectro-photometer.

As an example of the determination of T (weight of tar per unit area to give unit density) the figures given in the following table were obtained. The density measurements were made in a glass cell of which the parallel sides were 10.2 mm. apart, the capacity 5.55 c.c. and the area of the side 5.441 sq. cm. 0.6674 grm. of tar was suspended in 50 c.c. of solvent naphtha and portions were diluted to 50 c.c. before measurement.

### Density to light.

	2 c.c.	4 c.c.	6 c.c.	8 c.c.	10 c.c.
Red .....	0.37	0.77	1.13	1.51	1.87
Green .....	0.63	1.23	1.75	2.27	2.70
Blue .....	0.82	1.52	2.18	2.70	—

Density for 1 c.c. calculated from above.

	0.185	0.192	0.183	0.189	0.187
Red .....	0.315	0.367	0.291	0.284	0.270
Green .....	0.410	0.380	0.363	0.337	—
Blue .....					



It will be observed that the density to red light is in this case fairly even throughout the range; with green and blue light there is a gradual falling away.

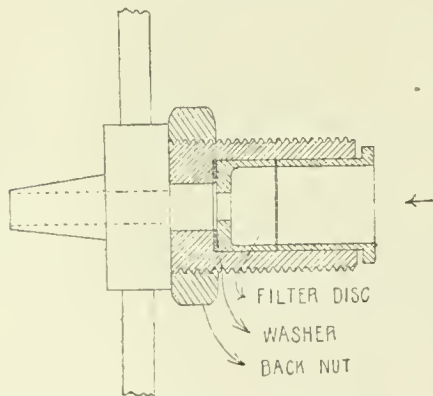
Taking the average density for 1 c.c. to red light we have the following facts:— $W=0.01335$  gm.,  $V=50$  c.c.,  $V_c=5.55$  c.c.,  $a=5.441$  sq. cm.,  $D=0.188$ .

$$T = \frac{0.01335 \times 5.55}{50 \times 5.441 \times 0.188} = 0.00145 \text{ gm. per sq. cm.}$$

The value of  $T$  varies for tars produced by carbonisation in various systems, and with the temperature of carbonisation in any one system. If the density does not increase in simple ratio with the weight, the points may be plotted and intermediate values read from the graph.

#### Sampling the gas.

The gas is filtered through a disc of filter paper cut by a cork borer or similar implement to a diameter of 1.8 cm. The disc is held between the ends of metal liners, 1.6 cm. diameter, sliding within a screwed metal nipple, thus exposing 2 sq. cm. of filtering area. A rubber washer is placed below the inner liner to secure tightness to gas as indicated in the figure. The nipple is screwed  $\frac{3}{4}$  inch gas pipe, and enters a cock screwed hard up to the gas main. It is important that the connection should have a small volume, and that the filter paper should be as close to the gas stream as possible. A  $\frac{3}{4}$  inch brass cock, full way, with male and female ends, screwed directly into the main, provides the shortest convenient connection. In order to ensure a fairly representative sample of the gas the sampling must be rapid to prevent the falling out of the larger tar particles. With



this object the difference of pressure between the two sides of the disc should be from 12 to 24 inches water column, depending upon the resistance of the paper disc. Speed is also necessary to complete the removal of the finer particles of fog by impingement within the pores of the filter paper. One disc only is necessary. A second one placed in series with the first and spaced by a metal liner is invariably unstained. The stain is always confined to the receiving surface of the first disc. Where the gas is under low pressure an aspirator is used to give the required pressure difference.

The volume of gas to be passed through the filter paper to give a stain within measurable limits, say 0.4 to 1.2, varies from 80 c.c. to 9000 c.c. depending upon the proportion of tar fog present. With the smaller volume the results become doubtful, since the volume of the voids in the connection and nipple bears an ever-increasing proportion to that of the gas sampled as the latter

becomes smaller. If the filter paper is of uniform texture and the connections short but of ample diameter, the stain obtained is evenly distributed. Very slight inequalities are averaged out by the density meter.

It is important that diffused light only should be used for measurement. Small pieces of ground glass covering the apertures and below which the filter paper or cell is placed secure this condition. Circles cut from the same sheet of paper have usually the same density and balance one another if placed over the two apertures. Of a set prepared for estimation, one disc is retained as a blank and divergent densities of others recorded and allowed for when the stain is measured, which should be with the blank in position over the wedge aperture. It is advisable to retain a second disc, which has been balanced against the blank, in order to check the setting of the density instrument.

It is not advisable greatly to exceed unit density of stain on the filter paper. Although the measurement in a glass cell to a density of about 1.5 usually increases evenly with the weight of tar present, the uniform conditions of dispersion under which the soluble and insoluble portions of the tar are viewed, cannot be obtained upon the relatively small section of the filter paper surface, which is never absolutely uniform. Considering an extreme case, the tar particles—larger than those present in the cell—may fall one upon the other to extinction of the light in that one spot. Long before such a state is reached the density will fail to increase proportionately with the weight of tar. For comparative purposes it is desirable to secure approximately the same density in each determination, and check the weight of tar per unit area for that density, averaging it over fairly narrow limits.

As an example of an estimation the following figures are given:— $A=2$  sq. cm.,  $D=0.93$ ,  $T=0.00145$  gm. per sq. cm.,  $V_g=3819$  c.c.

Tar fog  $= 2 \times 0.93 \times 0.00145 \div 0.003819 = 0.70$  gm. per cubic metre  $= 0.70 \times 0.436 = 0.30$  grain per cubic foot.

Interesting results have been obtained upon gas streams passing through various manufacturing plants.

	Grains per cubic foot.	Grms. per cubic metre.
Coal gas.		
A. Outlet condensers following seal in hydraulic mains .....	30	68
B. Outlet condensers following part sealed and part dry mains with retort house governors .....	15	34
C. Outlet P and A tar extractors following A .....	0.25	0.57
D. Outlet rotary exhausters following dry mains and condensers .....	8	18.3
E. Outlet rotary exhausters following B ..	6	13.7
F. Outlet turbo exhausters following B ..	0.6	1.37
G. Outlet Livesey washers following D ..	0.6	1.37
H. Outlet P. and A. tar extractors following E ..	0.17	0.39
I. Outlet P. and A. tar extractors following F ..	0.14	0.32
J. Outlet ammonia washers or scrubbers horizontal revolving type following G ..	0.4	0.9
Water-gas, partly carburetted.		
K. Outlet rotary exhausters following relief holder .....	2.3	5.2
L. Outlet Crossley tar extractor following K ..	0.17	0.39

It will be observed that retort house governors have an appreciable effect on the tar fog. The rotary exhausters remove a fair proportion of the remainder. Turbo exhausters reduce the amount to as low a figure as Livesey washers following

rotary exhausters. "P. & A." machines carry the removal to finer limits; the fact to observe is the relatively small difference in the outlet fog compared with a wide range at the inlet. Obviously efficiency should not be based upon the percentage removed. At one works where the A+C combination is used there is trouble with the "P. & A." machines making up; no trouble is experienced with the A+C or F combination; overloading may account for the difficulty.

#### *Accuracy of the method.*

It was not expected primarily that the method would yield absolute figures for tar fog with great accuracy. A short experience proves that if we take the densities of the stain per unit volume of gas, the figure is very constant for any given gas stream. Individual determinations reproduce the figure time after time if the variation of density is not too great. It has also been found that the quantity passing similar points in different works, calculated to an absolute amount from the stain readings, is in agreement, despite the fact that the densities of the individual tars to red light differ materially. Whether the method by which the density of the tar is ascertained is perfectly valid or not has not been settled by a direct weighing of the tar fog extracted from the gas, in view of the many difficulties, but the agreement just quoted suggests that it is. Contrasting the filtration and actual weighing of the tar with the method proposed, the former must give low results because of evaporation of the constituents of lower boiling point. With the second it is highly probable that evaporation will not affect the density to light since the colouring matter with the free carbon remains behind.

#### DISCUSSION.

The CHAIRMAN said that he had himself attempted to face the difficulties with which the author had dealt so ingeniously. He endorsed all that had been said in the paper as to the difficulty of getting a really satisfactory determination of tar fog by direct weighing. In order to obtain a sample some form of conveying tube must be inserted in the gas main and that obviously would disturb the stream lines. Moreover the impinging of tar particles on the sides of the tube resulted in their aggregation and elimination, for they were liquid and not gaseous. A similar difficulty was encountered with ammonia, which tended to elimination by solution. He asked Mr. Edwards if the temperature fell on the way to the filter-paper, because that seemed possible and would interfere with accuracy. The main difficulty in making the method more than approximate was that although dispersion in the comparison cell made every particle capable of exercising its quenching effect on light, that was by no means the case with the filter-paper, where particles might lie one behind the other. The paper showed that the difficulty had been recognised, but a much more considerable range of experiments seemed advisable for determining how far so convenient a method was also reliable. Had the results, showing that the effect of a turbo-exhauster was equivalent with regard to tar fog to that of a rotary exhauster followed by a Livesey washer, been determined by a number of experiments?

Mr. B. A. BURRELL said that the author was to be congratulated on his application of a system used in pure photography to the solution of a difficult problem connected with industrial chemistry. He had found that the various batches of filter paper were by no means uniform and he asked whether this had any effect on the opacity and therefore upon the estimation. Was it necessary to use a standard paper? Also had any attempt been made to use a Lovibond tinto-

meter, and if so could the Lovibond units be converted into Hunter and Driffeld units or *vice versa*?

Mr. G. L. MOSS asked if the author had made any estimation of tar fog before and after the benzol scrubbers, and also if the tabulated results from the P. & A. extractors had been obtained under pressure or suction, or some under one and some the other?

Mr. JAMES MILLER asked if the author had tried the use of a soluble organic medium instead of a filter paper—a layer of naphthalene, for instance, to be afterwards dissolved. He thought this would overcome the difficulty suggested by the Chairman with regard to the filter paper.

Mr. GEORGE DOUGILL confirmed what had been said with regard to the turbo-exhauster; he could guarantee its efficiency as a tar extractor.

The CHAIRMAN: In your experience does it carry it as far as the Livesey washer?

Mr. DOUGILL replied that that would be very difficult to say from works observation, but the gas at the Livesey washer was very clean after a turbo-exhauster.

Mr. H. J. HODSMAN asked if the amount of tar fog found did not depend on the point in the cross section of the main from which the sample was drawn, and whether Mr. Edwards' experiments bore on the point. Had the effect of sampling at a bend been compared with sampling from a straight length of main?

Mr. A. WOODMANSEY asked if it were feasible to base a method on direct observation through a column of gas in one of the mains. Some arrangement (*e.g.*, a current of fog-free gas) would be necessary for keeping the glasses clean.

Mr. EDWARDS, in replying as to the temperature of the gas affecting the results, said that all his experiments had been made with cold gas, *i.e.*, with gas not differing by more than 20° or 30° F. from the temperature of the surrounding air. If the metal instrument were allowed to remain in contact with the main long enough there might be some effect, but if the sampling were carried out in a few seconds he did not think there was any deposition of tar from the state of vapour on the filter paper. He had had determinations made on several days on which the temperature of the gas had not varied, while the temperature of the air had been 20° to 30° F. lower, but on every occasion he had obtained the same results at the same point, serving to show that there had been no condensation from the state of vapour. Moreover, the nature of tar fog rendered it very improbable that there would be any such action. It was produced in the retort, and was really free carbon, containing small proportions of the oils, and if it did take up more of these it could only be to a small extent. It was present right up to the Livesey washer, from 6 grains per cubic foot up to, say, 30. He intended to make determinations with hot gas if he could find a gas that contained little enough fog. With reference to the superposition of tar particles on the filter paper not giving comparative results with tar suspended in liquid in a cell, that was of course the crux of the question, and he had not been able to satisfy himself on this point. Regarding the question as to turbo-exhauster results in comparison with rotary exhausters and Livesey washers, he said that he had made perhaps two dozen estimations and they had always come out within 0.1. In reply to Mr. Burrell, he said that different qualities of filter paper were difficult to get, and he had only used one variety. The tintometer was probably a fairly suitable appliance, but not so handy as the density meter. He had not made any determinations before and after the washing



of the gas with oil for benzol. The results as regarded the P. & A. extractor were under pressure: he had not been able to determine whether there was greater efficiency under vacuum. Efficiency was somewhat difficult to define, and one must not take the quantity of tar inside the machine and regard the percentage extracted as a test of efficiency. This was shown by the combination of "A" and "C" in the table of results compared with "E" and "H." The turbo-exhauster was most efficient considering that it was really used for quite a different purpose. As to the position in the main from which it was best to take samples of gas, he had obtained his samples at the periphery. Very different results had been obtained at points facing the gas system and at right angles to it, and anything like the latter method must be avoided. Mr. Miller's suggestion as to the use of naphthalene instead of a filter paper was a very good one if it could be well worked out. It had also been suggested that if tar fog was present in sufficient quantity its density to light could be observed directly by diverting a small stream of the gas from the main stream, but he was afraid that was impracticable.

#### NOTE UPON APPARATUS FOR THE DETERMINATION OF BOILING POINTS.

BY ALFRED EDWARDS.

Commercial apparatus for the distillation and fractionation of liquids fails to provide for the total immersion of the thermometer stem, with the result that corrections must be applied for the unheated column of mercury. Boiling-point determinations have usually been made under the same conditions; the aim in designing the apparatus used has been to reduce its bulk.

It is desirable to use thermometers under the same conditions of immersion as were adopted when they were made, or as checked by the National

Physical Laboratory. Advantage may then be taken of the corrections without prior calculations for differing stem immersion.

Extended use of the boiling point method for the estimation of benzene and toluene in commercial mixtures (this J., May 31, 1916) and for the control of stills for rectification, emphasises the need for a modified boiling point apparatus, and a still-head has been devised to replace the apparatus described.

An inner tube, A (Fig. 1), about 48 cm. long and 13 to 14 mm. external diameter, is supported by a ring of cork in an outer tube, B, of 16 to 17 mm. bore. The inner tube has a hole blown in the side near the top and projects above and below the outer tube. The outer tube has a side limb, C, of 7 to 8 mm. bore, fused on 38 to 40 cm. from the top. C is bent upwards to form a reflux condenser, the effective length of which is 10 to 15 cm.; the water jacket has a diameter of 16 to 17 mm. Wide-mouthed flasks are attached to B by corks. The vapour rises in A, passes over the thermometer stem, issues by the hole at the top, and descends between A and B, being finally condensed in C and returned to the flask. The return of liquid tends to seal the space between A and B and secure a regular stream of vapour upwards through A. Instead of a cork joint a fused one may be used, as in Fig. 2, or the inner tube may be expanded at the top and supported upon a contraction of the outer tube, as in Fig. 3. The inner tube may be bulbed at the bottom, as in Fig. 4, or the outer contracted, as in Fig. 5, if the sizes of the tubing available allow too much space between them at this point.

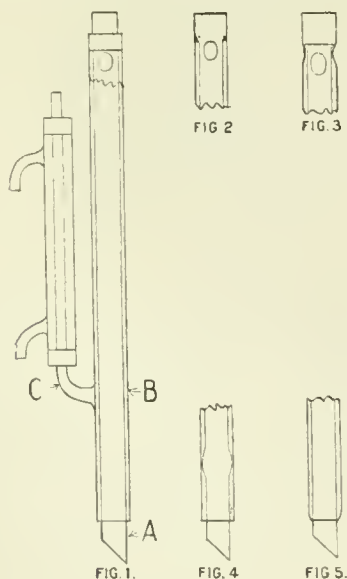
The advantages of this apparatus are:—Full immersion of the thermometer stem. The inner current of vapour is protected from draughts by a jacket of vapour. Condensed liquid is returned to the flask below the bulb of the thermometer. Possibilities of superheated vapours are remote. Temperature is maintained steady without fluctuation and without shielding from draughts.

#### DISCUSSION.

The CHAIRMAN said that Mr. Edwards' use of the term "immersion" avoided an ambiguity which undoubtedly existed at the present time as to the meaning of "exposed" stem. He (the Chairman) had always spoken of the correction for "exposed" stem, meaning thereby the stem exposed to the air, but Spielmann and Jones (this J., 1917, 489) used the term in exactly the opposite sense as meaning the stem exposed to vapour; there was no agreement among chemists or in the literature on the point. Did Mr. Edwards use the jacketing principle in testing liquids which had to be evaporated to dryness?

Mr. H. J. HODSMAN said that if one were determining the boiling point of a mixture, probably a considerable amount of the liquid might be in the upper part of the apparatus, and might conceivably affect the experiment. How far was this a possible source of error?

Mr. EDWARDS, in reply, said that in all apparatus in which total immersion was aimed at, there must be a large vapour phase compared with the liquid phase, and the only way to counteract the effects one had in mind was to use plenty of the liquid. It was possible to turn down the condenser and use it as an ordinary still-head, but that was of no advantage if the neck of the flask were long enough to immerse the thermometer completely, as advised by Prof. Young in his book, "Fractional Distillation."



## Bristol and South Wales Section.

Meeting held at Bristol University on Tuesday,  
December 18th, 1917.

DR. T. H. BUTLER IN THE CHAIR.

### NOTE ON A CURIOUS CASE OF CONDENSER TUBE CORROSION.

BY J. KEWLEY.

The condenser which exhibited the curious type of corrosion explained below was installed in March, 1915, for the purpose of cooling warm fresh water. It was of the ordinary marine type, with tubes of British Admiralty specification, and mild steel tube plates. The body of the condenser was of cast iron. The tubes were fitted into the tube plates by means of wooden ferrules. The metal of the tubes gave on analysis:—Cu 70.68%, Zn 28.81, Sn 0.17, Pb 0.18, Fe 0.20%. The fresh water circulated round the tubes, and brackish dock water circulated through them. The brackish water which caused the corrosion showed on analysis:—NaCl 13.48.4 grains per gallon,  $K_2SO_4$  16.9,  $CaSO_4$  36.0,  $MgSO_4$  116.7,  $MgCl_2$  147.5,  $MgCO_3$  1.1 grs. per gall. The fresh water entered at a temperature of about 50° to 60° C., emerging at about 25° C. The range of temperature was thus comparatively small.

After working for four months, the condenser appeared to be leaking badly, and when it was opened up very considerable corrosion had taken place. This was, with very rare exceptions, entirely confined to the ends of the tubes. The corrosion was, moreover, confined to a distance of not more than two inches from the end of the tube, and was confined entirely to the end of the tube at which the brackish water entered. The rest of the tube and the outlet ends were in perfect condition, and showed no trace of corrosion whatever. Further, the corrosion was much more pronounced in the bottom tubes of each of the three compartments, and the number of tubes which had to be replaced was greatest in the lowest compartment, and least in the top compartment. As the hot fresh water entered at the top and the cold brackish water at the bottom, the maximum corrosion took place at the point where the difference of temperature was least.

After replacing the worst tubes, the condenser was again boxed up, and after a further three months was again opened, and exactly the same state of affairs found as on the previous occasion. The difficulty was, however, overcome in a very simple manner. The bad ends of the corroded tubes were cut off and these tubes were lengthened by the addition of nine inches of similar tube joined on by a sleeve made of the same material but of larger diameter, the joint being sweated over with solder. All the tubes were so treated and put back in the condenser with nine inches of their length projecting at the outlet ends. After a run of three months the condenser was opened, and the tubes all knocked through a distance of two inches, so that the badly corroded inlet ends projected. During the next three months a further two inches was corroded away, and the process repeated. The tedious and expensive process of re-tubing the condenser was thus necessary very much less often. As the tube became eaten away at one end it was lengthened at the other.

It is of great interest to note that another condenser, working under the same conditions, showed no signs of corrosion over a period of two years. In this case, however, the tubes were

made of "Nergandin alloy" (Cu 70, Zn 28, Pb 2%), and tube plates and ferrules of Muntz metal.

## London Section.

Meeting held at Burlington House on Monday,  
January 7th, 1918.

DR. CHARLES A. KEANE IN THE CHAIR.

### CORROSION OF LEAD ROOFING.

BY J. S. S. BRAME.

Two interesting cases of corrosion of lead sheet used for roofing have been investigated.

In the first case cast lead sheets had been in position on a wood foundation for a long period. I am unfortunately unable to give the number of years as no record of the date of roofing has been kept. It was found that in places serious corrosion had occurred, the lead being in some instances perforated. Beneath the corroded parts thick crusts of white deposit were found, and these on analysis proved to have a composition well within the range of commercial white lead. The following are the results for two samples:—

	Sample A.	Sample B.	Required for $2PbCO_3$ , $Pb(HO)_2$ .
	%	%	%
Lead monoxide ( $PbO$ ) ....	85.40	84.10	86.32
Carbon dioxide.....	11.50	11.70	11.36
Combined water.....	2.15	2.05	2.32
Organic and other constituents (diff.) .....	0.75	2.15	—
	100.00	100.00	100.00

Examination showed that the lead had only corroded where in contact with oak beams; the other portion of the wood-work consisted of pine. In Bloxam's "Chemistry" it is stated that "the lead of old coffins is often found converted into a white earthy mass of basic carbonate, with a very thin film of lead inside it." It would be interesting to know whether this applies to lead in contact with other woods than oak. Miller, who did much valuable work on the action of water on lead, says "green oak wood, from the quantity of acetic acid which it contains, should not be used in contact with lead for building purposes." I have been told that in erecting sulphuric acid chambers it was recognised that oak and lead should not come in contact, but on inquiry I find that corrosion of lead under these circumstances is not generally realised, and therefore venture to record these observations.

The second case of lead corrosion was more remarkable and occurred where the metal had been laid on a coke-breeze concrete. Here patches of a thick crust of a red substance had accumulated between the lead and the concrete, and in places the lead was eaten through. In this case the action had been somewhat rapid, the building being only some 15 years old, when a leaky roof revealed the serious corrosion which had been going on.

The corrosion product had the following ultimate composition:—Pb 91.64%, Fe 0.084, O 7.450, organic matter, carbonates, etc. (diff.), 0.826%. The theoretical amount of oxygen required to form



PbO from the lead present is 6.57%, so that there is excess oxygen equal to 0.88%. From this result, and from the red colour of the corrosion product, it is evident that either lead sesquioxide ( $Pb_2O_3$ ) or red lead ( $Pb_3O_4$ ) is present in addition to the lead protoxide (PbO) which constitutes the bulk of the material. From the ultimate analysis the following alternative approximations of the probable composition of the material (exclusive of the minor constituents,  $Fe_2O_3$ ,  $CO_2$ , etc.) may be made:—

Lead protoxide (PbO) .....	90% or	86%
Lead sesquioxide ( $Pb_2O_3$ ) .....	9%	Red lead ( $Pb_3O_4$ ) 13%

It is usual to regard both the  $Pb_2O_3$  and  $Pb_3O_4$  as composed of protoxide (PbO) and dioxide ( $PbO_2$ ), and confirmation of the presence of lead dioxide is afforded by the evolution of some chlorine when the material is distilled with hydrochloric acid.

The coke-breeze concrete consisted of about 8 parts of coke-breeze and 1 part of Portland cement, the latter in the set condition being usually regarded as containing a certain proportion of calcium hydroxide, and the action was probably due to this free lime, which, in the presence of moisture, has been stated to form hydrated lead oxides with the metal. In some of the older textbooks it is stated that lead should not be placed in contact with mortar, plaster, etc., on account of corrosion, but I have been unable to trace any reference to the character of this action. Certainly the production of an oxide higher than PbO is remarkable.

#### DISCUSSION.

Dr. S. MIALl said that it was unfortunate that the true early history of both these examples was not available. As regards the first instance, he was very doubtful whether there was sufficient acetic acid in ordinary oak to act upon sheet lead placed in contact with it. He did not believe there would be any greater corrosion with dry oak than with dry elm or any other wood. The second instance of corrosion was remarkable and difficult to understand. Was it possible that the circumstances in which the sheet lead was placed were such that there was any formation of calcium plumbite which might afterwards form calcium plumbate? This would be readily attacked and might form a certain amount of peroxide or red lead.

Mr. G. NEVILL HUNTLY, referring to the second case, said that some years ago several examples of the corrosion of leaden pipes by mortar were shown him. The corrosion products were white. It appeared probable that a calcium plumbate was formed, but it could not be definitely proved. It was, however, a well-established tradition among plumbers not to allow lead pipes to come in contact with lime or mortar.

Mr. ARNOLD PHILIP pointed out that electric cable companies always stated that lead-covered cables must not be brought into contact with Portland cement. He had seen several cases of corrosion of lead-covered cables from the neglect of this precaution but he did not understand their origin.

Mr. WALTER F. REID said that the corrosive action of Portland cement on lead was well known but the cause of it was not known. Alkalis were present and a small percentage might cause considerable action on the lead. Portland cement made in the Thames valley was rich in alkalis and it would be very unsafe to put lead in direct contact with it, especially with a substance like coke, which contained a great deal of sulphur. It was also known that breeze contained rather more sulphur than coke and some action might be due to the sulphur, whether on the lead or other ingredients he was unable to say, because it was

not quite certain in what form the sulphur was present in the coke. The condensation of water he thought had a great deal to do with the corrosion of lead in contact with wood. He had noticed that where galvanised iron was fastened to wood there was condensation of moisture on the other side of the iron, and with continually recurring fog in such a climate as ours corrosion might be started.

Prof. BRAME, replying to Dr. Miall, said that the building from which the first instance of corrosion had been taken was over 200 years old and probably lead had been there the whole time. There was no record of it having been re-roofed. There was possibly a good deal in whether the wood was green or properly seasoned. There was something about oak which gave this corrosive effect; for instance, iron nails used in oak fencing were affected invariably. It was the red colour which he considered the most remarkable part in the second instance he had brought forward. He had seen pipes taken out where there was white corrosion but never coloured like that. It had been suggested that the sulphur in coke-breeze might cause the corrosion, but there was nothing more than a trace of sulphur in the corroded material. Dr. Miall's suggestion as to the action of the lime forming a plumbite or plumbate was probably the real explanation of the initial action in the second instance.

#### THE ACTION OF RAIN WATER ON A PORTLAND STONE.

BY J. S. S. BRAME.

It is well recognised that certain limestone buildings in large cities have suffered considerably from weathering, but beyond the general statement that rain water charged with acids, such as sulphurous acid, attacks carbonated stones with some rapidity, converting the carbonates into sulphates, I have been unable to trace any reference to the actual changes which occur.

My attention was directed to a thick black crust beneath the wide overhanging cornice of a building of Portland stone some 200 years old. The crust was so thick that it had almost completely blocked up the fine carving on the underside of the cornice. It was decided to remove this crust and samples were obtained for analysis.

The crust was almost black through included organic matter (soot, etc.). The air-dried material gave the following results:—Moisture 1.79%, organic matter 23.34%, mineral matter 74.85%.

Comparison of the composition of the stone (which apparently in the part taken had been little altered, as it consisted almost entirely of carbonates) with the mineral constituents of the crust is given below:—

	Lime-stone.	Inorganic constituents of crust:		
		Sol. in HCl.	Insol. in HCl.	Total.
	%	%	%	%
Lime (CaO) .....	52.71	25.10	7.71	32.81
Magnesia (MgO) .....	1.33	2.11	0.34	2.45
Iron and aluminium oxides ( $Fe_2O_3$ , $Al_2O_3$ ) .....	0.38	1.98	1.28	3.26
Sulphuric anhydride ( $SO_3$ ) .....	1.25	38.25	11.16	49.41
Carbon dioxide ( $CO_2$ ) ..	41.32	2.07	—	2.07
Silica ( $SiO_2$ ) .....	2.72	—	9.94	9.94
Organic and loss .....	0.29	—	—	—
	100.00	69.51	30.43	99.94

It is clear that the crust consists mainly of calcium sulphate, the next main constituent being silica. In the portion insoluble in hydrochloric acid a large quantity of fine needle-shaped crystals was found, these being presumably calcium sulphate. The acid rain water collecting on the upper flat side of the cornice, which was some 2 ft. 6 ins. wide, had slowly percolated through the stone, decomposing the calcium carbonate with formation of the sulphate, which was deposited by evaporation on the underside, thus bearing out the correctness of the general statement of the action of rain water containing traces of oxides of sulphur, resulting from the coal consumed in large towns.

Particular interest attaches, however, to the high percentage of silica in the crust. In the part insoluble in hydrochloric acid, the bases other than lime (which is entirely accounted for as sulphate) are in very small amount, and the silica is practically wholly in an uncombined condition. Of the total silica (2.72%) present in the original stone, about 17% was obtained in the soluble form on treating 50 grms. of the limestone with hydrochloric acid. It would appear then that the slightly acid rain water percolating slowly through the stone had brought about slow decomposition of certain silicates and the silica was left uncombined in admixture with the calcium sulphate, which formed over 80% of the crust.

#### THE RAPID ESTIMATION OF PYRIDINE BASES IN AMMONIA AND ITS SALTS.

BY T. F. HARVEY AND C. F. SPARKS.

The detection of pyridine or pyridine bases in ammonia solutions and in ammonium carbonate is frequently carried out by merely neutralising the greater part of the ammonia, and noting the odour of the slightly alkaline liquid. Some idea of the amount of the impurity may be formed from the intensity of the pyridine odour, but such a method can only serve for very rough purposes, or in the case when the ammonia under examination happens to be practically free from pyridine. No rapid and convenient method by which small quantities of pyridine or pyridine bases can be accurately estimated has been published, so far as we are aware.

In aqueous solution, François (*Comptes rend.*, 1903, 137, 324) this J., 1903, 1017) estimates quantities of about 100 mgrms. by preparing the aurichloride and igniting, while Labat employs *N*/20 bromine solution for the titration of decided quantities of pyridine, but the method is purely empirical and insensitive.

Pennock and Morton (*J. Amer. Chem. Soc.*, 1902, 24, 385) propose to neutralise 100 c.c. of ammonia solution with sulphuric acid, using methyl orange as indicator, and distil a portion of the resulting liquid. The distillate is cooled and precipitated with a slight excess of mercuric chloride, which removes all the ammonia as  $\text{NH}_2\text{HgCl}$ . The solution is filtered, and the pyridine titrated with *N*/10 acid in presence of methyl orange.

Milbauer and Staněk (*Z. anal. Chem.*, 1904, 43, 215; this J., 1904, 563) dilute 100 or 200 c.c. of ammonia with water and evaporate with an excess of dilute sulphuric acid nearly to dryness. The residual liquid is mechanically shaken with freshly prepared sodium bicarbonate and ether two or three times, the united ethereal extracts being shaken out with *N*/10 sulphuric acid and the excess of acid titrated in the presence of Patent Blue and sodium chloride. The method is said to be accurate.

Houghton (*J. Ind. Eng. Chem.*, 1909, 1, 698; this J., 1909, 1195), has described a method in which, after a preliminary distillation from slightly alkaline solution, the ammonia is destroyed by hypobromite, the unchanged pyridine being distilled into *N*/10 acid and titrated.

Other modifications involving distillation have been described, but none of the methods we have found is rapid, and the accuracy of some is very doubtful.

The method we have devised depends on the rapid and complete precipitation of pyridine or pyridine bases from an acid solution of the sample, which may be ammonia, ammonium carbonate, or other ammonium salt, as periodides, and subsequent titration of the sulphate of the bases with *N*/10 alkali. The use of iodine as a delicate precipitant for organic bases is well known, and has been used by Staněk (*Z. physiol. Chem.*, 1906, 47, 38) for the determination of choline and betaine. Choline is stated to form an enneaiodide which soon becomes crystalline, forming shining green rhomboidal leaflets, and the limit of precipitation for choline is said to be 1 part in two million. From solution in *N*/2 sulphuric acid we find that iodine will precipitate pyridine at a dilution of about 1 part in 20,000. At a dilution of about 1 part in 50,000, there is practically no precipitate formed. This is not sufficiently delicate for the purpose, but we find that the sensitiveness is greatly increased by the addition of sodium chloride to the solution. The salting-out action of sodium chloride upon atropine periodide was already known to one of the authors, and was discovered by him in conjunction with A. D. Powell during a previous investigation. In the presence of a sufficient quantity of sodium chloride, this action is so marked that 1 mgrm. of pyridine in 200 c.c. of *N*/2 acid (1 in 200,000) appears to be completely precipitated. It should be noted that the effect of working in strongly saline solution is in this case to cause precipitation of iodine itself even in the absence of organic base, and to this fact may perhaps be due the very complete removal of the base from solution. The periodides obtained under these conditions are almost certainly contaminated with co-precipitated iodine, which precludes the use of an iodometric method for their estimation. In the few cases where we have determined the iodine content of the precipitate, it has corresponded to something like 18 atoms of iodine per molecule of base. After precipitation and washing, the periodides are treated with a small excess of thiosulphate solution and then titrated, first in presence of methyl orange (methyl red is not available) and then with phenolphthalein, the differential titration being the basis of calculation.

The solutions required and the details of the method are as follows:—

*Special iodine solution.* Iodine, 13 grms.; potassium iodide, 13 grms. (more must not be used); water, to 100 c.c.

*Special wash solution.* 10-*N* sulphuric acid, 10 c.c.; water, 190 c.c.; special iodine solution, 10 c.c. Allow to stand overnight and filter out any precipitated iodine.

*Sulphuric acid* of about 10-*N* strength.

The indicator solutions actually used in the experiments were prepared as follows:—

*Methyl orange solution*, containing about 0.2 gm. of methyl orange in 1000 parts of water.

*Phenolphthalein solution*, containing about 1 part of phenolphthalein in 500 parts of 50% alcohol.

A stoppered separator capable of holding 300 c.c. or more, and having a tubulure above the tap that can be easily plugged with a small pledget of cotton wool, is used. This is dried internally or merely rinsed with strong alcohol and then



plugged with a small piece ( $\frac{1}{2}$  inch square should suffice) of cotton wool to serve as a filter. A conical ended glass rod answers well for inserting the plug, which should then be pressed down moderately, so that water will pass easily in a thin stream under the pressure obtainable from a small indiarubber hand-bellows. The tap is then closed, and 50 c.c. of water placed in the separator, followed by 50 c.c. of the ammonia to be tested (of sp. gr. about 0.885). 100 c.c. of 10-N sulphuric acid is then gradually added, the separator being cooled under the tap. The liquid then contains about 2.5% w/v of free sulphuric acid. 50 grms. of clean sodium chloride is next added, and when solution is complete and the contents are cold, 10 c.c. of the special iodine solution is run in. The separator is then shaken for some minutes till the precipitate aggregates and leaves the liquid fairly clear. After standing for about 15 minutes the liquid is forced through the cotton wool filter by means of an indiarubber hand-bellows, the tap being closed just before air reaches the plug. This filtration usually occupies about 5 minutes or slightly more. Twenty c.c. of the special wash solution is then added, and after the separator has been gently rotated, is forced out through the filter as before, but completely. A slight excess of saturated thiosulphate solution is then gradually added to the separator, with shaking, till all is colourless, the cotton wool being displaced either by shaking, or if necessary by means of a wire. If desired the excess of thiosulphate may be removed carefully by means of N/10 iodine solution, but this is not necessary unless too great an excess has been added. The total volume is now made up to about 20 c.c., two drops of methyl orange are added, and neutrality carefully established with N/1 sodium hydroxide, finishing with N/10 sodium hydroxide. 0.5 c.c. of phenolphthalein solution is then added and the pyridine sulphate titrated with N/10 sodium hydroxide (1 c.c. = 0.0079 gm. of pyridine or of pyridine bases calculated as pyridine).

It will be seen from the figures given below that although the quantities of base used in the experiments were small, varying from 5 to 50 mgrms., yet a considerable degree of accuracy was obtained. It is of course necessary if precise results are desired to exercise some care in the choice of the two end points. The correct end point in the methyl orange titration is attained when the colour changes from red to orange—one more drop should then produce the yellow colour. The phenolphthalein end point is a sharp one but is not permanent, and the first decided red colour (which lasts for about half a minute) should be taken. We have in no case found the small excess of thiosulphate used to interfere with the titrations, but as stated it can be removed if desired. It is possible to use glass wool well packed as a filtering medium and filtration is more rapid, but in that case a rubber stopper will be found necessary and the filter is much more difficult to displace from its position. The quantity of sulphuric acid used is intended to leave the solution about half normal in free acid, but considerable variation in this respect does not appear to influence the result. As will be seen, we have varied the acid between N/4 and N/1 and obtained the same figure. Larger quantities than 50 mgrms. of base have not been tried, but probably the method is capable of extension, using proportionately larger quantities of iodine solution.

#### Experimental.

The first series of experiments was made using a dilute solution of crude pyridine bases (a)—the latter had sp. gr. 0.983 and 95% distilled between 90° and 142° C. Titration to methyl orange gave 82% and to phenolphthalein 81.7% calculated as pyridine.

Pyridine bases taken.	c.c. N/10 soda to phenolphthalein after pptn. from 200 c.c. liquid.	% recovered base calculated as pyridine.
gm.		
0.005	0.53	85.5
0.01	1.05	82.9
0.02	2.1	82.9
0.03	3.15	82.9
0.04	4.2	82.9
0.05	5.3	83.5
	Theory =	81.7

Another sample of crude pyridine bases (b) was used; this distilled at 100°–140° C. and titrated to methyl orange as 85% and to phenolphthalein as 84.8% pyridine.

Pyridine bases taken.	c.c. N/10 soda to phenolphthalein after pptn. from 200 c.c. liquid.	% recovered base calculated as pyridine.
gm.		
0.005	0.52	82.1
0.01	1.1	86.9
0.02	2.2	86.9
0.03	3.18	83.7
0.04	4.2	82.9
0.05	5.3	85.5
	Theory =	84.8

A sample of ammonia was then taken which had been found to contain 0.0103, 0.0103% of bases calculated as pyridine. Pyridine base (a) was added in amount corresponding to 0.02% pyridine. The total pyridine found was 0.0302% (theory 0.0303%). To another sample of ammonia containing 0.022%, 0.023% of pyridine, was added pyridine base (b) equivalent to 0.031% pyridine. The total base recovered was then 0.055% (theory 0.056%).

A series of experiments was carried out with a sample of nearly pure pyridine of sp. gr. 0.986, which titrated 95.9% to methyl orange and 96.1% to phenolphthalein.

Pyridine bases taken.	c.c. N/10 soda to phenolphthalein after pptn. from 200 c.c. liquid.	% recovered base calculated as pyridine.
gm.		
0.005	0.62	93
0.01	1.21	95.6
0.02	2.45	96.8
0.03	3.64	95.9
0.04	4.85	95.8
0.05	6.15	97.2
	Theory =	96.1

Some experiments were then made with varying quantities of acid, using the same sample of ammonia throughout.

Normality of acid before pptn.	% Pyridine found.
Approximately N/4	0.0103
" N/2	0.0103
" 3N/4	0.0103
" N/1	0.0103

Two further samples of strong ammonia solution were examined, and gave respectively 0.028% pyridine (four estimations) and 0.01% pyridine.

An experiment in which the salt was purposely omitted gave only 0.0087% pyridine instead of 0.028%, showing that the ammonium sulphate formed did not of itself ensure precipitation of the pyridine.

Some samples of ammonium salts were tested with the following results:—

	Pyridine %
Ammonium carbonate	0.024
" chloride	0.003
" sulphate	0.010
" nitrate	0.013

Finally, in an attempt to prepare a solution of ammonium sulphate entirely free from pyridine, 100 c.c. of ammonia containing 0.028% pyridine was diluted, neutralised, and distilled from slightly alkaline solution as recommended by Houghton; 25% was distilled and the two portions were examined. The pyridine was then found to be distributed as follows:—

	Pyridine %
In the distillate .....	0.0095
In the residual liquid .....	0.0175
Total .....	0.0270

The retention of about two-thirds of the pyridine in the distilling flask seems to throw considerable doubt on methods involving separation by distillation.

The authors are indebted to Messrs. T. Morson and Son, Ltd., in whose laboratories this investigation was made.

#### DISCUSSION.

The CHAIRMAN said that the authors were to be congratulated upon having devised a means of separating the pyridine previous to its actual estimation. Such a method of work was likely to give the most satisfactory results. He suggested that the authors might find methyl iodide an effective reagent for getting the ammonia free from pyridine as the pyridinium iodide formed was a very characteristic compound.

Mr. A. CHASTON CHAPMAN said that he had on various occasions found himself confronted with the problem of determining traces of pyridine both in ammonia solution and in liquid ammonia. In the latter case its presence was particularly objectionable, owing apparently to its injurious effect on certain parts of refrigerating machinery. The method he had adopted was to concentrate the pyridine in the manner referred to by the authors and then to titrate the pyridine and residual ammonia, using different indicators. He had never felt very happy about the results but the method was, so far as he was aware, the best available. Pyridine formed a good many more or less insoluble compounds with metallic salts, but such experiments as he had made were not very successful, owing to the need for concentrating the pyridine and to the very small quantities of that substance usually present.

Mr. HARVEY, in reply, said that they had not endeavoured to obtain ammonia free from pyridine by the agency of methyl iodide. The accuracy of the figures had surprised both Mr. Sparks and himself in view of the very small quantities worked upon. The periodides were precipitated very completely in the presence of sodium chloride, but in the absence of the latter only partially. They had not isolated pyridine periodide, but this had previously been done by Dafert, who gave it the formula  $C_5H_5HI_3$ .

But the endeavour of the engineer and metallurgist to express in values some definite property, useful for their special needs and related to hardness, has caused them to give a meaning to the term not understood or used by mineralogists or laymen. To these it means simply that property which denotes resistance to penetration; while to the engineer it may mean abrasive, cutting, elastic, or tensile hardness. It is quite right that the engineer should endeavour to find simple tests that shall indicate to him the behaviour, in actual use, of the material under consideration, but he causes confusion if he groups all the different properties which he so uses under a term, such as hardness, to which a definite meaning is already appropriated. The object of these notes is to discuss the right use and application of the methods at our disposal for testing hardness. Many of the tests are of great importance in indicating specific properties, and efforts should be made strictly to limit the use of these tests to those instances where practice has upheld their utility. It would then naturally follow that instead of the results being loosely termed "Hardness numbers" they would be given a nomenclature indicating the property determined.

The methods used may all be grouped as:—

- (1) Indentation tests, whether (a) gradual, or (b) sudden.
- (2) Abrasion or scratch tests.

Though Wahlberg extends this classification to a third, in which he places Foepppl's method, the latter should rightly be placed under indentation tests. These depend on the permanent distortion of the material, under pressure, by means of a hard steel ball, cone, or knife edge; and the abrasion or scratch tests on the material removed from the surface during sliding contact with another body of such superior hardness that its surface remains unimpaired by the action.

As the tests that have been introduced are so numerous it is impossible to note more than two or three in each group, and attention will therefore be given to the following:—

- (1) *Indentation tests.* (a) Brinell test. (b) Shore's scleroscope. (c) Pellin test.

- (2) *Abrasion or scratch tests.* (a) Turner's sclerometer. (b) Martens' sclerometer. (c) Saniter's abrasion test.

*Brinell test.\** Brinell's machine is most widely used to-day. In this method a hardened steel ball, capable of resisting deformation, is pressed into the face of the metal to be tested. The spherical area of the indentation being calculated, and the applied pressure being known, the stress per unit area may be determined, thereby giving the hardness number. The results are, within limits, independent of the size of the ball and the pressure applied. The sample for test need only be small, and the actual operation occupies two or three minutes to perform. Two types are in common use: one of Swedish make known as the Aktiebolaget Alpha—a ball test; and the Martens, in which a conical indentation is produced and the pressure exerted by means of water and not weights as in the former. The Martens machine finds support in that the cones can now be prepared with sufficient accuracy to secure consistent results, and the depth of conical indentation possesses advantages in calculating. In the ball-test a 10 mm. ball is most commonly used with a weight of 3000 kilos. for steels, and 1000 kilos. or even 500 kilos. for brasses and comparatively soft alloys. In investigations of a particular class of

## Newcastle Section.

Meeting held at Bolbec Hall on Wednesday,  
December 12th, 1917.

MR. H. PEILE IN THE CHAIR.

## NOTES ON TESTING HARDNESS OF METALS.

BY J. W. CRAGGS.

In the mechanical testing of metals the determination of hardness is of considerable importance, because of the comparative reliability of the results and the ease with which it may be carried

\* Rapp, Congr. intern. Méthodes d'Essai. Paris, 1900. Wahlberg. Journ. Iron and Steel Inst., 1901, I., p. 243.



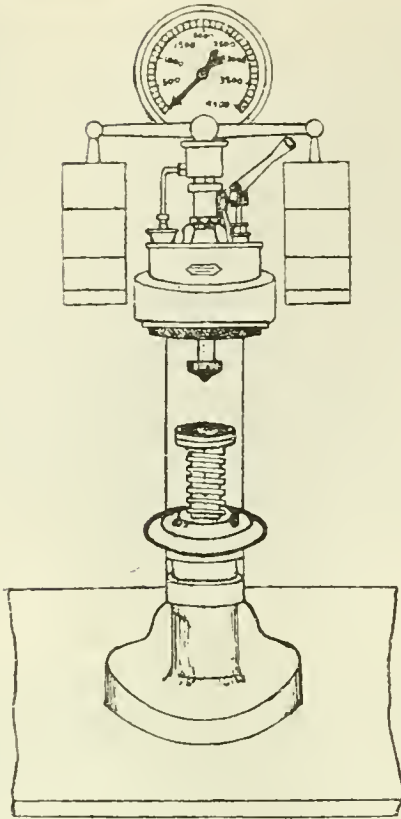


Fig. 1.  
Brinell test.

material it is advisable to use the same sized ball and equal pressures throughout where comparisons are being made.

*Shore's scleroscope.\** In this instrument a small steel cylinder or "hammer," with a hardened or diamond point, is allowed to fall from a height of about 10 inches upon the polished surface of the metal to be tested. The hardness is measured by the height of the rebound and for this purpose the tube is graduated into 140 divisions so that hardenite gives a reading of approximately 100. It follows that readings exceeding 110 are only given by materials of unusual hardness. One advantage possessed by Shore's scleroscope is its convenience. It is small and may readily be carried for the testing of any sample, large or small. The indentation produced might pass unobserved and could therefore be made on a finished article, for the area of the indenting point is stated to be only  $\frac{1}{2500}$  of a square inch. An illustration of the scleroscope is given in the *Journal* for 1915, page 536.

*Pellin test.* This, at first sight, appears to be a combination of the preceding tests; it is, however, based on the Brinell method and has been designed for the testing of the different metals used for all industrial purposes. It is essentially one in which the indentation is produced by a falling bar of known weight, having at the lower end a steel ball 2.5 mm. in diameter. The apparatus is arranged so that instant release may be obtained by an annular electro-magnet carried by a housing in

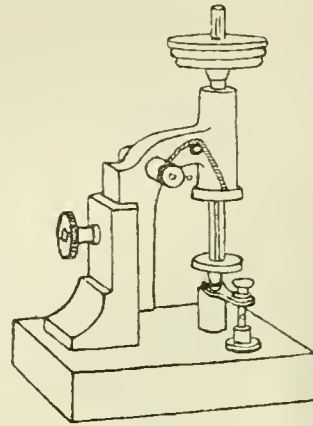


Fig. 2.  
Pellin test.

which the bar slides freely. When in position for use the bar is retained there by the magnetic attraction of the soft iron collar, fitted to it, to the magnet on the housing. The diameter of indentation may be varied by means of weights placed on the top of the bar or by altering the distance of fall. When the impression has been made its diameter is measured and, knowing the weight used, the hardness number may be calculated.

*Turner's sclerosometer.\** By this device a standard scratch is produced on the polished surface of the metal. A weighted diamond point is drawn once forward and once backward, and the hardness number is the weight in grms. required to

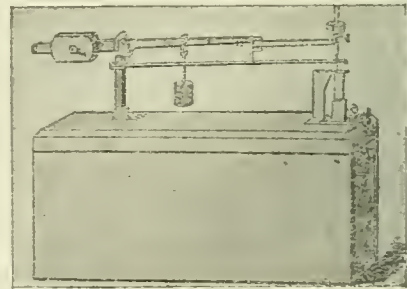


Fig. 3.  
Turner sclerosometer.

produce the standard scratch. Turner describes this as one just visible to the naked eye as a dark line on a bright reflecting surface, or as a scratch which can just be felt with the edge of a quill when the latter is drawn over the smooth surface at right angles to a series of such scratches made by regularly increasing weights. Of the many forms of sclerosometer it is that most generally used.

*Martens' sclerosometer.* Here the scratch is varied and the weight is kept constant. The usual load is 20 grms. and the point is stationary, whilst the specimen is drawn backward and forward by a traversing table. The determination is made by a careful measurement of the width of scratch, on the horizontal polished surface, by means of a microscope with micrometer eyepiece, or by photographing the scratch under a known magnification. The hardness number is the reciprocal of the width of the scratch in millimetres.

\* A. F. Shore, *Scleroscope*, Amer. Mach., 1907, 30, Part 2, p. 747.

\* Turner. Proc. Birmingham Phil. Soc., 5, Part 2.

*Saniter's test.\** Devised particularly for the determination of the wearing property of rail steel, this method attempts a reproduction of the actual stresses encountered in use. The test-

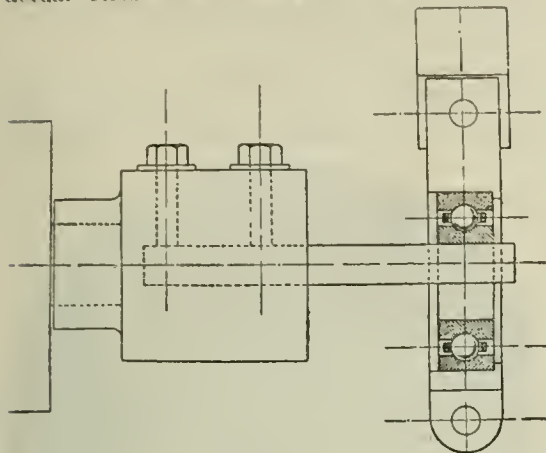


Fig. 4.  
*Saniter test.*

piece is 5 in. long by 0.5 in. diameter and is fixed in a chuck which revolves at 4000 revs. per minute. Near the free end the test-piece rotates by friction the inner ring of a ball bearing which is loaded to produce a pressure equal to 205 lb. at the point of contact. The length of this contact section is  $\frac{1}{4}$  in. and the total number of revolutions made by the test-piece 200,000. One test occupies about 50 minutes. The wear number is the ten-thousandth parts of an inch removed from the diameter of the bar during the test. To ensure accuracy Saniter recommends that the test-piece be measured in three places, and also in a plane at right angles, making six measurements in all, the average being taken as the correct number. These are all recorded before as well as after the test, for even in the original test-piece slight irregularities may occur. The smallest wear number represents the least wear, and consequently the best wearing steel. The test-pieces are always taken in a longitudinal direction relative to the actual rail.

#### *Value of the tests in practice.*

Confidence in the Brinell method for constructive, ductile materials, as a measure of the tenacity, is now general, as evidenced by its extensive use in engineering practice. It is very easily applied and when used correctly may be said to give definite results. Adopted originally for ordinary carbon steels, its application has been extended to special steels, and to alloys of the non-ferrous type, and if data be collected for each class of material the results can be used with certitude. McWilliam and Barnes† have examined steels of varying carbon content in the presence of 2% chromium and 3% nickel, and also with 0.2% vanadium. In all three types of steel they found the factor for the conversion of the Brinell number to the maximum stress to vary, though the variation was small. Their factors were chrome steels, 0.242; nickel steels, 0.239; and vanadium steels, 0.235. From these it may be observed that where a particular type is being regularly handled the factor may be determined and constancy obtained, whatever the carbon content or physical condition.

Knowledge of the material is an important reservation that one must note in seeking to apply the method. The general application to all alloys—ferrous and non-ferrous—without definite information of the type, involves liability to dis-

crepancies, e.g., with a cast cartridge metal, which is entirely  $\alpha$ , the factor is in the neighbourhood of 0.28–0.30; while with a brass of the Muntz metal class, in which there are practically equal portions of  $\alpha$  and  $\beta$ , it is from 0.25–0.27, and, in the case of an all  $\beta$  brass, approximates to 0.24–0.25. Such figures, of course, are only directly applicable to the copper-zinc series of alloys. The addition of a third element, say aluminium or manganese, to adjust the structure so as to give a particular type, would necessitate further investigation in the determination of special characteristics even though the micro-structure be identical with that of a pure copper-zinc alloy. With samples in which the structure is so small that numerous grains are included in the impression made by the ball, their different orientation will eliminate variations in the test, but with large grains this is not so. Appreciable disturbance is caused, so that in samples which possess a coarse structure, whether of a simple or complex nature, the character of the impression will depend upon the orientation of the crystal grain on which such an impression is made. Actual determinations made on two adjacent crystal grains gave unsymmetrical impressions of different forms that could not be measured. Similarly with a test on the crystal boundary the irregularities were such as to prohibit any comparison being made. A photograph of such an impression is shown in Fig. 5. We may conclude that metals and alloys possessing a coarse structure will tend to give unreliable results and may even be such as to prevent an approximation being made. This irregularity in the indentation is not usual with all soft or ductile materials, though other workers have had at times a similar difficulty with the ball

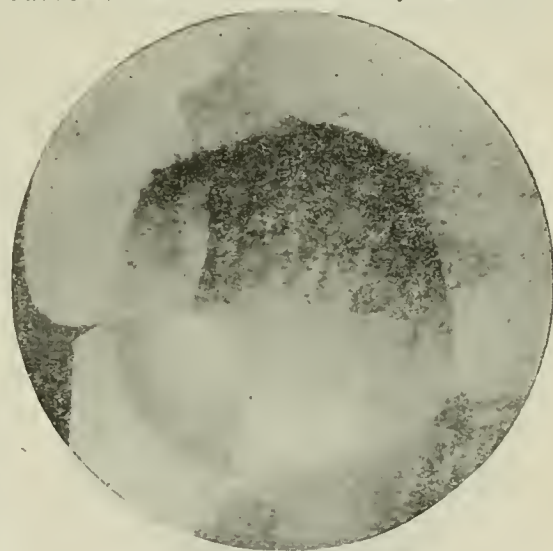


Fig. 5.  
*Large  $\beta$  crystals of Cu-Zn showing irregular Brinell indentation,  $\times 15$ .*

test. It was in this instance probably accentuated by the coarseness of the crystals, resulting in a condition in which slipping within the crystal was more easily induced.

With hard and brittle materials the Brinell test is of much less value. This fact is obvious if one thinks of the determination as giving, in a sense, a measure of the viscosity of the solid, for flow must take place. The indentation due to the pressure by the ball or cone is produced by compression, shearing, and translation along gliding planes. In a ductile body this readily occurs, as seen in photomicrographs Nos. 1 and 2, Fig. 6, in which the elongation of the crystals in an  $\alpha$ -brass

\* Saniter. *Journ. Iron and Steel Inst.*, 1908, III., p. 73.

† *Journ. Iron and Steel Inst.*, 1915, 91, 125.



is clearly shown, with the presence of numerous slip-bands. With a hard alloy fracture may occur at once or after a short interval, the crack or cracks radiating from the point of test.

Again, in testing hard material cracking may not take place and yet the determination may be unsatisfactory, for the balls, which contain approximately 1% of carbon, 1.5% of chromium, 0.2% of nickel, and are oil-quenched from 800° C., only possess a limited rigidity. When distortion of the ball is observed the impression made by it will not give a true figure. To show the unreliability of tests in which the Brinell number is recorded as 730 to 750, and in some instances still higher, chrome, nickel-chrome, nickel-chrome-vanadium, and high-speed steels were hardened and determinations made. Several balls were tried and in each case there was distinct flattening of the ball. The highest figure recorded was 653. In many instances, as will be noticed below, the maximum reading was 625.

Apart from these limitations, this test may be rendered valueless by careless manipulation, especially in regard to the time factor. Thomas\* has shown that differences may be recorded by varying the time of application of the pressure up to 8 or even 10 minutes, when testing mild steel. The differences, however, are only slight after one minute and he therefore urges the adoption of this as the standard time for the test. The thickness of the sample must also be such that stresses set up by the ball are dissipated before penetrating throughout the full depth of the piece. Many experiments led him to declare that 0.38 inch should be the minimum when a normal load of 3000 kilos. is used. If the thickness chanced to be from 0.15 or 0.20 to 0.38 inch the load should be less, say 1000 kilos., for satisfactory results; while an

possible is very small for each annealing, while with others a considerable amount of work can be done. From this fact it may be observed that where large indentations are made through excessive loading, the hardness numbers may be misleading. A standard load should be adopted for each individual class of alloy.

In using the Shore scleroscope some property other than the tenacity is indicated. If in falling the "hammer" produced no indentation a true measure of the elasticity would be given, but in

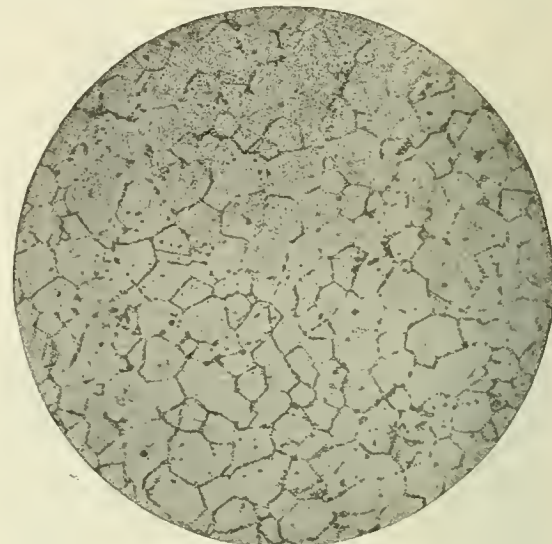


Fig. 6, No. 2.

*Cartridge metal—Stressed and unstressed.  
By Brinell ball,  $\times 100$ .*

practice this does not occur. Yet the penetration is so small that the result gives usually an approximate measure of the elastic limit. The figures are of great value when the nature and properties of the alloy under examination are familiar; or if a measure of the cold-work that has been applied is desired, for the test exhibits most strongly the "hard worked" or "écroui" condition. Probably this is due to the more rapid increase in the elastic limit than in the maximum stress, when work is applied, for the Brinell number does not increase at the same rate. It is because of this characteristic of the scleroscope test that many investigators find no proportionality between it and the Brinell test.

Generally, in the cast or normal condition a rough comparison of the Brinell and scleroscope numbers may be made by the use of the following factors, which have been calculated from hundreds of tests performed:—

Ordinary carbon steel .....	Scleroscope No. $\times 6.67 =$	Brinell hardness No.
Nickel and nickel-chrome steels ..	" " $\times 7.0 =$	" " "
Cast iron and bronzes .....	" " $\times 5.25 =$	" " "
Aluminium and light alloys .....	" " $\times 6.0 =$	" " "

It must be understood, however, that these factors should not be used carelessly; a knowledge of the physical condition of the metal is essential. In the paper by McWilliams and Barnes, already cited, it is declared that there is no constant factor for the scleroscope and Brinell tests in alloy steels. This conclusion is perhaps the better for scientific purposes, for each test is a measure of different properties. The above factors exhibit the general trend of the results and are only of value if correctly applied.

Experiments made with a view to determining

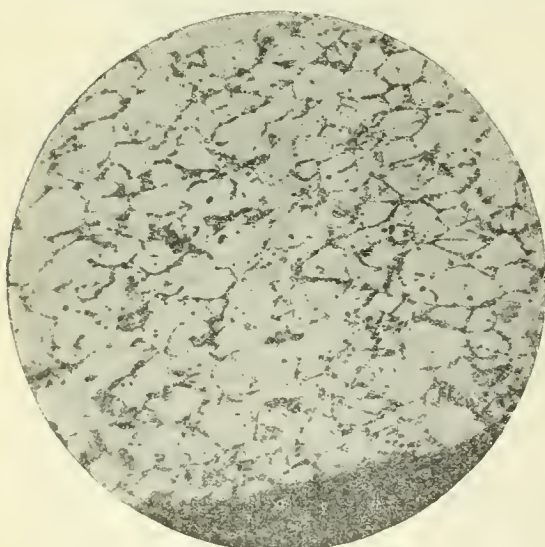


Fig. 6, No. 1.

*Cartridge metal—Stressed and unstressed.  
By Brinell ball,  $\times 100$ .*

interesting series of tests with thin pieces backed first by hard steel, and secondly by wood, showed that it was essential to support the sample by material as hard as or harder than the test-piece, otherwise the results were variable.

Many apparently strange results may be explained by the differences in the amount of cold-work that will produce "overstrain" in various alloys. With certain of the high tenacity light alloys, for example, the amount of cold-work

\* J. Iron and Steel Inst., 1916, I., p. 255.

the difference shown in the hardness by the Brinell, scleroscope, and simple scratch tests on a sample of nickel-chrome steel gave the following interesting results:—

or pass the tool on the figure. The cutting efficiency depends upon the stability of the constitution of the hardened steel over a wide range of temperature and is not related to the Brinell

Quenching medium.	Temp.	Brinell.	Sclero- scope.	File test.	Glass test.	Rose quartz test.
1. Rape oil .....	800	625	80	No mark.	Scratched with ease.	With difficulty.
2. Cold water .....	800	625	80	"	"	With great difficulty.
3. Boiling water .....	800	625	80	"	"	"
4. Contact with cold iron slab .....	800	625	80	Just bites.	"	With ease.
5. Strong air blast .....	800	625	77	No mark.	"	With difficulty.
6. Spray of cold water .....	800	625	77	Great difficulty.	"	"
7. Concentrated sulphuric acid .....	800	625	74	No mark.	"	"
8. Spray of water; face of sample at 800° C. with core at 600° C.	800	625	75	Great difficulty.	"	Just marks.

The "file hardness" test was made with a new 8-inch dead smooth feather-edged file for each test. The "glass hardness" test was applied so that the edge of the sample was drawn over the face of a piece of highly-polished plate glass. The "quartz" test was made in a similar manner to the file test, with crystals of rose quartz.

These results show no variation in the Brinell numbers and practically none in the scleroscope figures. The former test indicates practically the maximum obtainable by such a test and in each case a new ball was used. The scleroscope test, being an indication of the resilience or elastic hardness, shows slight variations, though test No. 4 is remarkable as exhibiting no change, while the quartz scratched the sample with ease. No doubt the hardness is best measured by resistance to penetration, but the actual value varies according to the mode and rate in which the test is made. Each test has its own definite value, as suggested above, and should be applied accordingly.

Sufficient data have not yet been collected to determine the true value of the Pellin test, and no generalisation can be made with any certainty. The principle on which the test depends, its simplicity, and the rapidity with which it may be carried out, suggest for it an important future. The smaller indentation gives probably an advantage over the Brinell method.

The sclerometer methods mentioned give better values than the above tests of the absolute hardness of the material. But, as Howe has mentioned, considerable difficulty is encountered in registering the dimensions of the scratch after it is obtained; while Turner confesses that the standard scratch is only definitely recognised after some experience. From the standpoint of works value the Brinell, scleroscope, and Keep's drill tests are the best for manipulation; yet the sclerometer should be used for its intrinsic worth. The penetration hardness is a property not indicated or measured by any of the others.

The abrasive hardness is now measured by Saniter's test. He has shown the lack of uniformity between the wearing properties and the Brinell hardness numbers. The test follows more closely than the others, in a simple fashion, the actual stresses the material will have to withstand in practice. A good example of the need of such a test is shown in the case of Hadfield's manganese steel. With a Brinell number of about 200 it would (if judged by a standard Brinell figure) be declared soft, yet it withstands abrasion excellently and is specially suited for work where first-rate wearing properties are essential.

Another instance in which some test other than the Brinell is necessary is in the determination of the cutting efficiency of tools. Inspectors often stipulate a standard Brinell hardness and reject

number in any way. A Brinell number may be as specified and the tool fail in practice.

The Brinell test gives a measure of the tenacity; the scleroscope an approximate measure of the elastic limit; the sclerometer gives the penetration hardness; while the Saniter test indicates the wearing properties. The determination of the machining hardness, the cutting efficiency, etc., must be conducted by other means. If better terms for the different hardnesses could be adopted to prevent overlapping and confusion in the tests, there can be no doubt that greater progress in their development and more extended use would follow. I suggest that with this object the following be adopted: for Brinell hardness, the "tenacity number"; for scleroscope hardness, the "resilience number"; for sclerometer hardness, the "penetration number"; and for the Saniter test, the "abrasion number." The terms are perhaps not the best that could be devised, but something along the line suggested should be attempted.

An excellent bibliography, chronologically compiled, is given with the Report of the Research Tests Committee in the Proceedings of the Institute of Mechanical Engineers, Oct.-Dec., 1916. This Report, with the discussion and correspondence, makes a very valuable contribution to the subject of hardness testing.

My thanks are due to Messrs. Sir W. G. Armstrong, Whitworth and Co., Ltd., for permission to publish the results here given; to the Chief of the Department, Mr. O. Smalley, and to my colleague, Mr. R. N. Richardson, for their assistance in the preparation of the paper.

#### DISCUSSION.

Professor LOUIS expressed his indebtedness to the author for his endeavour more accurately to define the term "hardness." The engineer and metallurgist were often engaged in work which overlapped, and although each might use the same word or term, yet the meaning which each attached to that word might not be quite the same. Any attempt to clear up such discrepancies was always valuable. Many years ago, before the modern appliances for determining hardness had been brought to their present state, he had been engaged in comparing the hardness of chilled castings, in the production of which he was then engaged; he had used the simple file test, and provided that he knew the other constituents, he could fairly well predict the percentage of phosphorus. His greatest difficulty had always been with case-hardened metal. He believed that of all the modern methods, Shore's scleroscope gave the most accurate results with this class of material; in this case it certainly was better than the Brinell test, but this part of the problem was very difficult.



The whole subject of hardness was an exceedingly complex one. With regard to the term "hardness," he preferred that it should be kept to its original use, *i.e.*, as used by mineralogists. In using Turner's sclerometer, very strange results were often obtained with soft materials—as an exaggerated example, say, indiarubber. He thought that this was largely owing to the fact that before the scratch was commenced, the diamond point had first of all to penetrate into the metal. He had often wondered if it would not be better to use a rapidly revolving cylindrical test-piece and steadily to press the diamond point against the metal under well-defined pressure. In that way more accurate results might be obtained.

Mr. W. H. YOUNG said that although he was constantly using both the Brinell and Shore tests he had found them to be of comparatively little use in testing gun-metals, cast iron, and phosphor bronzes when the object of the test was to compare them as regards their use as bearing metals.

Mr. CRAGGS, in reply, pointed out that the introduction and development of many of the tests commonly used to-day, were of very recent date. Many of the old-fashioned foundry tests that had been handed down from father to son were still practiced, but a saner view was now being adopted which tended to greater uniformity in results. The difficulty to be faced now was in the correct use of the modern tests and in their interpretation. Still further information was needed. Shore's scleroscope would be the best instrument to use with case-hardened samples, since the elastic limit and the maximum stress were almost the same in such material. In very thin case-hardened samples, the soft interior would lead to erroneous results with the Brinell test, as was shown in Thomas' work, where he referred to the backing or supporting of thin samples with hard steel and with wood. He quite agreed that the term "hardness" should be limited to that used by mineralogists. The suggestion as to a revolving test-piece in conjunction with Turner's sclerometer was worth further investigation, though perhaps Keep's drill test was essentially similar to the modification of Turner's test as suggested by Professor Louis. He quite agreed with Mr. Young, but he (the author) thought he had fully indicated this by his references to Saniter's abrasion test as being the only test suitable for metals that had to withstand wear. An indication of the carbon content of ferro-chrome, where only the carbon was variable, could no doubt be obtained by the adoption of a suitable hardness test, but this would be entirely dependent upon whether a polish could be given to the sample. He agreed that wear tests produced a worked condition in the sample at the point of contact. Sir Robert Hadfield had proved this in regard to manganese steel and had given figures showing the increase in the Brinell and scleroscope readings for materials quenched in water from 950°C. and air-hardened. The suggestion put forward for the testing of tools for hardness at temperatures attained by the tool in use, had not occurred to him, but was worth following up. The futility of testing at room temperature alone must be apparent to all.

## Nottingham Section.

### DISCUSSION ON NITRE CAKE.

Mr. G. E. GODBER has communicated the following in connection with the discussion of November 14th last on nitre cake (see this J., 1917, 1216 A):—

In using nitre cake with common salt for the

production of hydrochloric acid and saltcake, in a mechanical furnace at about 600°C., I have to deal with three sources of supply. 1. Made on the works. Average  $\text{H}_2\text{SO}_4$ , 32%. 2. From one of H.M. Factories. Average  $\text{H}_2\text{SO}_4$ , 31.0%. 3. Chemical dyewares. Average  $\text{H}_2\text{SO}_4$ , 29.5%. I have never noticed such a wide difference in the acidity as Mr. W. G. Timmans quotes in any delivery. In our own make each still residue is tested separately every day. The widest difference noted in these samples was 6%, due to extra acid having to be used to facilitate the running off process. The maximum difference in the four stills is roughly within 3%. Mr. G. C. Grisley does not say what he considers "standard quality" in saltcake. I should like to know if the following represent anything near his ideal working conditions. The average tests of 696 samples taken at regular intervals on each shift and representing together something like 1250—1300 tons, show acidity as  $\text{SO}_3$ , 1.66%; sodium chloride, 1.81%. About 28 to 30 tons of nitre cake is used per week. I have not had much trouble in regulating the conversions providing the heat remains good. If even charging and a good temperature are maintained, the control of the conversion is greatly facilitated. The pieces of saltcake seldom come from the furnace larger than a pea. I have never had any trouble with nitrous gases.

*Meeting held at Nottingham on Wednesday,  
February 23rd, 1916.*

MR. JOHN WHITE IN THE CHAIR.

## THEORY OF THE COAGULATION OF HEVEA LATEX.\*

BY M. BARROWCLIFF, F.I.C.

(*lately First Assistant Chemist, Department of  
Agriculture, Federated Malay States*).

Natural coagulation of rubber latex, unless special conditions are observed, is very slow and often incomplete, and is accompanied by putrefactive changes, causing the development of an offensive smell and discoloration of the coagulum. Whitby, in 1912, in a communication to the Congress of Applied Chemistry, mentioned that in sealed vessels which had been kept for some time coagulation had occurred without decomposition; but no significance was attached to the observation and it was not until two years later that M. Maude and W. S. Crosse, manager and engineer respectively of the Cicely Rubber Estate, discovered that this could be made the basis of a practical method for coagulating latex.†

Two theories have been advanced to account for the phenomenon by which rubber is produced by the natural coagulation of latex, without the addition of any coagulating agent. Whitby (*loc. cit.*) suggested that natural coagulation was brought about by a coagulating enzyme. This theory was based on the observation that latex can be prevented by heat from coagulating within the usual time, and that only after three or four days does coagulation, accompanied by putrefaction, again set in. He concluded that this excludes bacterial action, since natural coagulation commences within a few hours of tapping, and bacterial infection must have taken place subsequent to the latex leaving the tree.

\* The substance of this paper was communicated to the Nottingham Section in February, 1916. The whole of the experimental evidence obtained is given, although publication of parts of it has been anticipated by Campbell (this J., 1917, 274), whose conclusions are in accordance with those here expressed.

† Now being worked in the F.M.S. under the name of the "M.C.T." process.

In 1915, Eaton and Grantham\* advanced the view that the cause of natural coagulation is bacterial and not enzymic, and pointed out that the heating to which Whitby subjected his latex may have created conditions favourable to the development of anti-coagulating bacteria at the expense of the coagulating species.

The present writer, in 1914, carried out some experiments on the same problem and obtained results, hitherto unpublished, which supported the enzyme hypothesis, though not absolutely excluding the possibility that a bacterium capable of coagulating latex may exist also. Before describing these experiments it will be convenient first to discuss the evidence brought forward by Eaton and Grantham.

The following account is quoted from their paper:—

"Experiment 1.—Three flasks, plugged with cotton wool, containing fresh latex, were (in about 1 or 2 minutes) rapidly heated to 150° C. in a steam autoclave and slowly cooled. A slight amount of coagulation occurred, but most of the latex remained liquid. One flask, A, was opened, another, B, had a little fresh latex placed in it and was then resealed, and the remaining one, C, was left untouched. Twenty-four hours later the latex in flasks A and B was nearly completely coagulated and a yellow scum was forming on the surface. The latex in flask C was unchanged and has remained so. The flasks A and B gradually developed all the characteristic putrefactive changes. In both these cases coagulation apparently runs parallel with putrefaction.

"Experiment 2.—Three similar flasks containing latex were taken and heated in the autoclave at 90°–100° C. Two, D and E, were left untouched, and the third, F, was inoculated with a little fresh latex. On the day following partial coagulation had taken place in all three flasks. In D and E there was no trace of putrefaction. D was opened and the serum was found to be slightly acid and sweet smelling. In F a yellow scum had formed and putrefactive changes had begun. One day later E and F were completely coagulated but the putrefaction of F was more pronounced than that of E. A day later E began to develop a yellow scum and show signs of putrefaction. In both experiments there were control flasks which were not sterilised, in which the latex coagulated almost completely in the first 24 hours, formed a yellow surface scum and began to putrefy. Both the latex heated to 150° C. and 100° C. were afterwards readily coagulable with acetic acid.

"It will be seen that the results in Experiment 1 agree with Whitby's except perhaps that putrefaction was slightly more rapid. The rapidity varies greatly, however, in different experiments."

The authors attribute the coagulation occurring in D and E (Experiment 2) to a non-putrefactive bacterial change, the bacteria developing from the spores not killed by the heating to 100° C., and this received support from the fact that under conditions unfavourable to putrefactive bacterial changes coagulation could be obtained. In the presence of a small quantity of dextrose or other sugars complete coagulation occurred within 18 hours, and no putrefaction was noticed. In these instances a considerable quantity of carbon dioxide was evolved.

Several criticisms can be made against Eaton and Grantham's sterilisation experiments. In the first place the small amount of coagulum stated to have been formed during the heating may have occluded a quantity of enzyme and protected it from further action of the heat. This

enzyme would gradually diffuse out and subsequently bring about the coagulation that was in Experiment 2 actually observed.

Furthermore, it seems probable that the temperatures recorded in Experiment 1 are those of the autoclave, not of the latex.

It may seem difficult to explain, without adopting the bacterial theory, why of the two sterilised flasks the opened one coagulated whilst the untouched one did not, but in absence of more precise knowledge of the conditions of heating it may quite conceivably be supposed that in the one case enzyme was protected from destruction by the formation of a thicker clot of rubber than in the other. The following experiment, moreover, carried out by the present author, gave an entirely different result:—To 100 c.c. of rapidly boiling water was added slowly from a burette 100 c.c. of undiluted latex from old trees, the mixture being vigorously boiled throughout the addition. By this means sterilisation was effected without trace of coagulation. The resulting solution was divided into two portions and to one was added a drop of fresh latex. Both were left overnight in uncovered beakers. The following morning the solution that had been inoculated with the fresh latex had, to a large extent, coagulated, to the same degree in fact as had an unsterilised control, whilst the other portion remained perfectly liquid. It continued to have a slightly acid reaction and until the third day there were no signs of putrefactive fermentation having set in. The room in which the experiment was conducted opened out on to a rubber plantation, and there was no reason why this latex should not have become infected with the coagulating bacteria, did such exist, equally as quickly as that issuing from freshly-tapped trees. The contention that conditions suitable for anti-coagulating bacteria may have been produced cannot be sustained since the latex inoculated with fresh latex coagulated.

Whitby's conclusion that natural coagulation is attributable to an enzyme must therefore be considered valid.

Further evidence in support of the enzyme theory was then sought for. It was found that coagulation in closed vessels took place completely in normal time in the presence of toluol, the bactericide recommended by Emil Fischer as being non-toxic to enzymes. Thymol similarly did not inhibit or retard coagulation. On the other hand, hydrocyanic acid, which is fatal to nearly all enzymes, completely inhibited coagulation.

Another interesting fact ascertained was that latex sterilised in the manner described did not, contrary to that sterilised by Eaton and Grantham, behave normally when treated with the normal minimum quantity of acetic acid used in coagulating. No coagulum was formed, only, and very slowly, a flocculent, non-coherent mass. When, however, a trace of fresh latex was added, followed by the acid, a perfectly normal coagulation took place. This experiment not only supports the enzyme hypothesis, but indicates that the action of acetic acid on latex is to be considered as one of enzyme activation, and that it is not the acid *per se* which produces coagulation. This hypothesis will explain an observation made independently by Eaton and Morgan, who found that the addition to latex of a considerable quantity of strong hydrochloric acid resulted in no coagulation taking place; it may be assumed that the acid added was sufficient in quantity to destroy the enzyme.

Further support for the view that coagulation is enzymic is afforded by the fact that it was found to be greatly accelerated by the addition of very small quantities of calcium salts, a property common among enzymes. The addition of a

\* Agricultural Bulletin, Fed. Malay States, Nov., 1915.



soluble calcium salt such as calcium sulphate or calcium chloride, even in as small a quantity as 0.002% of calcium, to latex contained in a closed bottle, decreased the time of complete coagulation from about 10 hours to 6 hours, whilst 0.01% of calcium reduced the time to about 4 hours.

On the other hand, when the calcium salts already present in the latex were converted into insoluble compounds, such as by the addition of neutral sodium oxalate, coagulation under similar conditions was completely inhibited.

Coagulation is also prevented, as has long been known, by making the latex alkaline; sodium citrate, however, is not an anti-coagulant. Formaldehyde, which paralyses the action of most enzymes, acts as an inhibitor also.

A further argument against the bacterial theory is that on most rubber estates there are to be found trees the latex of which coagulates almost immediately it oozes from the tree, thus stopping further flow, whereas that from neighbouring trees equally exposed to bacterial infection remains fluid as usual. In 1912, when visiting an estate on which the latex from certain blocks of trees, exposed to flooding by brackish water, exhibited to a very marked extent this property of rapid coagulation on the tree, the author tried to remedy the trouble by sponging the cuts with solutions of various antiseptics, but without effect.

It can now be suggested that this premature coagulation may be due to the presence in the latex of an abnormally large amount of soluble calcium salts, derived from the salt water. Incidentally it may be pointed out that the circumstance of this almost instantaneous tree coagulation affords evidence that the coagulating enzyme is not itself a product of bacterial action.

As already mentioned, natural coagulation only takes place completely when the latex is contained in closed vessels. The vessel must either be nearly full, or, if not, the air in the space above the latex must be under increased pressure. The explanation why coagulation is complete under these conditions and not in open vessels is most likely that in the one case the carbon dioxide which is liberated cannot escape, and so prevents the development of the putrefactive alkali-forming bacteria.

It was further found that a normal natural coagulation in closed vessels did not occur if the latex was shaken or subjected to vibration, only a small quantity of a non-coherent flocculent body resulting. In open pans the latex may be sufficiently disturbed by the gas bubbles evolved for coagulation to be adversely affected. The activity of the coagulating enzyme was found to be greatly increased by heat, and it seemed, although accurate determinations were not made, that the optimum temperature lay between 55° and 60° C. It may be deduced from this that in the Brazilian method of coagulating latex over a smoky fire, the temperature plays an important part. It would be interesting to know to what degree the coagulating enzyme is activated by the heat and by the acid fumes respectively in this process.

The strength of the evidence brought forward that the coagulation of latex is the work of an enzyme is perhaps best shown by citing for comparison the properties of the enzyme rennet and its action on milk. Rennet produces in milk, as is well known, a colloid precipitation, separating it into casein and whey. The action of rennet does not take place if the milk is even slightly alkaline. Rennet is destroyed by hydrocyanic acid and by formaldehyde, but is not affected by bactericides such as toluene. It is activated, on the other hand, by the addition of acid, also by soluble calcium salts. If, however, the calcium in the milk be converted into an insoluble oxalate

or fluoride, the action of the enzyme is altered and a series of soluble caseoses is formed instead of a coagulum of casein. The rate of action of rennet on milk increases with temperature up to an optimum of 41° C. At temperatures much above 60° C. the enzyme is destroyed. Shaking renders rennet inactive. It is thus seen that in no essential is there a difference between the enzyme coagulation of latex and of milk.

Further comparison may enable us to extend our knowledge of the former process and of its product, rubber. Milk can be coagulated by acid without rennet being present. The coagulum thus formed differs from that produced by rennet; the latter encloses all the calcium phosphates which are contained in the milk, whilst the curd produced by acid only encloses a portion, the remainder being dissolved by the acid. It will be interesting to learn whether the coagulum produced in absence of acid, with and without addition of calcium salts, contains more calcium than rubber coagulated by acid, and if so, what is the effect on the properties of the rubber.

The coagulum produced by rennet at its optimum temperature, 41° C., is very much firmer than that formed either at higher or lower temperatures. The firmness is dependent on and in inverse proportion to the percentage of water in the coagulum. Initially soft, this becomes on standing firmer and poorer in water until the maximum firmness is attained.

Precisely similar behaviour is shown by the rubber coagulum, which from being at first soft and flabby, continually hardens and contracts until, after about four days, the maximum contraction is reached, when the rubber is extremely tough and tenacious. By carrying out the coagulation at higher temperatures, up to 55° C., probably the time required for this maximum contraction would be greatly shortened.

It is interesting to note that the rate of vulcanisation decreases and the mechanical properties of the vulcanised rubber improve according to the length of time, up to four or five days, the coagulum is allowed to remain in its serum before being machined and dried.

### Conclusions.

Further evidence has been brought forward that the natural coagulation of *Hevea* latex is due to the action of an enzyme, as suggested by Whitby.

It has been shown that "acid" coagulation of latex is, in all probability, also brought about by the coagulating enzyme, the acid acting as an activator of the process. Coagulation by smoke and heat according to the Brazilian method must similarly be attributed to enzyme action.

The fact that complete natural coagulation occurs in 10–12 hours in closed vessels, under certain conditions, whilst in the open it may be incomplete at the end of two or three days, is believed to be due to the prevention, by carbon dioxide, of the development of alkali by the action of putrefactive bacteria. Carbon dioxide has been stated by Eaton and Grantham to be produced by the action on sugars of bacteria normally present in latex, and some may be formed this way.

The liberation of carbon dioxide may, however, be detected even when coagulation takes place in the presence of bactericides; but there is no evidence as to whether this signifies that the gas is a product of the chemical changes that take place during coagulation or whether it is present initially and expelled through being less soluble in the separated serum than in the latex.

The author has been unable to follow up the lines of further experiment that suggest themselves owing to his departure from the East early in 1915.

## Annual Meeting.

### THE NATURAL ACCELERATORS OF PARA RUBBER.

BY D. F. TWISS.

(See this J., 1917, 785.)

#### DISCUSSION.

Mr. B. J. EATON (Kuala Lumpur, F.M.S.) writes as follows:—

I note in the paper recently communicated by Dr. Twiss at the Annual Meeting of the Society that, while crediting the writer and his collaborators with the discovery of the maturation process for obtaining a rapidly vulcanising rubber, he states that Dr. Stevens has recently traced the differences in the rate of vulcanisation of certain types of plantation Para rubber (including the "slab" rubber first so-called and discovered by the writer and his collaborators) to the decomposition of the protein matter of the retained serum during the maturing process. The writer is compelled to claim priority in respect of the latter discovery also, which is exclusively the work of the chemical laboratory of the Agricultural Department of the Federated Malay States. We have not only discovered a new type of rubber, which was first named "slab" by us, but in addition, were the first to show the nature of the changes which caused the accelerated rate of cure of such rubber and also of other types of rubber (sheet and crêpe) under certain conditions.

The significance of our second paper in this Journal (July 15, 1916) entitled "Vulcanisation experiments on plantation Para rubber (II.): The cause of variability and some contributory factors," does not appear to have been fully realised or has been overlooked by Dr. Twiss in this respect. In that paper we attributed the effects produced to the decomposition of the protein matter in slab rubber during maturation (and also in sheet, provided means were adopted to retain a certain proportion of moisture during the maturation period), and thus obviously to organic nitrogenous bases or amino acids. We also carried out the decomposition of such protein from latex (after separation from the serum by boiling) and showed definitely that the decomposed protein when added to ordinary slow-curing Para rubber (crêpe) had a very marked effect in accelerating the rate of cure.

Other experiments with decomposed casein gave similar but less marked results. At the time, the difference between the effects produced by the decomposed latex protein and decomposed casein were attributed to the probable difference in the composition of the original proteins and the consequent formation of different quantities or types of organic bases (accelerators). More recent experiments by the writer indicate that it is possible that the type of decomposition may be responsible for the difference in the effects between the two decomposed proteins, but this work is not yet complete.

In the Agricultural Bulletin, Federated Malay States (Vol. V., No. 2, November, 1916) and in various lectures before Planters' Associations in this country, during the last eighteen months, the causes have also been definitely attributed to the formation of such bases. The writer has suggested that possibly putrescine (tetramethylenediamine), which is known to be an accelerator, may be one of the bases formed.

We have, in addition, in this Journal (July 15, 1916), shown the presence of an accelerator preformed in the latex. Whether this accelerator is itself a nitrogenous base and similar to or the same substance as the accelerator formed by the

decomposition of the proteins, or a different substance entirely, remains to be proved. We have shown that the crude extract (evaporated serum, after removal of the protein coagulated by heat) is less effective, in equal quantities, than the decomposed protein material, but this may be due to different quantities of the same accelerator in the crude mixtures.

In the case of "slab" rubber, both accelerators are effective, *i.e.*, the acceleration in rate of cure of "slab" is an additive effect, since coagulum which has matured and partially dried, also retains more of the serum residue after washing and crêping than coagulum which is machined on the day of or the day following coagulation.

In addition, we have shown that the decomposition changes can be followed, under certain conditions, when no other factor is involved, by the loss of nitrogen in the finished dry rubber.

In more recent experiments (this J., 1917, 1217) the writer has shown that the biological changes which take place can be accelerated by drying (incubating) slab rubber at a temperature of about 120° F.

In conclusion, Dr. Stevens' results have merely confirmed and added a little more detail to the results of the previous researches of the writer and his collaborators. The writer and his collaborators have been prevented from following up more rapidly this part of the research, by pressure of other work and further research on numerous important side issues in connection with estate practice in raw rubber preparation.

Dr. TWISS writes as follows in reply:—

In the above remarks Mr. Eaton states that the significance of the well-known investigations (this J., 1915, 989; 1916, 715) which he has made in conjunction with various collaborators, appears to have been overlooked in my recent paper (this J., 1917, 785). My statement in this connection was very brief and therefore of necessity gave an incomplete account of the matter. I agree entirely with Mr. Eaton in his view that his work led to the recognition of the probability that decomposition products of protein matter are responsible for the increased rate of vulcanisation of "matured slab" rubber, but the evidence to this effect was only circumstantial and the actual presence of organic bases of the necessary type was first directly demonstrated by Dr. Stevens in his experiments with plantation rubber (this J., 1917, 368).

## Communication.

### NOTES ON EXHAUST GAS ANALYSIS.

BY G. R. CLARE.

For the control of gas engines, furnaces, etc., using producer gas, it is important to know the percentage of the producer-gas used that is rejected as unburnt. This is usually ascertained by calculation from the percentages of carbon dioxide, carbon monoxide, oxygen, and hydrogen found by the ordinary gas-analytical methods. The amount of methane present in the producer gas being so small (1 to 4%) it is considered unnecessary to estimate it separately in the exhaust.

This method being rather tedious owing to the slow absorption of oxygen by pyrogallol and the time spent on calculation, and in view of the fact that an approximation only is needed, the writer found on investigation that the time spent on both the analysis and the calculation could be shortened to at least one quarter. Only the carbon dioxide and monoxide and hydrogen are



estimated, the estimation of oxygen being eliminated and the calculation reduced to a simple formula (and curve if necessary) which are arrived at as follows:—

The percentage of burnt producer gas present in the exhaust is given by:—

$$\frac{100 \times (\% \text{ of perfect exhaust gas in the exhaust gases})}{\text{Perfect exhaust gas per 100 vols. of producer gas.}} \quad (\text{i.})$$

The perfect exhaust is the volume which would be formed by the complete combustion of 100 volumes of producer gas with just the requisite amount of air, and consists solely of carbon dioxide and nitrogen, the water vapour formed being condensed. The volume of  $\text{CO}_2$  formed is the sum of the % of  $\text{CO}_2$ , CO, and  $\text{CH}_4$  present in the producer gas, while the volume of nitrogen is the sum of the nitrogen present in the producer gas and the nitrogen necessarily introduced in the air with the consumed oxygen. The  $\text{CO}_2$  due to perfect exhaust gas present is obtained by subtracting the  $\text{CO}_2$  due to the unburnt gas from the  $\text{CO}_2$  in the mixed exhaust gases.

Therefore the % perfect exhaust present is:—

$$\frac{100 \times (\% \text{CO}_2 \text{ in exhaust gases} - \text{CO}_2 \text{ due to unburnt gas present})}{\text{CO}_2 \text{ in 100 volumes of perfect exhaust.}} \quad (\text{ii.})$$

The  $\text{CO}_2$  in 100 volumes perfect exhaust we have seen is:—

$$100 \times (\% \text{CO}_2 + \% \text{CO} + \% \text{CH}_4 \text{ in producer gas})$$

Volume of perfect exhaust formed by 100 volumes producer gas.

Substituting we have:—

$$\frac{(\% \text{CO}_2 \text{ in exhaust gases} - \text{CO}_2 \text{ due to unburnt gas}) \times \text{volume of perfect exhaust per 100 vols. producer gas}}{\% \text{CO}_2 + \% \text{CO} + \% \text{CH}_4 \text{ in producer gas.}} \quad (\text{iii.})$$

The burnt gas is therefore given by this formula

$$\frac{100}{\text{Volumes perfect exhaust per 100 vols. producer gas}}$$

which works out to

$$\frac{100 \times (\% \text{CO}_2 \text{ in exhaust} - \% \text{CO}_2 \text{ due to unburnt gas})}{\% \text{CO}_2 + \% \text{CO} + \% \text{CH}_4 \text{ in producer gas}} \quad (\text{iv.})$$

We will now assume that the producer gas has the following mean composition:—16%  $\text{CO}_2$ , 11% CO, 26%  $\text{H}_2$ , 3%  $\text{CH}_4$ , 44%  $\text{N}_2$  and no oxygen. These particular figures are taken as an example, as they work out to a simple formula.

Then  $\frac{16}{40} \times \% \text{combustibles in the exhaust}$  may be substituted for the %  $\text{CO}_2$  due to unburnt gas present in the exhaust, and

(iv.) becomes:—

$$\frac{\% \text{CO}_2 \text{ in exhaust} - 0.4 \times \% \text{combustibles in exhaust}}{0.3} \quad (\text{v.})$$

The percentage of unburnt gas in the exhaust being  $\frac{100}{40} \times \% \text{combustibles present}$ , the percentage of producer gas used which is rejected as unburnt gas is:—

$$\frac{75 \times \% \text{combustibles in exhaust}}{\% \text{CO}_2 \text{ in exhaust} + 0.35 \times \% \text{combustibles in exhaust}} \quad (\text{vi.})$$

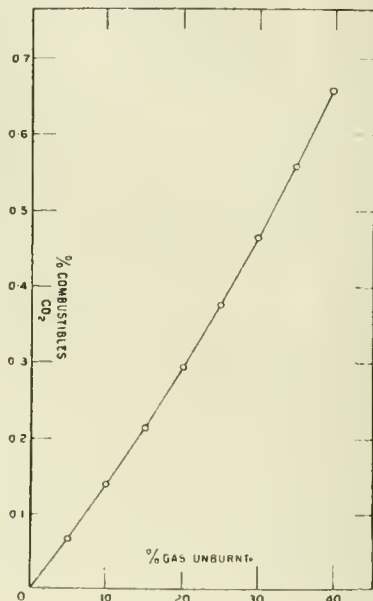
It is obvious now that at any fixed percentage of gas unburnt the value

$$\frac{\% \text{Combustibles in exhaust}}{\% \text{CO}_2 \text{ in exhaust}}$$

must be fixed also.

Substituting values and plotting them a regular curve is obtained.

% Combustibles % CO <sub>2</sub> .....	0.068	0.140	0.215	0.294	0.377	0.465	0.558	0.656
% Gas unburnt	5	10	15	20	25	30	35	40



Given an analysis of exhaust gas in terms of %  $\text{CO}_2$ , CO, and  $\text{H}_2$ , we see at a glance the percentage of the gas used which is rejected as unburnt. It will be found that this formula and curve are sufficiently accurate for practical purposes even when the composition of the producer gas varies fairly considerably.

By this method an analysis may be completed and the required value obtained in five minutes' time, which is a great saving over the old method.

Of course, other values may be substituted for the sample analysis of producer gas taken and a slightly modified formula and curve obtained; as previously mentioned, that particular set of figures was used to give a simple formula.

## Canadian Section.

*Meeting held at Toronto on Friday, December 21st, 1917.*

MR. S. B. CHADSEY IN THE CHAIR.

### HISTORY OF THE NICKEL INDUSTRY SINCE 1910.

BY E. P. MATHEWSON, B.Sc. (NICKEL).

(Abstract.)

The history of the nickel industry in Canada and the United States was the subject of a most interesting address by the late D. H. Browne, before this section of the Society in January, 1911 (see this J., 1911, 248). This paper is intended to fill in the gap between that date and the present in the history of the nickel industry.

In the first place, the use of nickel has increased immensely during the past seven years, and the great war has emphasised its importance to a very large extent. Some of the greatest engineering structures of the world have been recently completed, and but for the use of nickel as an alloy in the steel employed, their construction would have been entirely out of the question. One of the most recent of these structures is the new Quebec Bridge.

The lack of nickel in Germany after the first few months of the war caused our enemies to offer great inducements to producers to supply them with nickel. They were successful in securing a supply from Norway, which, however, by means of certain negotiations on the part of the British Government, was cut off almost entirely, and in May of 1917, the refinery at Kristiansands was damaged by fire to such an extent that no nickel has been produced at that plant since that time.

The affair of the Deutschland is familiar to all and caused a feeling of uneasiness amongst the Allies at the time.

It is no secret to state that the nickel producers, particularly the International Nickel Company, of which the Canadian Copper Company is the chief subsidiary, became much alarmed at what they considered a lack of market for nickel proportionate to their producing capacities.

Just before the war broke out, the manufacturers of structural steel seemed suddenly to become aware of the value of nickel steel and its use was increasing rapidly. When the war broke out, the demand for nickel in the war industries absorbed the entire supply, and it is generally believed that after the war the demand for nickel will continue to a still greater extent.

In connection with the automobile industry, the consumption of nickel is quite large and growing constantly. For making of shafting, particularly marine shafting, nickel seems to be essential, but the great consumption of the metal will be in the form of nickel steel alloy for structural purposes.

Within the past few years, the enormous reserves in the lower levels of the Creighton Mine have been discovered, and at present a very important investigation is being made by the Mond Nickel Company on the lot west of the Creighton Mine, where diamond drilling is being undertaken with the object of striking the Creighton ore body extension at a depth of 3800 feet. This is twice the depth of any of the workings in the nickel region of Canada. Some very interesting diamond drilling has been undertaken in the Eastern part of the Sudbury district by the Long-

year Company, they having drilled through 100 feet of wash and found nickel ores at some distance below the wash.

Among interesting changes in smelting are the following: The Mond Nickel Company has moved to a point eight miles east of Sudbury on the Canadian Pacific Railway and constructed a modern smelter and model town called Coniston.

The old Murray Mine is now the principal holding of the British America Nickel Corporation, Ltd., which is the successor of several of the older companies. The depression in financial circles at the beginning of the war had its effect on the nickel industry and particularly on this company, and work was suspended. But later, as the company owned the Hybinette patents for refining nickel, which are considered very valuable and which had been in operation for a number of years at the Kristiansands refinery, Norway, it was decided to resume the construction of the plant at Nickelton, and this work is now well under way.

Some time ago nickel became a political issue in Canada, but with the entry of the United States into the war on the side of the Allies, this ceased to interest politicians. However, one good result of the nickel agitation was the appointment of the Royal Ontario Nickel Commission, which recently completed its labours and published a voluminous report which is generally considered to be the finest thing of the kind ever offered to the public (see this J., 1917, 499).

Since the report of the Commission has been made public, the rate of taxation on nickel properties has been materially increased, with a corresponding increase in revenue to the Government, which more than justified the expense of the Commission.

The importance of Canada as a nickel producer has been maintained through all these years, and it now is apparent that the country will soon become equally important in the refining of nickel.

The International Nickel Company has under construction, and near completion, a modern refinery at Port Colborne, Ontario, which will have an initial capacity of 7500 tons of nickel per annum, and provision has been made for enlargement to double that capacity.

The British America Nickel Corporation's plant will be nearly the same size as the Port Colborne plant, but will utilise the Hybinette electrolytic process, which is quite distinct from that used by the International Nickel Company, and in it also will be made provision for enlargement as the needs of the industry demand. This plant, however, will not be completed until the year 1919.

## Liverpool Section.

*Meeting held at the University on Friday, December 21st, 1917.*

MR. A. T. SMITH IN THE CHAIR.

### VITAMINES.

BY PROF. W. RAMSDEN.

(Abstract.)

It has been discovered during the last 20 years that animals as various as pigeons, fowls, rats, pigs, and man if fed exclusively on polished rice develop the disease known as beri-beri, characterised by serious changes in the peripheral nerves, and that a water, or 90% alcohol, extract of the



rice-polishings contains a substance of which very minute quantities prevent or cure the disease.

By careful and laborious experiments with rice and other diets it has been shown that this substance is widely spread in the common foodstuffs and that its presence is essential for life. Attempts to isolate it in a pure state in sufficient quantity for chemical investigation have not hitherto been completely successful, but enough has been ascertained to make certain that it is neither a protein, fat, nor carbohydrate, nor indeed any of the known constituents of plants or animals. It is free from phosphorus, insoluble in absolute alcohol, ether, benzene, acetone, or oils, soluble in water or 90% alcohol, and is adsorbed by fullers' earth or animal charcoal. It survives a good deal of cooking at 100° C. but is slowly destroyed at 120°. It is resistant to 5% sulphuric acid. Funk has proposed for it the name "the anti-beri-beri vitamine" and this term "vitamine," although open to many objections, has been widely adopted for substances of this type. The name "sitaoid," meaning a medicine-like substance associated with food, is suggested by the writer as accurately descriptive and not implying a knowledge of their chemical characters which we do not possess.

Most foodstuffs contain adequate amounts of the anti-beri-beri substance but, it is important to note, white flour, polished rice, and arrowroot, and probably also "corn-flour," and much of what is sold as sago, tapioca, and semolina, contain none. Ordinary white bread doubtless contains a little derived from its yeast, but certainly not enough for health unless supplemented by other foodstuffs. Wheat-bran contains a little, wheat-embryo contains a good deal.

It has also been discovered, thanks mainly to the pioneer researches of Hopkins, Stepp, Osborne and Mendel, and McCollum and his collaborators, that rats and mice require not only the anti-beri-beri substance in their diet but at least one other vitamine or sitaoid, and in its absence get sore eyes (Xerophthalmia) and are unable to grow. It will be convenient to refer to this as the anti-sore eyes substance (McCollum's Fat-soluble A).

As found in animal substances it is slightly soluble in water but much more so in oils, ether, and hot alcohol. It is very resistant to ordinary cooking operations, but is slowly destroyed by prolonged exposure to daylight. It has not been identified with any of the chemically known constituents of plants or animals. It is either completely free from phosphorus and nitrogen or, alternatively, it is active in incredibly minute amounts. As found in the higher plants it is insoluble in ether, oil, or hot alcohol. The relations between the plant and the animal anti-sore eyes substances have not yet been ascertained. Proved to be indispensable for mice and rats, and especially for the growth of the young, there are strong reasons also for suspecting it to be equally indispensable for man. It is present in quantity in yolk of egg, cod liver oil, all animal fats investigated (with the remarkable exception of lard), milk, cheese, butter, margarine made from animal fats, green leaves, soya beans, millet, and flax seed. It is absent from lard and all vegetable oils (almond, maize, cottonseed, sunflower seed, linseed, and soya bean oils) and margarines made from them. It is present, although apparently in small amount only, in the (entire) seed of wheat, oats, maize, hemp, rye, and cotton seed.

There are strong reasons for believing that lack of yet a third vitamine or sitaoid is mainly responsible for the disease known as scurvy. This anti-scorbutic substance is remarkable in that although surviving ordinary cooking operations it usually fails to survive slow drying or long "preservation" of the food in which it is contained. It is present in all fresh vegetables, fruit, or meat.

It is rarely or never present in dried vegetables. It is absent or inadequate in amount in dried seeds, but it is present in seeds, e.g., barley, peas, which have just been allowed to germinate (Furst, Weill and Mouriquand, Miss Chick).

McCollum's observation that guinea-pigs on a diet of oats do not get scurvy if given laxatives is explicable on the assumption that the anti-scorbutic substance is essential for the normal functioning of the intestine.

The table given below indicates some of the commoner foods in which one or more of the three vitamins have been definitely proved present or absent. The feeding experiments necessary for each such proof are time-consuming and demand very many precautions. Anything like exact comparisons is at present impossible, and the number of plus signs in the table indicates merely the general impression conveyed by the different observers.

	Anti-Beri-beri.	Anti-Xerophthalmia.	Anti-Scurvy.
Egg-yolk .....	++++	++++	o
*Milk (cow's) ....	+	++	+
Butter .....	little or none	++++	o
Whole meal wheat flour .....	++	little or none	o
White wheat flour .....	o	little or none	o
Potatoes .....	+		++
Cabbage .....	++	+++	+++
Dried peas .....	++	very little	o
Orange juice ....			++
Dried soya bean, millet, and linseed .....	++	++	o
Fresh meat and fish .....	+	+	+
Tinned meat ....	o		o
Cod-liver oil .....	o	+++	o
Lard .....	o	o	o
Margarine:			
from animal fats .....	o	++	o
from vegetable do. ....	o	o	o
Yeast .....	++++	little	o

\* Milk vitamine-content depends largely on the mother's diet.

There is evidence that unless a nursing mother is obtaining adequate amounts of the first two vitamins in her food the milk she produces is deficient in these bodies. Adults tolerate lack of these substances in food much longer than young growing animals.

It is clear that the facts of which a brief account has been given above are of much importance to this community, and should be widely known.

#### DISCUSSION.

Mr. A. T. SMITH said that bread made from white flour was an example of human interference with nature's providence, since the outer portions of the wheat-berry (which, if included, would give a darker flour) contained much more of the vitamins than the central portion of the grain.

Prof. MACDONALD mentioned that the medical profession in Liverpool had done much work in connection with beri-beri and similar diseases, and he thought the subject of Prof. Ramsden's discourse was very important.

Dr. A. HOLT asked what would be the effect of giving an animal a diet abnormally rich in anti-beri-beri substance.

Mr. J. TWOMEY wished to know if there were any theories of the action of vitamins in the body.

Prof. RAMSDEN, in reply, said that presumably large amounts of vitamins had no further effect, i.e., a certain quantity of the substance was required by the animal and above this any excess was metabolised or eliminated. Anti-beri-beri substance would not long survive a temperature of 130° C. To formulate any theory of the action of vitamins would be too speculative until more was found out about their chemical

constitution, etc. The anti-scorbutic vitamin seemed to be preserved in the juice or extract of limes, oranges, and lemons. Green vegetables contained large amounts of all three vitamins. Dr. Mori of Japan had found 400 cases of conjunctivitis in children due to a diet of cereal food lacking in fat-soluble A vitamin. Dry peas and beans contained little or no anti-scorbutic vitamin, but if they were allowed to germinate for 48 hours they became rich in this body.

## Manchester Section.

Meeting held at Grand Hotel on Friday, December 7th, 1917.

MR. WILLIAM THOMSON IN THE CHAIR.

### THE OXIDATION OF RUBBER.

BY S. J. PEACHEY AND M. LEON.

In a paper read before the Manchester Section of this Society in 1912, one of the authors described a simple form of apparatus designed for the purpose of carrying out quantitative investigations on the oxidation of rubber by air (this J., 1912, 31, 1103). As the result of a series of experiments on carefully purified rubber it was shown:—(1) that the reaction between rubber and oxygen, which under ordinary conditions is so slow as to be imperceptible, becomes sufficiently rapid at a temperature of about 85° C. to render possible direct observation of the volume of gas absorbed; (2) that the presence of a normal amount of natural resin (about 3% for the type of rubber employed) exerts a marked retarding effect on the rate of oxidation; (3) that the amount of oxygen ultimately taken up by the unit molecule of rubber ( $C_{10}H_{16}$ ) corresponds closely to four atomic proportions.

The work has now been extended in several directions with the results recorded below.

#### *Action of dry oxygen on purified plantation rubber.*

In the experiments referred to above a weighed quantity of purified raw rubber (i.e., rubber freed from resins and proteins) was deposited in the form of a thin film on the interior surface of a flask connected with a measuring burette containing mercury, the whole apparatus (*loc. cit.* Fig. 1) being filled with a known volume of moist oxygen. The flask was heated in a water bath at a constant temperature of 85° C. and the volume of oxygen absorbed was periodically measured as long as absorption took place. The rate of oxidation of the rubber was thus observed and the total amount of oxygen taken up was readily determined. Under such conditions it was found, as previously stated, that for every unit molecule ( $C_{10}H_{16}$ ) of rubber oxidised, the amount of oxygen absorbed approximated very closely to four atomic proportions. In these experiments the assumption was made that the residual gas consisted wholly of oxygen and that no volatile products, or at least none possessing an appreciable vapour pressure, were produced during the oxidation. That this assumption is not quite justified is shown later.

In order to ascertain whether either the speed

of the reaction or the amount of oxygen ultimately taken up was influenced by the presence of moisture, it was considered desirable to carry out parallel experiments using carefully dried oxygen. For this purpose the apparatus previously described was employed, but the oxygen, instead of being saturated with water vapour as in the earlier experiments, was carefully dried by passing through tubes containing solid potassium hydroxide and phosphorus pentoxide respectively. The film of rubber also was dried *in vacuo* over phosphorus pentoxide. It was found that in these circumstances the rubber underwent oxidation at practically the same rate as when moist oxygen was employed and that the amount of gas taken up was very nearly the same in the two cases. This is illustrated by curve B (Fig. 1), which represents the oxidation of purified rubber by dry oxygen; curve A represents the result of a parallel experiment with moist oxygen and is included for comparison. In a typical experiment 0.2413 gm. of rubber absorbed 78.9 c.c. of oxygen (dry) measured at 0° C. and 760 mm. pressure, a volume which corresponds to an absorption of 3.97 atoms of oxygen per unit molecule ( $C_{10}H_{16}$ ) of rubber. It appears therefore that the presence or absence of moisture exerts no appreciable influence on the course of the reaction.

#### *Formation of carbon dioxide during the oxidation of rubber.*

In the earlier experiments on the behaviour of rubber towards moist oxygen at a temperature of 85° C., it was assumed that the gas remaining in the absorption apparatus after the reaction consisted of oxygen only and that the oxidation of the rubber was not accompanied by the formation of an appreciable quantity of any product possessing a measurable vapour pressure at the ordinary temperature (except water, which was present throughout). A more careful examination of the residual gas has now revealed the presence of an appreciable and somewhat variable amount of carbon dioxide, which appears to be a normal product of the oxidation. Portions of the residual gas were drawn off with the aid of a Sprengel pump and analysed in a Haldane apparatus. The results obtained in a number of experiments are shown in columns 3 and 7 of the accompanying table. It will be seen that during the oxidation of a molecule ( $C_{10}H_{16}$ ) of rubber approximately one quarter of an atomic proportion of carbon is converted into carbon dioxide. At first sight this might suggest that the rubber complex must contain at least 40 atoms of carbon, but it is more probable that the formation of carbon dioxide is the result of a secondary reaction.

The conclusion previously arrived at, viz., that approximately four atoms of oxygen are absorbed during the oxidation of the  $C_{10}H_{16}$  unit of the rubber molecule, is confirmed by the later experiments and is not in any way affected by the observation that a certain amount of carbon dioxide is produced during the reaction and is included in the measurement of the residual oxygen. The carbon dioxide is formed, however, from an equal volume of oxygen and it follows that the amount of oxygen interacting with the rubber is somewhat greater than that which is absorbed and which is contained in the resinous oxidation product. The figures given in columns 5 and 6 represent respectively the number of atoms of oxygen taken up by the unit molecule of rubber to form resinous oxidation products, and the total number of atoms of oxygen interacting with the rubber. The latter are not very concordant and do not approach very closely to whole numbers, and it is not easy in the present state of our knowledge to interpret their significance.



TABLE I.

	Rubber.	Oxygen absorbed.	Carbon dioxide produced.	Oxygen interacting.	No. atoms oxygen taken up by $C_{10}H_{16}$ .	No. atoms oxygen interacting with $C_{10}H_{16}$ .	Atomic proportion of carbon oxidised to $CO_2$ per $C_{10}H_{16}$ .
	gm.	gm.	gm.	gm.			
I. ..	0.2590	0.1271	0.0199	0.1416	4.16	4.64	0.23
II. ..	0.2265	0.1099	0.0191	0.1238	4.11	4.63	0.26
III.	0.2392	0.1265	0.0212	0.1358	4.27	4.82	0.27
IV.*	0.2413	0.1128	0.0151	0.1238	3.97	4.36	0.18

\* Dry oxygen was used in this experiment.

#### Action of moist oxygen on vulcanised rubber.

The results of the experiments described in this and in the earlier communication are of considerable interest from the purely chemical standpoint, but in that raw rubber finds very few applications in the industries it is naturally of greater importance from the point of view of the technologist that some knowledge should be gained regarding the behaviour of vulcanised rubber on prolonged exposure to air or oxygen. The results of some preliminary experiments in this direction are here recorded.

The apparatus employed was that previously described. In order to obtain a thin film of vulcanised rubber suitable quantities of a solution of raw rubber in benzene and a standard solution of sulphur in the same solvent were run into the flask, which was then rotated slowly while the solvent was evaporated in a current of air. The film was subsequently dried *in vacuo* until constant in weight. The flask was then heated in an oil bath, provided with an agitator, to a temperature of  $140^{\circ}C$ . for  $2\frac{1}{2}$  hours. The rubber employed was pale plantation crêpe and experiments were made with both the acetone-extracted rubber and with that containing a normal percentage (3.08) of natural resin. 0.2528 gm. of acetone-extracted rubber was vulcanised as described above with the addition of 0.0256 gm. of sulphur, corresponding to 9.2% on the weight of the mixture. The volume of oxygen absorbed amounted to 92.3 c.c. measured at  $0^{\circ}C$ . and 760 mm. pressure, corresponding to 4.43 atoms of oxygen per unit molecule of rubber.

0.2497 gm. of raw rubber containing 3.08% of natural resin was vulcanised with the addition of 0.0217 gm. of sulphur, corresponding to very nearly 8%. The volume of oxygen absorbed amounted to 77.9 c.c. measured at  $0^{\circ}C$ . and 760 mm. pressure, equivalent to 3.79 atoms per unit molecule of rubber. The residual gas was found to contain 0.0137 gm. of carbon dioxide—an amount which would be produced by the oxidation of 0.16 atom of carbon per molecule of rubber. No sulphur dioxide was formed.

The relative rates of oxidation of the resin-free and resin-containing samples of vulcanised rubber are illustrated in Fig. 1, the curves (C and D) being obtained by plotting the volume of oxygen absorbed against the time of heating. It will be seen that, as in the case of unvulcanised rubber, the presence of the natural resin greatly retards the rate of oxidation. Whilst the oxidation of the resin-free sample was complete in 314 hours, that of the resinous rubber required a period of nearly 800 hours.

Further, it will be seen that vulcanised rubber, whether resinous or resin-free, oxidises far less rapidly than the corresponding raw rubber, although the amounts of oxygen ultimately taken up are of the same order in the two cases. This latter observation, taken in conjunction with the fact that sulphur dioxide does not appear amongst the products of the reaction, suggests that during

the oxidation of vulcanised rubbers neither the free nor the combined sulphur suffers attack.

The product of the oxidation of vulcanised rubber remains adhering to the flask in the form of a highly lustrous, dark brown and brittle film which could not be brought into solution by treatment with any organic solvent and which resisted the action of alkalis and moderately strong mineral acids. An examination of this product and of that formed by the oxidation of raw rubber is in progress.

#### Oxidation of balata.

In order to ascertain whether the mode of oxidation of balata differed in any essential point

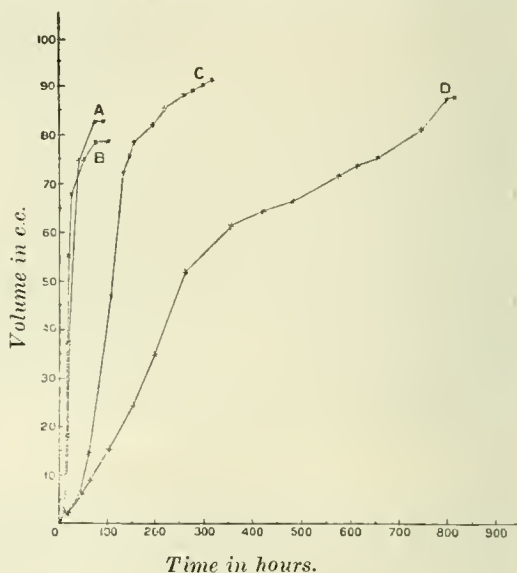


FIG. 1.

from that of rubber, a quantity of the highly purified material was prepared and its oxidation studied by a similar method to that described above.

Good block balata was shredded and after thorough extraction with acetone was dissolved in warm light petroleum, the solution being then filtered and precipitated with alcohol. The precipitated balata was well washed with fresh alcohol, again dissolved in light petroleum, and precipitated with acetone; after washing with acetone it was once again dissolved in light petroleum and the solution cooled with ice. The balata separated as a pure white coagulum which was washed with a little ice-cold petroleum and dried *in vacuo*. The dried material was dissolved in pure benzene and the resulting solution was employed for producing thin films on the interior

surface of the oxidation flask. The oxidation was carried out with moist oxygen at a uniform temperature of 85° C.

The figures obtained are shown in Table II. They are quite concordant in the two experiments and appear to indicate that the oxidation of balata differs from that of rubber in that five atoms of oxygen are absorbed during the oxidation of a molecule of the hydrocarbon; in other words the resinous products of the oxidation of balata collectively contain an atom of oxygen more than those formed from rubber. Further, the amount of carbon dioxide produced corresponds with the oxidation of one-third of an atomic proportion of carbon per unit molecule of balata entering into reaction, as against one-fourth of an atomic proportion in the case of rubber. It is evident, therefore, that the mechanism of the oxidation differs in the two cases and it is to be presumed consequently that the two hydrocarbons differ essentially in constitution. It is anticipated that an investigation into the nature of the oxidation products may throw further light on this subject; such an enquiry is, however, beset with difficulties, mainly on account of the colloidal nature of the substances formed, which renders their separation and characterisation extremely tedious.

The rate of oxidation of the purified balata is comparable with that of raw rubber freed from resin, and by plotting the volume of oxygen absorbed against the time of heating, a curve (Fig. 2) is obtained, the first half of which, representing the early stages of the oxidation, is very similar to that shown in Fig. 1 (A). In each

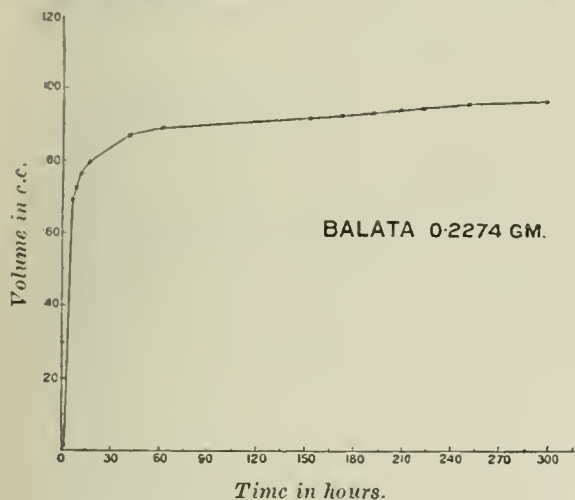


FIG. 2.

case the oxidation commences after a few hours' heating and proceeds very rapidly during a period of from twenty to thirty hours, but beyond this point the curves diverge widely. Whilst the oxidation of the rubber is completed rather abruptly in a period of well under 100 hours, that of the balata suffers a gradual deceleration and requires for completion a period three times as long.

### Products of the oxidation of rubber.

The investigation of the products of the oxidation of rubber presents numerous difficulties. Rubber is essentially a colloidal substance and up to the present every compound directly derived from it has proved to possess a similar physical character. The isolation and characterisation of such compounds constitutes an exceedingly laborious task in that the ordinary processes of separation and the usual criteria of purity are in almost every case inapplicable. Fractional precipitation from different solvents is the only available method of purification, and ultimate analysis at each stage of this process is the only means for ascertaining whether a product presents the character of a pure substance. Having arrived at a constant composition it is still impossible to state with certainty that the product in question is an individual substance.

In spite of these difficulties, however, a certain amount of progress has been made, and in view of the fact that the work has been unavoidably interrupted for the present, the authors feel that it is desirable to record the results so far obtained, although any conclusions drawn from them may need revision at a later date.

In the earlier paper on the oxidation of rubber (*loc. cit.*) it was shown that when a thin film of rubber averaging from 0.2 to 0.4 gm. in weight is exposed to the action of oxygen at a temperature of 85° C. until absorption of the gas ceases, the product of the reaction dissolves almost completely in alcohol, and on evaporation of the solvent is obtained in the form of a yellowish-red and highly viscid gum. The quantities obtained in this manner are too small to permit of a thorough investigation.

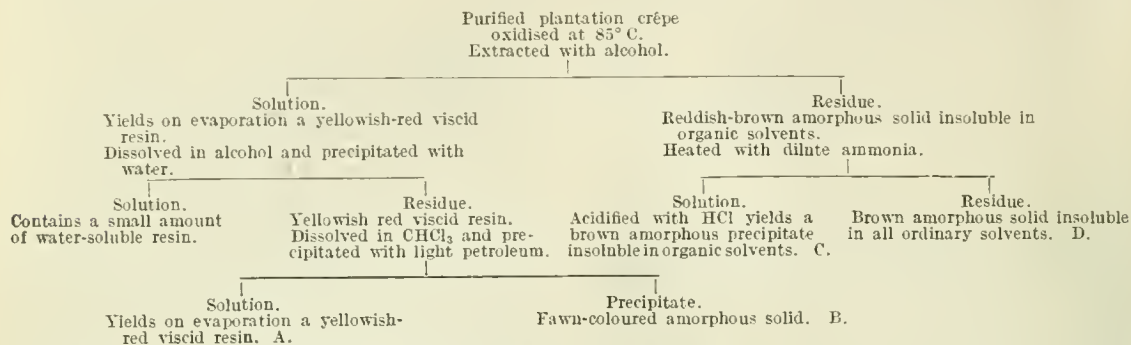
In order to obtain sufficient material for examination, about 50 grms. of extracted plantation rubber was oxidised continuously over a period of six months by exposing in shallow dishes to an atmosphere of oxygen in an electrically heated oven maintained at a temperature of 85° C. The oxidising rubber was periodically extracted with alcohol at intervals of 48 hours, the soluble products being thus removed and fresh surfaces of rubber exposed. The result of this treatment was found to differ unexpectedly from that of the small scale experiments in that exhaustive oxidation of the masses of rubber yielded, in addition to the alcohol-soluble gum, an insoluble and highly stable residual product incapable of undergoing further change under the conditions of the experiment. In its resistance to chemical attack this substance appears to be comparable with cellulose. The alcoholic extract was evaporated to a convenient bulk and mixed with a large excess of water, whereby the bulk of the dissolved gummy products was precipitated, the aqueous liquor yielding on evaporation a small quantity of a product soluble in water. The precipitated material was dried at 80° C., dissolved in chloroform, and by the addition of petroleum ether was separated in the form of a sticky mass, which, by repeated solution and fractional precipitation, was ultimately obtained in the solid form. The chloroform-petroleum liquors yielded on evaporation a yellowish-red viscid resin.

TABLE II.

	Balata.	Oxygen absorbed.	Carbon dioxide produced.	Oxygen interacting.	No. atoms oxygen taken up by $C_{10}H_{16}$ .	No. atoms oxygen interacting with $C_{10}H_{16}$ .	Atomic proportion of carbon oxidised to $CO_2$ per $C_{10}H_{16}$ .
I. ..	gm. 0.2274	gm. 0.1372	gm. 0.0259	gm. 0.1558	5.13	5.82	0.35
II. ..	0.3034	0.1859	0.0360	0.2118	5.21	5.93	0.36



A tedious recital of the numerous devices of fractionation employed is unnecessary here, and it will suffice to indicate diagrammatically the scheme employed in separating what appear to be individual substances from a somewhat complex mixture of oxidation products.



It will be seen that four main products were obtained. The alcohol-soluble portion of the oxidised rubber yielded the two substances, A and B, the one being soluble, the other insoluble in light petroleum. The alcohol-insoluble residue was resolved into the two substances, C and D, the one soluble in aqueous alkalis, the other insoluble. None of the four substances contained nitrogen, although this was found to be present in the small amount of water-soluble product previously mentioned, a fact which suggests that the latter is derived from the protein of the rubber. Substances A and B were purified by solution and precipitation processes until they attained a constant composition; the final analytical results were as follows:—

#### Substance A.

- (1) 0.2078 grm. gave 0.5491 grm.  $\text{CO}_2$  and 0.1872 grm.  $\text{H}_2\text{O}$ .
- (2) 0.2259 grm. gave 0.5973 grm.  $\text{CO}_2$  and 0.2039 grm.  $\text{H}_2\text{O}$ .

whence  $\text{C} = 72.07, 72.11$ ;  $\text{H} = 10.01, 10.03\%$ . These figures agree closely with the formula  $\text{C}_{16}\text{H}_{26}\text{O}_3$ , which requires  $\text{C} = 72.18$  and  $\text{H} = 9.81\%$ .

#### Substance B.

- (1) 0.0902 grm. gave 0.2102 grm.  $\text{CO}_2$  and 0.0645 grm.  $\text{H}_2\text{O}$ .
- (2) 0.0796 grm. gave 0.1859 grm.  $\text{CO}_2$  and 0.0560 grm.  $\text{H}_2\text{O}$ .

A second specimen of B prepared by oxidising a film of rubber in a flask gave similar figures:—

- (3) 0.2043 grm. gave 0.4723 grm.  $\text{CO}_2$  and 0.1471 grm.  $\text{H}_2\text{O}$ .
- (4) 0.3050 grm. gave 0.7096 grm.  $\text{CO}_2$  and 0.2212 grm.  $\text{H}_2\text{O}$ .

whence  $\text{C} = 63.41, 63.69, 63.05, 63.45$ ;  $\text{H} = 7.91, 7.82, 8.00, 8.06\%$ . These figures agree fairly closely with the formula  $\text{C}_6\text{H}_5\text{O}_2$ , which requires  $\text{C} = 63.71$  and  $\text{H} = 7.97\%$ .

#### Substances C and D.

The insoluble mass left after exhausting the oxidised rubber with alcohol was finely ground and extracted in a Soxhlet apparatus with acetone, benzene, and chloroform successively. It was found to be partly dissolved by prolonged boiling with dilute ammonia solution, and this treatment was carried out exhaustively, the ammonia extracts being subsequently precipitated by the addition of hydrochloric acid.

The original insoluble residue, which constituted about 50% of the total oxidation products, was thus separated into approximately 20% of a brown

amorphous and brittle solid, soluble in alkalis (substance C) and 80% of a reddish coloured amorphous substance, insoluble in alkalis and in all the common organic solvents (substance D). After repeated extraction with boiling water these products were dried and analysed.

#### Substance C.

- (1) 0.1432 grm. gave 0.3293 grm.  $\text{CO}_2$  and 0.0982 grm.  $\text{H}_2\text{O}$ .
  - (2) 0.1402 grm. gave 0.3230 grm.  $\text{CO}_2$  and 0.0938 grm.  $\text{H}_2\text{O}$ .
- whence  $\text{C} = 62.71, 62.83$ ;  $\text{H} = 7.62, 7.43\%$ .  $\text{C}_{11}\text{H}_{16}\text{O}_4$  requires  $\text{C} = 62.46$ ;  $\text{H} = 7.55\%$ .

#### Substance D.

- (1) 0.1199 grm. gave 0.2778 grm.  $\text{CO}_2$  and 0.0895 grm.  $\text{H}_2\text{O}$ .
  - (2) 0.0954 grm. gave 0.2227 grm.  $\text{CO}_2$  and 0.0688 grm.  $\text{H}_2\text{O}$ .
- whence  $\text{C} = 63.19, 63.67$ ;  $\text{H} = 8.29, 8.01\%$ .  $\text{C}_6\text{H}_5\text{O}_2$  requires  $\text{C} = 63.71$  and  $\text{H} = 7.97\%$ .

The following is a brief summary of the properties of these products in so far as these have been determined.

Substance A, possessing a composition agreeing with the formula  $\text{C}_{16}\text{H}_{26}\text{O}_3$ , is a clear, brownish-yellow resin of neutral character. It is insoluble in water but possesses a remarkably wide range of solubility in organic solvents; it is freely soluble for example in alcohol, acetone, ether, ethyl acetate, amyl alcohol, amyl acetate, acetic acid, formic acid, glycerol, chloroform, carbon tetrachloride, carbon bisulphide, light or heavy petroleum, phenol, benzene, naphthalene, aniline, and pyridine.

Attempts to obtain derivatives of the substance by treatment with the usual reagents have mostly failed so far; nitric acid, however, attacks and dissolves the substance, and the addition of water precipitates a yellow compound which has not yet been obtained in a pure condition.

Substance B, which in composition agrees with the formula  $\text{C}_6\text{H}_5\text{O}_2$ , is a fawn-coloured amorphous powder of feebly acidic character. It possesses no very definite melting point but softens and begins to intumesce at  $132^\circ\text{C}$ , gradually swelling as the temperature is raised until it occupies three times its original volume; it does not char below  $250^\circ\text{C}$ . On heating 0.3 grm. of the substance in a small tube connected with a bulb containing barium hydroxide, no evolution of carbon dioxide could be detected.

The substance is insoluble in water, ether, light petroleum, and carbon bisulphide; it dissolves freely in alcohol, acetone, ethyl acetate, amyl alcohol, amyl acetate, chloroform, phenol, naphthalene, and pyridine, and with difficulty in benzene. Dilute acids are without action, but concentrated hydrochloric acid dissolves the substance yielding a dark brown solution; on warming a vigorous reaction sets in, accompanied by considerable charring. Concentrated nitric acid dissolves the

substance in the cold, yielding a reddish-brown solution which evolves nitrous fumes copiously on warming and on dilution with water throws down a flocculent yellow precipitate. After drying this substance darkens somewhat at  $185^{\circ}\text{C}$ , but does not melt or char below  $250^{\circ}\text{C}$ . Substance B dissolves readily in dilute caustic soda and the solution when shaken with benzoyl chloride yields a brown mass which after washing and drying forms a granular powder bearing a close resemblance to D. It is insoluble in all the common organic solvents.

Attempts to determine the molecular weight of B by the ebullioscopic method resulted in failure, no elevation in boiling point being observable in a 1–2% solution in chloroform or benzene. Titration of the substance with  $N/20$  solution of caustic soda yielded figures of doubtful value owing to the indefiniteness of the end point.

The neutralised solution yielded precipitates with most metallic salts, but on examination these proved to consist of the original colloidal material.

Oxidation by potassium permanganate in dilute alkaline solution appears to yield oxalic acid as the main product, identified by the analysis of its barium salt. The addition of powdered permanganate to an acetone solution of the substance yields a brown gummy product readily soluble in water, alcohol, acetone, and chloroform, but insoluble in light petroleum. This substance was purified by repeatedly dissolving in chloroform and precipitating with petroleum spirit, and was finally obtained in the form of a light yellow powder which remains unchanged on heating to a temperature of  $250^{\circ}\text{C}$ .

Substance C agrees in composition with the formula  $\text{C}_{11}\text{H}_{16}\text{O}_4$ . This product, which appears to possess an acidic character, has been superficially examined only.

Substance D appears to have the same composition as B, but is widely different in properties. It forms a gritty powder of a reddish-brown colour and a faint odour recalling that of naturally occurring rubber resin. Heated above  $250^{\circ}\text{C}$ , it chars without melting, emitting an odour resembling that of a burning carbohydrate.

Its resistance to the action of solvents is quite remarkable; it is insoluble in water, aqueous alkalis, alcohol, acetone, ether, ethyl acetate, amyl alcohol, amyl acetate, acetic acid, chloroform, carbon tetrachloride, carbon bisulphide, glycerin, petroleum spirit, benzene, phenol, aniline, pyridine, and piperidine. Formic acid appears, however, to attack the substance on prolonged boiling, partly dissolving it to form a brownish-coloured solution from which a yellow granular substance is precipitated on the addition of water; this, on drying, forms a fine yellow powder readily soluble in chloroform but insoluble in light petroleum. In appearance and in solubility it closely resembles B. A quantity of the material was purified by precipitation from chloroform solution, and on analysis yielded the following figures:—

0.1078 grm. gave 0.2533 grm.  $\text{CO}_2$  and 0.0708 grm.  $\text{H}_2\text{O}$ .

0.0872 grm. gave 0.2045 grm.  $\text{CO}_2$  and 0.0559 grm.  $\text{H}_2\text{O}$ , whence  $\text{C}=63.90, 63.96\%$ ;  $\text{H}=7.29, 7.12\%$ .  $\text{C}_9\text{H}_{16}\text{O}_2$  requires  $\text{C}=63.71$  and  $\text{H}=7.97\%$ .

The residue from the formic acid treatment resembled the original substance D, being insoluble in all the ordinary organic solvents; in colour, however, it was almost black. Its composition was not definitely established.

Substance D is not attacked by dilute mineral acids, but concentrated hydrochloric and sulphuric acids produce a considerable amount of charring.

Concentrated nitric acid attacks the substance vigorously, forming a brownish-red solution which on dilution yields a flocculent yellow precipitate. The filtrate, after evaporation with alcohol to expel

nitric acid, leaves a light brown gum which deposits small crystals of oxalic acid on standing. The gum was not obtained in a sufficiently pure condition for analysis.

The yellow compound precipitated by water from the nitric acid solution of D was washed, dried, and purified by dissolving in acetone and precipitating with benzene. It forms an amorphous, yellow powder readily soluble in alcohol and acetone, but insoluble in ether, benzene, light petroleum, and chloroform. Analysis yielded the following results:—

(1) 0.1191 grm. gave 0.2248 grm.  $\text{CO}_2$  and 0.0568 grm.  $\text{H}_2\text{O}$ .

(2) 0.2172 grm. gave 0.4117 grm.  $\text{CO}_2$  and 0.1002 grm.  $\text{H}_2\text{O}$ .

(3) 0.1746 grm. gave 7.8 c.c.  $\text{N}_2$  at  $19.5^{\circ}\text{C}$ . and 737.8 mm.

(4) 0.1554 grm. gave 7.4 c.c.  $\text{N}_2$  at  $17.8^{\circ}\text{C}$ . and 741.2 mm.

whence  $\text{C}=51.48, 52.07$ ;  $\text{H}=5.30, 5.13\%$ ;  $\text{N}=5.01, 5.44\%$ .

$\text{C}_{11}\text{H}_{13}\text{NO}_6$  requires  $\text{C}=51.8$ ;  $\text{H}=5.1$ ;  $\text{N}=5.4\%$ .

Attempts to determine the molecular weight of the substance resulted in failure.

Summarising the results of this investigation, as far as the oxidation products of rubber are concerned, it may be stated:—

(1) That when purified rubber in considerable mass is exposed to the action of oxygen at a temperature of  $85^{\circ}\text{C}$ , for a period of six months or thereabouts, with frequent extraction of the soluble surface oxidation products, at least four substances are produced: (A) a light reddish-yellow transparent viscid resin of neutral character, insoluble in water but soluble in most organic solvents and possessing a composition agreeing with the formula  $\text{C}_{16}\text{H}_{26}\text{O}_3$ ; (B) a fawn-coloured amorphous solid of feebly acidic character, insoluble in water but soluble in most organic solvents and possessing a composition agreeing with the formula  $\text{C}_6\text{H}_9\text{O}_2$ ; (C) a brown, amorphous substance insoluble in water and in practically all organic solvents, but soluble in dilute alkalis; its composition agrees with the formula  $\text{C}_{11}\text{H}_{16}\text{O}_4$ ; (D) a reddish-brown amorphous substance insoluble in water, in alkalis, and in all organic solvents with the exception of formic acid, which attacks it chemically, converting it into new substances; its composition agrees with the formula  $\text{C}_6\text{H}_9\text{O}_2$ ; concentrated nitric acid attacks the substance yielding oxalic acid, a water-soluble gum, and a yellow insoluble powder of the formula  $\text{C}_{11}\text{H}_{13}\text{NO}_6$ .

The literature on the subject is not extensive. Spiller, in 1865 (*J. Chem. Soc.*, 18, 41), published an analysis of a resin-like substance extracted from perished rubber, and showed that in composition it approximated to the formula  $\text{C}_{30}\text{H}_{35}\text{O}_{10}$ , while Herbst has stated (*Ber.*, 1906, 39, 523) that by passing air for several days through a boiling solution of Para rubber in benzene, two products are obtained: (A) a yellow, amorphous solid, produced in small quantity only, soluble in benzene but insoluble in petroleum, and agreeing in composition with the formula  $\text{C}_{10}\text{H}_{16}\text{O}_3$ ; (B) a brownish-red syrup readily soluble in petroleum and agreeing in composition with the formula  $\text{C}_{10}\text{H}_{16}\text{O}$ .

The results recorded in the present paper tend to show that the oxidation of rubber is in reality a process of considerably greater complexity than has been suspected hitherto. While Herbst's observations indicate the formation of what appear to be two simple addition products, it would now appear that at least four distinct compounds are produced, none of which exhibits any simple relationship to the original molecule of rubber, whether this latter be formulated in accordance with the views held by Harries (*Ber.*, 1905, 38, 1195) or with those put forward more recently by Pickles (*J. Chem. Soc.*, 1910, 97, 1085).



It should be noted that Herbst's description of the two compounds which he represents by the formulæ  $C_{10}H_{16}O$  and  $C_{10}H_{16}O_3$  respectively, accords roughly with that of the two compounds A and B to which the authors assign the respective formulæ  $C_{16}H_{26}O_3$  and  $C_6H_9O_2$ . The difference in composition is, however, considerable. In the compound which he represents as  $C_{10}H_{16}O$ , Herbst found an average amount of 80.14% carbon (a figure which is not by any means in good agreement with the suggested formula) as compared with 72.09% found by us in the compound which we represent as  $C_{16}H_{26}O_3$ . In the more highly oxidised compound, Herbst found 65.54% carbon as against the mean figure 63.55% found by us in the compound which we formulate as  $C_6H_9O_2$ . The lower percentages of carbon found by us suggest a more exhaustive oxidation.

#### DISCUSSION.

Mr. H. L. TERRY noticed that the behaviour of vulcanised rubber on oxidation was quite comparable to that of raw rubber, both extracted and unextracted, and that no volatile sulphur compounds were detected. It was generally accepted in commercial circles that vulcanised rubber decayed more readily than unvulcanised rubber. He had in his possession a skein of elastic thread of raw Brazilian Para rubber 32 years old which was absolutely sound. The problem was to produce a vulcanised sample capable of withstanding atmospheric conditions to a greater degree than was possible at the present time. He had also a sample of vulcanised rubber, in appearance resembling shellac, which could be ground up in a mortar, and forming the hard yellow resin mentioned in the paper. There was at present no really practical knowledge of the composition of this substance and no solvent had as yet been discovered. Both Burghardt and Spiller had made experiments with it and failed to achieve any satisfactory results. Spiller's resin contained 27% of oxygen—a higher figure than shown in any of the authors' analyses. The conclusion to be drawn from the paper seemed to be that the panacea had not yet been discovered for preventing oxidation in vulcanised rubber goods as ordinarily manufactured.

Mr. PAYMAN asked whether the authors had detected any hydrogen peroxide in the gaseous product of the oxidation process, thus indicating that auto-oxidation took place in the presence of the normal oxidation.

Mr. ROWELL hoped that the authors would continue their researches with regard to balata containing its natural resins, as it was of great importance to manufacturers to know the rate of decay of balata when put to work on an ordinary textile belt. Balata contained a certain proportion of saponifiable resin, but when put to work a larger proportion of resins which saponified could be obtained owing to the process of oxidation.

Mr. MORGAN asked if the author had conducted experiments with ozonised air as well as oxygen.

The CHAIRMAN said that he had observed that if very thin sections of indiarubber threads, for instance, when perished, were treated with colouring matter such as an alcoholic solution of methylene blue or magenta, the oxidised portions took up the colouring matter and the good rubber did not. Minute bubbles of air were observed inside the threads and around each of these was a distinct colour. Some of the thread sections showed stratified layers of colour when thus treated. Again, it was curious that if ozonised air were passed over an unstretched rubber thread it appeared to have little or no action upon it, whereas a stretched thread perished almost immediately.

Mr. PEACHEY, in reply, said that the proportion of oxygen found by analysis in substances B and D

was 29%, a higher figure than any previously quoted. To take one analysis, the actual percentages of C, H, and O were 63.19, 8.29, and 28.52 respectively. Hydrogen peroxide appeared to be formed in small quantity during the oxidation and was detected by rinsing out the flask with water after the completion of the oxidation and applying to the aqueous extract the titanous sulphate and the potassium iodide tests. There appeared, however, to be a trace of levulinic acid amongst the products and possibly a little levulinic aldehyde peroxide might be formed, and thus account for the peroxide reaction. It was undoubtedly the case that peroxides were present in an aqueous extract of the material. All the main products of the oxidation were insoluble in water with the exception of the small amount of substance indicated on the left-hand side of the table. It might be possible in the future to conduct further experiments with regard to balata, but it must be remembered that research work on the pure balata hydrocarbon was hardly comparable in its results with that on the technical material. All the experiments had been made in approximate darkness, and the effect of light had not been investigated. It was not possible to draw any conclusions from the experiments with regard to atmospheric oxidation of rubber at ordinary temperatures. A higher temperature had been employed in order that oxidation might proceed at an observable rate. There was apparently no advantage in storing rubber in darkness under water. No experiments had been made with ozonised air, though the effect of ozone on a solution of rubber in chloroform had been studied by Harries with results which were familiar to all rubber chemists. When rubber was stretched it broke after a few minutes' exposure to the action of ozone, whereas if unstretched the action was not so quickly manifested.

#### Newcastle Section.

*Meeting held at Bolbec Hall on Wednesday, January 16th, 1918.*

MR. HENRY PEILE IN THE CHAIR.

#### COKE AS A FUEL FOR THE BLAST FURNACE.

BY G. W. HEWSON.

The substitution of coke for charcoal or coal as a blast-furnace fuel introduced an era of increasing productiveness from the blast-furnace, resulting from greater height of furnace which could be used, and increased pressure of blast permissible. The increase in the height of the furnace was largely due to the ability of the coke to withstand the crushing action of the descending materials much better than coal or charcoal, and thus reach the tuyères before which there is the greatest combustion.

The chief object of the use of fuel in a blast furnace is the production of intense local heat in the vicinity of the hearth; by its combustion it also acts as a reducing agent.

Quoting from Forsythe, the characteristics which make a fuel desirable for use in a blast-furnace are:—

- (1) A well-developed cell-structure. A porous fuel will present more surface to the action of the blast than a dense one, and therefore facilitates and hastens combustion.
- (2) Firmness—a fuel which changes its shape in the furnace, either through being

crushed by the weight of the accompanying materials, or through softening under the action of heat, is undesirable, as the filling of the interstices of the charge with fine or pasty material impedes the current of gases and hampers combustion.

(3) Purity—other conditions being equal, it is evident that the higher the fixed carbon the more efficient the fuel. The non-carbonaceous material develops no heat, but forms a slag which absorbs heat in melting.

Whilst charcoal is more desirable than any other fuel for its readiness to combine with the oxygen of the blast, and also for its purity, the impossibility of the supply meeting the demand rules it out of consideration—except in a limited way—as a blast-furnace fuel.

In a general way coke comes next in efficiency and value to charcoal, and is more desirable than coal, hence its almost universal adoption. The demand for coke became so great that thousands of beehive ovens must have been built with the sole object of supplying coke for blast-furnaces, and so good was this coke that at a later stage of coke-making many managers' preferred beehive coke to by-product coke. Beehive ovens, however, had their limitations, and it was these which led to a change of practice in manufacture. Coking small coal in such ovens was only possible as long as the coal possessed sufficient agglutinative power to produce coke at temperatures within the limits of what beehive ovens were capable of withstanding.

In about 1855 the demand for coke commenced to increase and beehive ovens began to be superceded on account of the increasing scarcity and price of coal suitable for coking in this type of oven. The first uses to which retort ovens were put were to produce coke from coal low in volatile matter and agglutinative power, to obtain a greater yield of coke, and to increase the production of coke by shortening the coking period. Successive improvements have been effected, such as improvements in the arrangements for controlling the admission and circulation of gas and air in the flues in such a way as to produce higher temperatures and to distribute the heat uniformly along the whole length and height of the oven walls; adapting the dimensions of the ovens and the coking time to suit the nature of the coal; improving the firebrick work so as to allow the side walls to be diminished in thickness, in order to give a more rapid conduction of heat to the ovens. Later, modifications have from time to time been made to collect various by-products from the so-called waste gases expelled from the ovens.

Variations in the rate of coking and temperature have been made to improve the yield of by-products. There is a limit to which this can safely be carried without deterioration in the quality of the coke, and thus adversely affecting the production of iron from the blast-furnace. The latter, though unchanged in principle for several centuries, still remains the most complex in principle and most difficult to operate of any metallurgical apparatus, and while there are so many factors involved which can only be regulated within comparatively wide limits, it is not only desirable but also very necessary that all factors affecting the operation of the furnace which can be controlled and regulated should receive very serious attention.

Among the chief of these is the physical and chemical composition of the coke used. The lack of regularity and uniformity in the quality of the coke is a very serious handicap to the producer of pig iron. The fundamental principle underlying successful operations at the blast furnace, is that the composition and quantity of the slag, together

with the temperature of the smelting zone and the hearth determine the quality of the iron produced. These in turn are decided principally by the quality of the coke used as fuel, and by the distribution of the burden. In the past the production of pig iron from the blast furnace has been largely a question of charging the materials in the quantities found by experience to give a good marketable product. Fortunately the range of composition of saleable pig iron has been somewhat wide, and it has been possible to grade the iron made, and send supplies to customers according to fracture or analysis of the finished product. Modern requirements, however, demand from the blast furnace a product within narrower limits of composition, and of greater uniformity than has hitherto been obtainable. Again, these demands are such that a slight modification in the furnace working may have a very detrimental effect upon the operations; in other words, the margin of safety of working is much restricted. Any irregularity in the blast furnace reacts more readily upon such iron than upon iron higher in silicon, as the margin of reserve heat is less, and the product is in danger of becoming useless, and at the same time the furnace becomes distinctly cold, and serious harm may result in producing "off grades" of pig iron.

The principal source of heat in the furnace is the coke charged, and any variation in its composition or heat-giving capacity at once affects the heat in the smelting zone and hearth of the furnace. With reservations which will be considered later, the available carbon of the coke is a measure of its heat-producing capacity.

Steel furnaces have in the past taken pig iron containing from 2% to 3.5% Si, largely because thereby they were more assured of an iron sufficiently low in sulphur, to ensure a steel within the limits allowed. With the introduction and development of the basic open-hearth process, lower silicon content in the iron is the practice—about 1%—while as the sulphur can be partially eliminated in this process, the maximum limit of sulphur can be raised above what is demanded for hæmatite iron for the acid process. To ensure a good output of steel, however, it is essential that the sulphur content must not be excessive. Using iron with 1% Si and below, it is more difficult to keep the sulphur regularly below 0.1% and also more difficult to control the furnace operations, than when making foundry iron or iron for the acid steel process.

With the extraordinary demand for various distillates to-day, coke is in danger of losing its supremacy, and of taking a very secondary place in the list of products obtained from the distillation of coal. There appears a wide field for research in the direction of ascertaining the best temperature and rate of gasification for each class of coal used, as coals from different depths and different districts have not the same coking powers.

The physical composition of the coke is no less important than the chemical character. The most important physical characteristic is the hardness or density, which is the measure of its resistance to abrasion or crumbling. A good hard coke will also produce more intense heat than soft coke. The efficiency of heat utilisation in a blast furnace depends upon the concentration of heat, and this is best produced by a hard fuel.

**Hardness test.** To make a hardness test, an average sample of coke is crushed in a Baxter crusher, or on a plate with the usual hammer, to pieces from  $\frac{1}{2}$  in. up to 1 in. across. This is sieved through a  $\frac{1}{2}$ -in. mesh, and that remaining on the sieve is dried at 100° C., and 28 lbs. weighed and placed in a drum or rattler 26 in. diam. and 16 in. depth, together with a dozen cast iron balls  $1\frac{1}{2}$  in. diam. The drum is given 1000 revolutions in about 1 hour, and the coke is withdrawn and



sieved on an  $\frac{1}{8}$ -in. mesh; that remaining on the sieve is weighed, and calculated to percentage of the original weight. This percentage is given as the hardness number.

For example, if 27 lb. 2 oz. remain on the  $\frac{1}{8}$ -in. sieve and 14 oz. passes through, the hardness is  $27.125 \times 100 \div 28 = 96.9$ .

**Available carbon.** The chemical composition of the coke resolves itself ultimately into the amount of "available carbon" which it contains. The value of the fuel is also affected by the physical nature of the coke. By the term "available carbon" is understood the quantity of carbon present in the coke in excess of that necessary to melt the slag formed by the ash and sulphur of the fuel, together with the limestone used to flux them. According to Forsythe the ash requires double its weight of limestone, and the sulphur three and one half times its weight to form slag. The slag formed requires 25% of its weight of carbon to melt it. When this is deducted from the fixed carbon the remainder is called "Available carbon." For example, a coke containing fixed carbon 86%, ash 10%, sulphur 1%, will require  $(2 \times 10) + (3.5 \times 1) = 23.5\%$  of its weight of limestone. With 55% of limestone passing to slag, then  $10 + 1 + (23.5 \times 0.55) = 23.9$  lbs. of slag per 100 lbs. of fuel.  $23.9 \times 0.25 = 5.97$  lbs. of carbon to melt the slag.  $86 - 5.97 = 80.03\%$  "Available carbon." Each 1% of ash affects the available carbon by 0.5% and each 0.1% of sulphur affects it by 0.08%.

TABLE I.  
*Beehive cokes.*

No.	Ash.	Sulphur.	Fixed carbon.	Available carbon.	Hardness.
	%	%	%	%	
1. ..	7.29	0.89	91.86	87.38	96.5
2. ..	9.42	0.98	89.25	83.59	97.2
3. ..	12.33	1.15	86.85	79.54	94.4
4. ..	11.89	0.88	87.45	80.43	96.4
5. ..	7.88	0.97	91.52	86.67	96.3
6. ..	13.24	0.84	86.06	78.50	94.2
7. ..	9.96	0.81	89.55	83.53	97.5
8. ..	9.78	1.03	89.19	83.30	97.0
9. ..	9.33	0.96	89.89	84.29	97.3
10. ..	8.14	0.81	91.00	86.14	95.3
11. ..	8.50	0.74	90.79	85.79	96.9
12. ..	8.79	1.11	90.43	85.00	93.7
13. ..	8.57	1.04	90.70	85.44	95.8
14. ..	7.18	0.86	92.17	87.77	96.4
15. ..	10.05	1.11	87.92	81.83	95.9
16. ..	5.67	0.90	92.56	88.91	95.7
17. ..	11.23	1.23	86.31	79.52	95.5
18. ..	7.77	1.15	90.20	85.28	94.0

TABLE II.  
*By-product cokes.*

No.	Ash.	Sulphur.	Fixed carbon.	Available carbon.	Hardness.
	%	%	%	%	
19. ..	8.21	1.01	90.00	84.95	96.5
20. ..	7.90	1.55	89.80	84.50	96.4
21. ..	10.30	1.60	87.47	80.90	96.4
22. ..	10.20	1.04	88.23	82.12	95.5
23. ..	7.50	1.18	90.69	85.89	96.2
24. ..	8.50	1.35	89.52	84.07	96.0
25. ..	8.55	1.18	89.51	84.16	96.4
26. ..	7.39	0.99	90.72	86.12	91.7
27. ..	7.29	1.00	91.90	87.34	93.6
28. ..	9.80	0.91	88.55	82.74	93.4
29. ..	7.87	0.80	91.66	86.94	97.5
30. ..	8.85	0.78	90.38	85.17	96.4

All the analyses have been given on the dried coke in order to make them more comparable. The influence of appreciable variation in the amount of moisture contained in the coke can easily be understood.

Cokes Nos. 20 to 28 inclusive were made from

differing classes of coal in the same set of ovens, and as nearly as possible under similar conditions, each extending over one week, and afford a striking example of the need for studying the correct temperature and period of coking for each class of coal.

Cokes below 94 hardness caused serious trouble in working and resulted in the furnaces scaffolding or hanging and getting decidedly cold. Cokes below 85% available carbon when in use have caused irregularities if burdened at the same rate as others, evidently from shortage of sufficient heat-producing carbon. The best all-round cokes are Nos. 16 and 29.

These tables emphasise the desirability of using one class of coke exclusively upon one furnace if sufficient supplies are available. Where such a procedure has been adopted more uniform results in the working of the furnace and the quality of the iron have been observed. For example, the difference in the quantity of available carbon between cokes 16 and 17 is equivalent to 2 cwt. per ton of iron. Consider a furnace working regularly and satisfactorily on coke No. 16, changed to No. 17 without any account being taken of its inferiority, the result would be equivalent to taking off the burden 2 cwt. of coke per ton of iron made, a change no one controlling furnaces would knowingly make; yet such is the practice at many blast-furnace plants of mixing in the same bunker small parcels of coke from different ovens and different coals, that some such violent change as instanced above will take place.

The bad effect of soft coke is cumulative and proportionate to its softness, owing to its liability to crumble during handling between the ovens and the furnace bell, resulting in a large quantity of breeze and small coke being charged into the furnace, and also owing to the crushing of the coke in the furnace. Both these causes result in less available carbon reaching the tuyères than would be the case with harder material.

**Sulphur.** The function and behaviour of sulphur in the furnace have been exhaustively investigated by Wüst and Wolff in 1905; their conclusions may here be given:—"Contrary to the generally held opinion, the sulphur in the coke does not reach the level of the tuyères of the blast furnace without undergoing alteration, but a great portion of it is previously volatilised by the ascending gases, and is then largely absorbed from the gases by the descending charge, and in this condition arrives in front of the tuyères. Up to 800° C. the sulphur is principally absorbed from the sulphur-laden gases by the oxides of iron, while from 800° upwards the position is reversed and the lime becomes the chief absorbent of the sulphur."

**Determination of moisture.** The moisture in coke is all superficial and is in no way combined with the coke substance, hence coke cannot be crushed without losing moisture. It is a mistake to crush a sample as finely as is usual for coal or other minerals; anything up to 1-in. cubes should be taken, and say 1000 or 2000 grms. weighed out, and dried at any temperature between 100° and 200° C. Samples for moisture determinations should be dried as quickly as possible after taking, and should be kept in an air-tight container if necessary to convey them any distance.

#### DISCUSSION.

Dr. J. E. STEAD wrote that in his experience the densest and hardest retort carbon had to be used in order to produce the highest temperature in their small furnace for determining the melting point of firebricks, etc. With cold blast they attained to a maximum temperature of 1750° C., whereas ordinary coke of the porous variety would not give anything like that temperature, and charcoal, which was still more porous, gave a still lower result, so there seemed to be some doubt

whether hard, porous coke was best for giving the highest hearth temperatures.

Mr. G. WEYMAN said that it was a matter of great difficulty to get uniform coal from the pit. In one case he had tested each waggon of a train of 15 cars of coal from one colliery; great care had been taken in the sampling. The ash in individual waggons varied from 3% up to 24%, and the average of the whole train-load was about 10%.

Mr. E. F. KNOTT referred to the question of phosphorus, which was a matter of considerable importance to coke makers at the present time when low-phosphorus coke commanded an enhanced price.

Mr. A. SHORT remarked that coke suffered a great deal in transport. Users ought to see that it was carefully handled and not allowed to get crushed more than could be helped. He suggested that coke makers ought to establish a joint large-scale experimental plant where various coals could be coked in different ovens and under different conditions of coking. He asked what hardness in Mr. Hewson's determinations would be satisfactory.

Dr. S. H. PATERSON, referring to Dr. Stead's statement, suggested that the temperature attainable was probably dependent upon the depth to which surface combustion took place beneath the face of the fuel. Were this determined, it might explain the differing calorific intensities of various forms of carbon.

Mr. C. H. RIDSDALE said that the densest coke that could be produced was porous enough for blast furnace purposes. A big coke, like beehive coke, helped to keep the furnace very open, as now, more than ever, the tendency was to get small ores. With small ores and small coke, it was not possible to drive the furnaces. 18 cwt. of large coke would give better working and just as much effective heat as a ton containing 2 cwt. of dead small. In a particular furnace working (basic), where a large proportion of the material was reduced by solid carbon on the hearth, the coke consumption being 27 cwt. per ton of pig, an efficiency of only 2300 heat units per unit of coke was obtained. The loss of 1153 heat units, equivalent to  $\frac{1}{2}$  cwt. of coke, was sufficient to reduce the quality of pig from open grey mottled to ordinary white—the former a perfectly good iron for steel-making, the latter unsuitable. That same  $\frac{1}{2}$  cwt. difference in coke equivalent was equal to a reduction in quantity of iron produced of 0.8 cwt. in pig, the iron passing into the slag instead of being reduced and passing into the pig, and raising the slag for a quarter of an hour (the time it took to make a ton of pig) to a 2.44% iron content. The same effect could be produced by a variety of causes, such as leakage of water at the tuyères, drop in temperature of blast, badly calcined stone reaching tuyères, etc., and, amongst others, a drop in quality of coke used, due to more ash, and hence less carbon. In 1900 he had made some careful balance-sheets of heat received and consumed by blast furnaces with coke consumptions varying from 21 to 31 cwt. The data so obtained had constantly proved to be reliable in practice. They showed clearly that, however high the coke consumption, of the whole coke charged most of the heat yielded went in doing what might be termed "dead work"—work the effect of which was not apparent (effecting the necessary chemical changes, heating the escaping gases, etc.), and that it was only the small balance over what was necessary for those purposes—namely, a very small excess of heat—which, once the "dead" requirements were satisfied, produced the effects noticed on grade of iron. The effects of small variations of heat available were thus accentuated when they occurred in the hearth. Thus, in the case of a furnace using 27 cwt. of

coke, the heat produced by 21 cwt. was used up for purposes other than the fusion of slag and pig, and only that from 6 cwt. was used in the hearth for that fusion. Looked at in that light, one got a very different impression of the effect of  $\frac{1}{2}$  cwt. of coke or its equivalent, for it equalled 81% of all used in the hearth. That explained why any drop in quality, either chemical or mechanical, abstracting so large a proportion of the heat from the hearth, told so heavily on the product. As to the size of the coke, the breaking from large to small pieces was not so important as the crushing to fine dust, which blew out into the flues and gave trouble in the dust-catchers and the flue-dust.

Dr. J. T. DUNN said a question arose as to what was meant by "hardness." He thought it was more a question of what had been termed "friability." He had always understood that the requirement in coke was that it should not, in passing down the blast furnace and rubbing about the walls, etc., produce fine coke, but that it should simply break up into smaller pieces—that, in fact, the ultimate portions of the coke should be hard, and that it was not quite so important that the block as a whole should be hard.

Mr. RIDSDALE remarked that it was not that the coke would not resist the static pressure. What was wanted was resistance to breaking up and crushing when coke was dropped first into the trucks, then into the bunker, and finally into the furnace, and then had several tons of heavy ore dropped on top of it.

Mr. HEWSON, in reply, said that he intended making modifications in his method of testing hardness of coke and hoped by this to get a sharper line of division between good and bad coke. At present he considered coke satisfactory if the hardness number was above 94. Gas coke would only give a hardness number of 90–92.

The CHAIRMAN said that washing coal prior to coking would no doubt improve the chemical composition, but he rather thought that it had not a beneficial effect upon the physical properties of the coke.

## Nottingham Section.

*Meeting held at Nottingham on Wednesday,  
January 23rd, 1918.*

DR. R. M. CAVEN IN THE CHAIR.

### SOLUBLE GLASS.

BY R. M. CAVEN, D.S.C. (LOND.), F.I.C.

Sodium metasilicate is readily soluble in water, from which it separates in hydrated crystals having the composition  $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$ . Such a silicate however, is of little if any commercial interest, the kinds of soda-glass in use for various purposes having a ratio of silica to soda by weight of at least 2 to 1, and frequently 3 to 1, or even more. In the former case the product is known technically as "alkaline silicate," in the latter as "neutral silicate."

These silicates are not chemical compounds, since they may contain silica and soda in varying proportions. They probably consist of solid, anhydrous solutions of the monosilicate,  $\text{Na}_2\text{SiO}_3$ , or perhaps the disilicate,  $\text{Na}_2\text{Si}_2\text{O}_5$ , in excess of silica.

Glasses so rich in silica are not readily soluble in water; indeed, even when finely pulverised they dissolve but slowly and imperfectly in boiling water; and the larger the proportion of silica the less is the solubility.



It is necessary for soda-glass to be dissolved in water, *i.e.*, to become in reality soluble glass, before it can be technically effective. This is true whether it is to be employed in the well-known form of a viscous solution or in a solid state.

Soluble glass solution is at present prepared in either of two ways. In the first way finely divided silica, in the form of flint, infusorial earth, quartz, etc., is heated with caustic soda solution under a steam pressure of three or four atmospheres. At the elevated temperature thus attained by the alkaline liquid the silica slowly dissolves, and the dilute solution of soluble glass which results is subsequently evaporated to the desired consistency.

In the second, and probably more usual way, sodium carbonate, or a mixture of sodium sulphate and coal, is fused with the required amount of silica in a furnace at a temperature of about 1100° C. The result is a hard, transparent glass, which is broken mechanically, or else cracked by dropping it whilst molten into water, and is subsequently dissolved in water.

How this dissolution may be best effected has been the subject of much investigation, but the method which has survived and is employed at the present day consists in heating the broken glass with water under a steam pressure of about 5 atmospheres. The glass yields to this treatment with water at a temperature of about 150° C., just as silica does when heated similarly with dilute alkali; but the resulting solution is dilute, and needs to be evaporated considerably to obtain a solution of commercial utility; and, moreover, it cannot easily be evaporated to dryness because of the formation of a skin on the surface of the colloidal liquid as it becomes concentrated; and if this difficulty is overcome intumescence occurs later, so that what eventually results is a more or less solidified froth.

The preparation in the solid form of a soda-glass readily soluble in water is, however, very desirable, not only to save the difficulty and cost of carriage of the liquid solution, but because the solid itself would have very distinct uses. (See Henkel, Eng. Pat. 23,391, 1911, this J., 1912, 489, and R. F. Bacon, this J., 1917, 36, 11.)

Methods of preparing such a solid, soluble glass from the almost insoluble glass obtained by high temperature fusion have now been discovered (Spensley, Battersby, and Holmes, Eng. Pats. 11,959, 1914; 108,576, 1917; Caven, Eng. Pats. 106,247, 1917; 108,372, 1917; this J., 1915, 834; 1917, 712, 1007), and it is the purpose of this paper to explain the chemical principles underlying these methods.

Briefly, the processes consist in grinding the nearly insoluble glass either with water (Spensley, Battersby, and Holmes) or dry (Caven), and suitably heating the product, thoroughly mixed with a small amount of water, until a vitreous solid results, which dissolves readily and almost completely even in cold water.

This statement of procedure, taken in conjunction with the fact that agitating the same powdered glass with excess of hot water has little effect upon it, points to the conclusion that the quantity of water employed influences the degree of solution which takes place, and that in a sense opposite to what is usual. Ordinarily the larger the amount of solvent employed the more readily will a solid be dissolved; in the solution of soda-glass, however, excess of water hinders solution, and the reduction in the amount employed down to a certain minimum promotes it.

The following results show that this is the case. In these experiments finely ground neutral silicate was employed in which the silica-soda ratio was 2.91:1. This was shaken with water in the proportion of 1 of silicate to 1, 2, 3, 4, and 5 of water,

for 6 hours at atmospheric temperature. The bottle containing the 1:4 mixture was broken, but the solutions in the other bottles were found to be of the following relative strengths, much solid remaining undissolved in each case:—

*Solid contents of equal volumes.*

Glass: water.	SiO <sub>2</sub> .	Na <sub>2</sub> O.	Total.	SiO <sub>2</sub> :Na <sub>2</sub> O by weight.
1:1 ..	0.0754	0.0321	0.1075	2.35
1:2 ..	0.0346	0.0159	0.0505	2.18
1:3 ..	0.0263	0.0107	0.0370	2.44
1:5 ..	0.0140	0.0066	0.0206	2.13

SiO<sub>2</sub>:Na<sub>2</sub>O in original glass=2.91.

These figures plainly show that the concentration of the solution formed diminishes as the proportion of water used increases, and indeed the total amounts of solid matter in solution are found to vary almost exactly in the inverse ratio of the amounts of water employed. A further interesting point is brought out by the ratio of silica to soda in solution. This is considerably less than in the original anhydrous glass; that is to say, the glass has not dissolved as a whole, but there has been a preferential solution of soda, so that the glass remaining undissolved is relatively poorer in soda than the original glass.

Similar results were obtained when the same powdered neutral silicate was agitated with water in tubes immersed in boiling water, in this case for half an hour. The solutions were in each case filtered and diluted when cold to 100 c.c. Aliquot portions taken for titration and evaporation then yielded the following results:—

*Relative strengths of equal volumes.*

c.c. water per 2 grms. powdered glass.	SiO <sub>2</sub> .	Na <sub>2</sub> O.	Total.	SiO <sub>2</sub> :Na <sub>2</sub> O by weight.
10.....	0.0782	0.0288	0.1070	2.72
14.....	0.0535	0.0208	0.0743	2.57
18.....	0.0423	0.0164	0.0587	2.59
22.....	0.0340	0.0136	0.0476	2.52
26.....	0.0269	0.0115	0.0384	2.33

SiO<sub>2</sub>:Na<sub>2</sub>O in original glass=2.91.

The same diminution in solubility on dilution is observable here as in the experiments with water at atmospheric temperature; and the silica-soda ratio is also less than in the original glass, although a little higher, especially with the smaller amounts of water, than with the cold water.

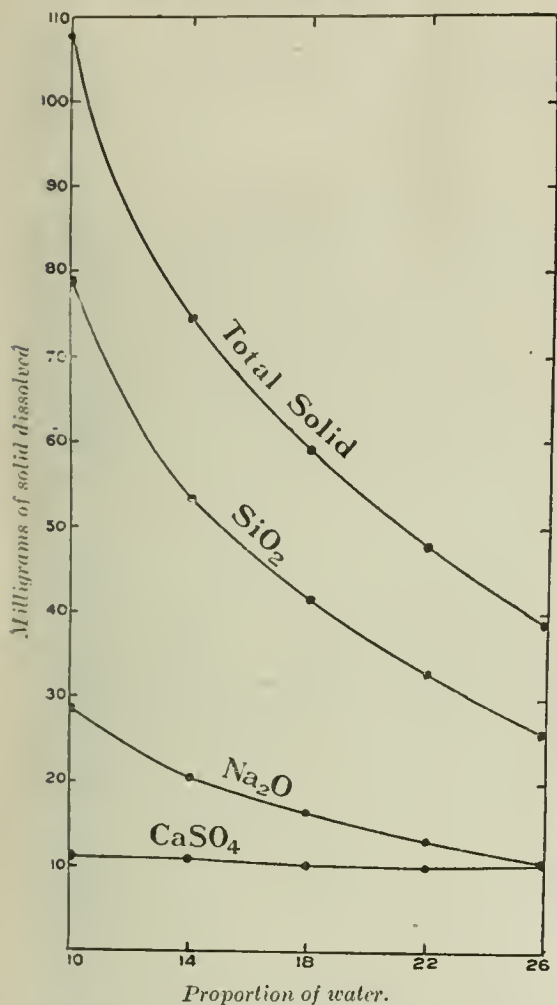
In order to make a comparison between the solubility of the soda-glass and that of an ordinary sparingly soluble salt, quantities of purified calcium sulphate, weighing 2 grms. each, were shaken in the same way with amounts of hot water equal to those employed in the preceding experiment.

Five c.c. portions of each of the filtered solutions were then evaporated and ignited, and the following weights of anhydrous calcium sulphate obtained in the five cases: 0.0112, 0.0113, 0.0100, 0.0104, 0.0108 gm. Thus the solutions formed were of the same strength, irrespective of the quantity of water used, and the above amounts of calcium sulphate correspond to saturated solutions of the salt.

That the behaviour of soda-glass towards water is in marked contrast to that of a sparingly soluble salt which dissolves without chemical change, is strikingly manifest when the above results are shown in the form of curves (see fig.) Thus while the solubility of calcium sulphate, which is independent of the quantity of water used, is shown by a horizontal straight line, that of soda-glass is

represented by a curve which descends with increasing amounts of water. So it is clear why the glass cannot be dissolved in much water. And further, the soda-curve is less steep than the silica-curve, owing to the preferential solution of the soda.

From these results two conclusions may be drawn: first that if the glass is to be dissolved



effectively in water the minimum amount of water must be employed; and second that if it is possible for the glass to dissolve as a whole, without preferential solution of soda, this, too, will take place with the minimum amount of water.

A modification of the above experiments bears out these conclusions. If instead of being agitated with hot water, the finely divided glass is allowed to lie in a layer at the bottom of the containing vessel beneath the water, its particles gradually cohere to form a vitreous mass, and the supernatant liquid is found to contain silica and soda in the ratio in which they exist in the anhydrous glass, the solution having been gradually formed by diffusion from the vitreous product below it; and after sufficient time has elapsed the latter is found to be completely soluble in water in all proportions.

The supernatant water may be omitted; that is to say the powdered glass may be mixed with not more than enough water to cause it to vitrify when heated to a temperature approaching

100° C. for about four hours. The product is then a hard, tough glass which can be reduced to powder and dissolved in any proportion of water.

To produce this result sufficient water must be mixed with the finely powdered, anhydrous glass to make a thick paste in which every particle is thoroughly wetted. From 2 to 3 parts by weight of water to 8 parts of the glass is a convenient proportion. If more than this proportion of water is used and the mixture is agitated during heating the resulting hydrated glass will not be perfectly solid, but slightly viscous like pitch; whilst if the mixture remains quiescent during heating the product will be in two layers, the upper of which will be a viscous fluid, and the lower a vitreous solid. Eventually, by the addition of more water, a limit will be reached beyond which, if the mixture is agitated, some of the glass will not become hydrated or be rendered soluble. If much less than the above proportion of water is used the glass particles will not be completely vitrified, but instead will form a caked mass, physically resembling sandstone, in which unchanged particles of the anhydrous glass are embedded in a matrix of the soluble, hydrated glass. Thus Henkel (Eng. Pat. 9730, 1898, this J., 1898, 763) mixed water with the powdered soda-glass in the proportion 1:6, and heated the mixture to 120° C. The result was an opaque mass which disintegrated and partially dissolved in water, but left behind much of the original glass unacted upon.

The following are the percentages of water, and residue after stirring with much hot water, found in a specimen of hydrated neutral soda-glass obtained by the above-described process (I.), together with the percentages of the same components in the product obtained from the same powdered glass by the Henkel process (II.).

	I.	II.
Water, % .....	23.2	16.2
Insol. matter, % .....	0.9	24.9

The reason for the above difference lies chiefly in the deficiency of water used in the second case, though partly also in the high temperature employed in the early stages of heating. The amount of water used should be sufficient to leave not less than 20% in the finished product, and the initial temperature should be about 70° C. Very little water then evaporates though the final temperature should rise to 100° C.

The conditions for the production of a hard, vitreous, soluble, hydrated soda-glass are thus the following:

(i.) The anhydrous glass must be pulverised sufficiently; it should pass through a sieve with 50 meshes to the linear inch in the case of the neutral glass, though a sieve of 20 meshes suffices for the alkaline glass. The powder should not be exposed to the air unduly, since it readily absorbs water and carbon dioxide. This is especially the case with the alkaline silicate.

[Some of the pulverised neutral silicate which had been kept in a screw-top bottle for about twelve months was found to contain 18% of water and 4% of carbon dioxide. It did not yield a good hydrated product.]

(ii.) Sufficient, but not too much water must be mixed with the pulverised glass; the weight of water should be between 25 and 38% of the weight of glass.

(iii.) The mixture, which may be placed in a flat cake tin, coated inside with lime, and loosely covered, should be heated sufficiently, but not too strongly. The initial temperature should be about 70° C. and the final temperature, after about four hours' heating, should not exceed 100° C.





manufacture, and which contained only 0.03% of iron, together with some commercial sodium carbonate, containing a trace of iron. The green tint of the resulting glass was very pale, but the hydrated glass prepared from it was decidedly grey. Moreover it is obviously useless to counteract the green tint by manganese oxide, since this would only aggravate the colour on hydration.

Fortunately, however, the dark colour of the hydrated glass is not detrimental when the latter is to be employed for making solutions of soluble glass, since the liquor may be bleached by oxidation, and then all the iron is found in the small sediment that remains after settling, from which a clear and practically colourless solution can be withdrawn.

#### DISCUSSION.

Mr. W. A. RICHARDSON considered that Dr. Caven's work threw light upon the problems of flint in at least two points. In the first place his contention that the colour was due to ferrous-ferrie oxide was in agreement with such evidence as was available. Most authorities (*e.g.*, James Geikie and Prof. Sollas) stated that the flint colour was due to the presence of carbonaceous matter. If any carbon were present in the flint only a minute quantity had ever been proved, and this was, judging from the amount present in other rocks coloured black by carbon, quite incompetent to produce the intense black coloration of flint. On the other hand analyses given by Hardman (*Sci. Trans. Roy. Soc. Dublin*, 1878, p. 85) for Irish cherts showed 1.15%  $\text{Fe}_2\text{O}_3$  and 0.15%  $\text{FeO}$ . This he gathered from Dr. Caven's investigations was ample for the purpose. Another point which struck him as interesting was the hardening of the artificial glass on loss of water. This might have some parallel in the weathering of flint. In the analysis quoted above the loss on ignition was 1.43%. In the weathering of flints, particularly in the Thames Valley gravels, he had noticed the following points:—

(A) A probable loss of water accompanied by a gradual leaching out of the iron content. The flint turned to paler and paler shades of grey and was finally completely bleached. It also became more porous as shown (1) by the fact that it was opaque, not transparent or translucent as the black flint would lead one to expect, and also (2) by its subsequent absorption of red hydrated ferric oxide, in which the gravels abound. This absorption gave the flint a secondary brown to red staining.

(B) The flint became harder probably owing to the loss of water from solid solution. This point was illustrated by the use of fresh chalk flints in S.E. England for building, in preference to those picked from the surface of the land, because these latter were too hard to dress (*J. A. Howe, "Geology of Building Stones," p. 328*).

(C) The weathering action often etched the surface, giving it a pitted appearance showing that a portion of the flint was less resistant to weathering agents.

Mr. H. DROOP RICHMOND asked if it was not possible to prepare sodium silicate free from iron by the use of precipitated silica. In regard to Dr. Caven's scheme illustrating the mechanism of gradual solution, he wondered if some advantage would not accrue, if in place of sodium hydroxide and hydrated silica the existence of two distinct sodium silicates were postulated, one rich in soda, the other poor, relatively to silica. In fact he wondered whether the alkali was as free from the silicic acid as the scheme suggested. One of the conventional tests made use of in the technical examination of commercial sodium silicates was a determination of the free alkali that remained in solution after precipitation with barium chloride. Had Dr. Caven any knowledge

as to the composition of barium silicate produced under such conditions (alkaline precipitation)? When determining sulphate in water analysis he had obtained high results by direct precipitation with barium chloride, because the silica in solution had been precipitated by the barium. The composition of the barium silicates need not of necessity be similar, but certainly they possessed more than academic interest.

Mr. J. M. WILKIE, referring to Mr. Richmond's remarks, expressed a preference for the postulation of a number of silicates of varying contents of sodium and silicic acid. In such concentrated solutions hydrolytic dissociation could not be anything like complete, but, of course, this did not in any degree invalidate the value of Dr. Caven's schematic treatment. It appeared to him that the composition of barium silicate would vary with the conditions of precipitation and that the ratio of barium oxide to silica would be affected by adsorption and the presence of the ions in solution.

Dr. NAEF said that it was possible to obtain silica free from iron by dialysis and that such a product might be utilised in an attempt to make a sodium silicate free from iron.

Mr. CALAM described an experiment in which coal gas had been burnt in a glass vessel; during the combustion of the gas a layer of iron oxide had been precipitated on the glass. The presence of the iron oxide was attributed to the decomposition of iron carbonyl present. He also referred to another experiment in which powdered window glass had been heated with a small quantity of water under pressure, the resulting product having been a solid mass of glass. The suggestion was put forward that possibly quartz had been formed from a mineral like geodite in a similar way.

Mr. LEWIS stated that at the present time, large quantities of silicon were being manufactured, by the electric furnace process for the purpose of generating hydrogen by treatment with caustic soda, and suggested this method as a possible means of obtaining pure sodium silicate.

Dr. HEDLEY said that he had made soluble glass on a large scale but had never succeeded in rivalling the pale green specimens shown. He remarked on the very minute traces of impurities required to produce coloration, referring to the work of Verneuil, who had shown in 1911 that the colour of sapphires was due to traces of titanium oxide—from 0.03 to 0.06%—and who had made artificial sapphires containing minute amounts of iron and titanous oxides. Again, the colour in diamonds was due to even more minute traces of iron, titanium, and manganese, and since such deep colorations were produced by mere traces it was not to be wondered at that very great difficulty was experienced in producing a colourless silicate, even when using pure quartz and sodium carbonate. In connection with the experiment described by Mr. Calam of treating window glass with water at a high temperature, he referred to the proposed process for the manufacture of potassium compounds, which was based upon the fact that feldspar on treatment with caustic soda at 270°–300° was changed into an artificial leucite (*this J.*, 1917, 1234). With regard to obtaining sodium silicate as a by-product in the production of hydrogen, he stated that the cost of preparing one cubic metre of hydrogen from silicon and caustic soda was 53 pence, from ferro-silicon 10 pence, and from water and iron 1.5 pence. Thus in order to be a commercial success the silicate resulting from the reaction would have to pay for the difference between 1.5 pence and 53 pence; so that as to whether its collection would "pay" seemed highly problematical.

Dr. CAVEN, in replying, said with regard to Mr. Droop Richmond's remarks that the difficulty



of obtaining a colourless sodium silicate would not be overcome by using iron-free silica, since commercial sodium carbonate contained traces of iron, and there was the probability of iron contamination from the crucibles employed for the fusion. He had prepared some alkaline silicate from colourless quartz and commercial sodium carbonate and the colour of the product was pale green, resembling that of the best commercial specimens. He saw no objection to the suggested alteration of the theory of the process of hydration, since the essential principle was unaffected thereby. Whether or not free sodium hydroxide was produced on hydrolysis might be a question of dilution; certainly it was formed in the presence of much water. Sodium metasilicate appeared to be hydrolysed in aqueous solution according to the following scheme:

$$2 \text{Na}_2\text{SiO}_3 + \text{H}_2\text{O} \rightleftharpoons \text{Na}_2\text{Si}_2\text{O}_5 + 2 \text{NaOH}$$

(Kohlrausch, *Zeits. physikal. Chem.*, 1893, 12, 773; Kahlenberg and Lincoln, *J. Phys. Chem.*, 1898, 2, 77). He could give no information on the properties of barium silicate. In reply to Dr. Hedley, he was surprised to hear of fused sodium silicate which was almost black. Undoubtedly the pale green glass was a commercial product.

## Yorkshire Section.

*Meeting held at Leeds on Monday, February 4th, 1917.*

PROF. J. W. COBB IN THE CHAIR.

## MODERN METHODS OF SULPHURIC ACID MANUFACTURE.

BY G. L. MOSS.

The heavy chemical industry, in which this country has held her own, has of late years been inclined to rest on its laurels, and it needed the stimulus of war to arouse it to a sense of its inefficiency.

To take the case of the sulphuric acid manufacturer, how many could honestly answer "yes" to all the following questions:—

Are you getting all the available sulphur out of your raw material?

Are you getting the greatest possible output of acid per cub. ft. of chamber space?

Is your nitre consumption at an irreducible minimum?

Are your repairs also at a minimum?

Can your plant deal with any class of ore on the market without prejudicing your output and quality of product?

Are you getting the greatest possible output per unit of ground space?

It is gratifying to know that in this industry there have been many important developments during recent years, and the author thinks it will be of general interest to record the main lines on which these have been made.

The conventional method of erecting a sulphuric acid plant was to make the structural work of the towers and chambers entirely of wood. The chambers were supported by heavy stanchions and joists to which the lead was fastened by straps; the tops were usually left exposed to the weather, and sometimes the sides as well. As a rule, however, the sides were protected by a sort of matchboarding with gaps in the joints for air circulation. The tops of the towers housing the circulating tanks were similarly covered.

The towers were usually square in cross-section and the Glover was often packed with large lumps

of lava stone, flints, etc., the Gay-Lussacs with coke. Now for towers which are essentially scrubbers, both the shape and the packing were wrong. Owing to the timber employed in construction, however, round towers were not a feasible proposition, but during recent times more efficient means of packing were employed. Blocked or partially blocked Glovers were common owing to accumulation of dust and they could not be easily washed down. "Ratholes" frequently developed in the coke packing of the Gay-Lussacs, resulting in the gas traversing one side of the tower and the acid running down the other, with consequent loss of nitre. Many plants in this country to-day have only one Gay-Lussac tower. Lunge states that he has proved that coke in Gay-Lussacs has a reducing effect on nitrous gases. There are now many forms of patent packings on the market to-day, but in the author's opinion, good vitrified chequer brick for the Glover and rings for the Gay-Lussacs are the most satisfactory.

The fifty-second annual report of the Chief Alkali Inspector for the year 1915 (this J., 1915, 1011) states that of 338 Glover towers in the United Kingdom, 130 were packed with flints, 127 with bricks, 28 with bricks and flints, 23 with rings, 16 with bricks or tiles and rings, 8 with tiles, and 6 with bricks and tiles; and of 440 Gay-Lussac towers, 269 were packed with coke, 58 with bricks, 43 with glass, 25 with rings, 17 with tiles, 10 with bricks and tiles, 8 with rings and coke, 7 with bricks or tiles and coke, 4 with rings and tiles, 3 with Lunge-Rohrmann tiles, and 1 with balls.

The old chambers naturally varied in size with the output of the plant and they frequently attained a length of some 120 ft. The height usually ran from 15—20 ft., and the cross-section was approximately square. A cubical capacity of anything up to 30 cub. ft. per pound of sulphur burned per 24 hours was allowed, though in recent times this has been reduced to from 12 to 20 cub. ft. Steam was used as a rule in the chambers, being introduced either at the top or ends—if the latter, frequently in a single jet. The trunks connecting the towers and chambers were square in section and braced with timber in a similar manner to the chambers. All this timber naturally rendered the plant a prey to fire, and several plants have been completely destroyed by this means. Apart from this danger the structure was very flimsy and prone to rot. The wood under the influence of acid and heat would perish, and unless constantly renewed when this happened, was liable to collapse under wind pressure. This is no uncommon occurrence, and several chambers have been literally blown down. The method of strapping the lead was very rigid and left no room for movement under expansion and contraction. Tearing of the straps on the chamber sides resulted.

On the older types of plant hand burners were usual, burning lump ore. With this type of burner the sulphur in the burnt ore is usually high, the gas of uneven composition, and labour costs high. There is not much trouble with dust as a rule. On the introduction of mechanical burners burning fines, sulphuric acid makers began to realise what dust troubles are. The mechanical burner gives much greater efficiency in abstracting the available sulphur from the ore and a constant gas composition. At the same time much less labour is necessary for a given output. The great disadvantage is dust, and when burning some of the low-grade Spanish and Portuguese ores the trouble is very serious. No dust chamber ever built can extract it all. It is in such an extremely fine state of division that it defies all attempts at settling it out and rapidly turns the Glover acid into a thick porridge, blocking the packing at the same time. This impedes the denitration of the strong nitrous vitriol and increases the nitre con-

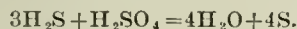
sumption. The usual method is to flush the tower down with chamber acid from the overhead feed tank, which always upsets the working of the chambers for 6 to 8 hours, with further loss of nitre. Efforts to overcome these troubles by increasing the size of the dust chambers, introducing filtering materials, etc., have not been successful. Too large dust chambers sometimes mean insufficient heat for the Glover tower. The author hopes to show how these difficulties have been successfully overcome, whereby practically water-white Glover acid, absolutely free from dust, has been produced.

For purposes of comparison one or two more features of the older plants are worthy of mention. On hand burners, only one fan is necessary and this is situated as a rule between the two Gay-Lussacs (where two are employed). This was usually steam driven and consequently subject to varying speeds, causing unequal gas composition by varying the draught. Very often no fan at all was used, the outlet being connected with a stack. Wind speed and direction here had its influence on the draught. Even with suitable chambers it was impossible to get a low chamber space owing to the varying conditions imposed by the appliances then in use. The essence of intensive working is constant gas composition and nitre supply in the first place.

The old means of elevating the circulating acid was by intermittent eggs—egg-ended castings holding two or three tons, which were filled and blown up by operating the valves by hand.

One of the principal features of the plant to be described, is the method in which de-arsenication is effected. Before describing the details of this system, it will perhaps be of interest to outline briefly the old method with its attendant difficulties.

The most generally applied process is that in which hydrogen sulphide is employed; the arsenic being precipitated as arsenious sulphide. The gas is usually generated by the action of weak sulphuric acid on ferrous sulphide. The gas is passed up a short tower, down which the acid is fed, afterwards running to the filtering apparatus, where the liberated arsenious sulphide is separated. The acid goes to the concentrators and the sulphide mud is periodically dug out of the filters, after being washed with water. Another form of apparatus is that in which the contact between the gas and acid is effected by a rotating splashing device. Whatever apparatus is used, however, the process suffers from a grave disadvantage, viz., that effective de-arsenication and filtration can only be carried out at an inconvenient dilution of the acid, between 100°—112° Tw. (sp. gr. 1.50—1.56), which is considerably below the strength of chamber acid made at most English works. At higher concentrations the acid is decomposed by the hydrogen sulphide according to the equation:—



The precipitated sulphur is exceedingly difficult to filter and causes trouble in the subsequent concentration. Any nitre present in the acid consumes hydrogen sulphide and thus increases the cost of the process. It is evident, therefore, that to dilute Glover acid of 140° Tw. (which holds the most of the arsenic) to a convenient strength for de-arsenication is a troublesome and costly process, both as regards the bulk of liquid to handle and the fuel required to reconcentrate it. As a rule it is not de-arsenicated and is sold with all its dust and arsenic. If it is de-arsenicated at all it is not usually diluted, and what is regarded as the lesser evil, i.e., precipitated sulphur and high hydrogen sulphide consumption, is allowed to take its chance. If the arsenic content is high it

has to be passed more than once through the apparatus.

The plant to be described is designed and erected by Messrs. Simon-Carves, Ltd., of Manchester. Even at first glance, there is a pleasing contrast between the new and old systems. The neat and compact brick buildings at once create a striking comparison with the old timbered structure, with its rambling outline and towers stuck at different corners. Closer examination reveals the great improvements effected from a chemical engineering standpoint.

In the first place the plant is quite weatherproof and practically fireproof (insurance only costs about 1s. 6d. per cent.) The only timber employed in its construction is for the chamber floors and the distributor platform. The method of suspending the lead (which will be later described in detail) and its protection from the weather, combined with the extra cooling effect obtained by the greater height of the chambers, contribute in an ideal way to intensive working and long life.

The whole chamber house is made up of a skeleton framework of steel and built in with 4½-inch brick suitably pigeon-holed to provide air circulation. A large glass louvre at the apex of the roof provides an exit for the warm air and ample illumination.

The burner house is of more solid brick construction and is well lighted and ventilated. It provides housing for the burners and dust chamber, the pyrites elevating machinery, together with the gas heater, and, in a separate room, the machinery for propulsion of the gases, compressed air pumps, etc. The towers are situated in a traverse of the chamber house and are all neatly arranged side by side.

The burners consist of a steel casing enclosing a series of firebrick arches swept by revolving arms which are actuated from a central vertical shaft. The special features of these burners are the following:—The shaft and arms are air-cooled, the air being supplied by a fan blast. The hot air, after leaving the top of the shaft, serves to dry the pyrites spread on a specially constructed floor, before being fed into the hoppers. The arms are held in place by an ingenious locking device which at the same time permits their easy removal and replacement. The burners are chain-driven from a shaft running alongside in a trench, each burner being provided with a clutch. A trip arrangement automatically unmeshes the drive in case of overload. Each burner has a rated capacity of 8 tons of ore per day with a 50% load variation either way. Only ¼ horse-power is required to drive it.

The gases from the burner trunks, containing up to 10% of SO<sub>2</sub>, are led into the dust chamber, where a preliminary settling of the dust is effected. They afterwards pass to the wash tower (instead of, on the old system, the Glover tower), where the dust is removed, together with most of the arsenic, by a copious flood of acid. By this means they are cooled down to little above atmospheric temperature. Final cooling, necessary for the elimination of arsenic, takes place in the filters placed in the open air. These are small leaden chambers packed with selected coke. The gas, now perfectly clean, only requires heating to be ready for the chamber process. Heat exchange is effected by passing it back through the dust chamber in contact with the hot gases from the burners, after which it traverses a gas heater, where a temperature of 350°—400° C. is imparted to it by a coke fire. On a works where waste gas is available this operation is most conveniently and economically conducted by that agency, the need of a fireman being dispensed with. The hot gas now enters the potting oven, similar in most respects to established practice. From this point the course through the Glover chambers and Gay-Lussac towers is precisely similar to other chamber



plants, but the gas possesses the great advantage of being absolutely free from dust.

The manner in which greater efficiency of chamber space and many conveniences in working are effected will now be described. The towers are circular in section; those working on hot gas, the Glover and wash tower, are suitably lined with heat-resisting brick and packed with vitreous chequer work. The Gay-Lussacs are ring-packed. The two former towers are provided with spacious ponds to facilitate cleaning, and these ponds are water-jacketed in order to preserve the lead. Even distribution of acid on the towers is ensured by specially-designed distributors. The leadwork of the towers is suspended from the joists carrying the distribution floor. The lead of the chambers is similarly suspended from the principals of the roof, by means of flat steel strips, covered with lead, and fixed at right angles to the chamber sides. These have the effect of gills and materially assist in the radiation of heat from the chamber sides. These gills are slung from a system of joists in the structural steel work. It will be observed that by this method of free suspension the leadwork is permitted to adjust itself to temperature change without tearing at straps, etc. The design of the tops of the chambers is of importance. Segmental in section and supported by curved gills, the shape is conducive to intensive working. If we accept the theory that the gases in the chambers have a spiral motion on a horizontal axis, it is obvious that this shape of top leaves no room for stagnant pockets of gas, as would one of square section. With their great height of up to 65 ft., and the above methods of securing rapid heat dissipation, these chambers can work at 6 to 8 cub. ft. of space per pound of sulphur compared with 14 to 20 cub. ft. on the wooden system. Some well-managed works of the latter type have been able to get down to 10 to 11 cub. ft., but only at the cost of great wear and tear in leadwork.

A device which contributes much to the efficiency of chambers, is the use of water sprays instead of steam. The water is supplied to the sprays at a pressure of about 70 lb. and enters the chambers in the state of a fine mist. Apart from the cooling effect, much better regulation of the "drips" is possible.

Another very efficient type of chamber is circular in section, and is, in fact, a tower, but unpacked. Suspended in a similar manner, even lower chamber space can be obtained—about 4 to 5 cub. ft. The earliest attempts at reduction in chamber space consisted in devices to cause greater interaction of the gases by increased surface contact whilst retaining the original shape of the chambers. Lead curtains in the chambers were tried, but as the metal was subject to no cooling action, they soon collapsed. Glass partitions were also tried, without success. Lunge towers were the most successful, but never attained extensive use in this country.

The Opl system replaces the chambers entirely with towers. This seems to be the ideal process, as very low "chamber" space is claimed for it—2 cub. ft. In practice, however, it is evident that it is still in an experimental condition as there is very much pumping of acid round the towers, with consequent power consumption. Loss of nitre, when it sets in, occurs with great rapidity and unremitting scientific supervision is necessary.

Water-cooled chambers of conical shape are now being tried, with which a chamber space of 4 to 5 cub. ft. is claimed.

An important item on a sulphuric acid plant is the addition of nitre or nitric acid. Most English works use nitre applied by means of a "potting oven," which is a miniature nitric acid plant. The nitric acid evolved from regular small charges mingles with the hot gases entering the Glover tower. There is much to be said for

this system as regards its effect on the working of the chambers, but it has the drawback of being wasteful of acid (27—32% free acid in the nitre cake), and moreover if the charges are at frequent intervals (as they should be) it occupies practically one man's whole time each shift.

Continental practice favours the direct use of nitric acid. This is usually fed down the Glover tower in conjunction with the nitrous vitriol. In the author's opinion this system has very little to recommend it. Its one great advantage is that a "falling off" in the chambers can be more quickly remedied than by any other means, inasmuch as a large quantity of nitric acid can be almost instantaneously added just at the place where it is needed. That this is greatly in its favour, anyone familiar with intensive working will freely admit, as it is an accepted fact that the lower the chamber space the quicker is the nitre lost. This is but natural owing to the increased velocity of the gases, which becomes still greater with deficient nitre. Unless an excess of nitre is applied at once, the loss increases rapidly owing to the reaction working round into the back chambers and thus preventing re-oxidation. The effect of this soon shows itself in the strong nitrous acid coming from the first Gay-Lussac, the nitric oxide content of which rapidly falls. As this acid goes back to feed the Glover tower, it is obvious that on arrival there it will contain insufficient nitre to convert the sulphur dioxide in the gas, so that the addition of extra nitre must be kept up until the nitric oxide content of the first Gay-Lussac acid is again normal and the lower strength nitrous vitriol is worked off. Otherwise a vicious circle is set up and the plant goes from bad to worse. In cases of this description the nitric acid fed down the Glover tower is of great value, as it is possible to get sufficient in to convert all the sulphur dioxide, even supposing the acid from the first Gay-Lussac contained no nitric oxide. This is not usually possible in a potting oven (which is also much slower in effect) as in very big charges the nitre cake will have to come out of the pots "uncooked," i.e., containing uncomposed sodium nitrate.

On a well-managed plant, however, the above state of affairs is only of infrequent occurrence, and under ordinary circumstances the nitric acid system becomes an intolerable nuisance. Nobody likes handling carboys of anything, least of all nitric acid. These must be hoisted to the top of the Glover tower and emptied into the feeding pot or siphoned out. Both are objectionable, as the apparatus used must be of earthenware or glass, neither of which materials is safe in the hands of the ordinary workman. Breakages are liable to cause fires through the acid coming in contact with the wood flooring.

On a plant making 140 tons of B.O.V. per week, the consumption of acid is only about 1 gallon per hour. The adjustment of such a comparatively minute feed is no small matter of difficulty, and the very position of the apparatus (at the top of the tower) renders it liable to neglect. A further drawback is that the strength of the acid is liable to variation, whereas that of nitre is very consistent.

Another practice, which is not to be commended for obvious reasons, is that of feeding a strong aqueous solution of sodium nitrate down the Glover tower in case of a bad shortage. Sodium nitrate sprays are sometimes employed in a small first chamber where the acid is to be used for superphosphate—the sodium sulphate produced being of no consequence. When nitric acid was first employed it was usually fed into the chambers. The devices employed were various and need not be described here. The practice has been abandoned owing to its destructive action on the lead

and tendency to produce a high nitre content in the bottom acid.

In the author's experience the best method is a combination of the nitric acid and potting systems. The former is only used in cases of necessity, and the method of introducing it eliminates the drawbacks described in connection with feeding down the Glover tower.

In the first place a standard strength of nitrous vitriol is determined, which at a given feed, is known to be sufficient to meet the gas from the existing load on the plant. We will assume that this strength is 12 c.c. of nitric oxide per c.c. of acid. The quantity of nitre introduced at the potting oven is arranged to maintain this same figure in the strong nitrous vitriol from the first Gay-Lussac tower, which, of course, should have the same quantity of acid feed as the Glover tower. In the case of something occurring which causes the plant to "fall off," or, in other words, create a tendency for the reaction to work round to the back chambers, the steps taken to right things are these:—The amount of sulphur dioxide in the burner gases is temporarily reduced (for a few minutes only is enough as a rule). If the position seems to warrant it, an addition is made in the potting, but not to the extent of causing the nitre cake to be discharged uncooked. In the meantime tests taken on the Gay-Lussac acid will show a gradual decrease in nitric oxide, very often preceded by a sharp rise of short duration. This acid is allowed to accumulate in the bottom tank and made up to standard figure with nitric acid before sending up to feed the Glover tower. By this means the apparatus for elevating the acid to the Glover tower also elevates the nitric acid, and an acid of absolutely constant strength is fed down the tower. The reaction becoming normal again soon allows the strong nitrous vitriol to resume its ordinary strength, when the addition of nitric acid ceases.

By this method a minimum of acid is used; the handling of it is reduced owing to its only having to be lifted the height of the low level strong nitrous tank into which it is emptied from a suitable platform. Moreover the plant is rapidly "brought round" and a great saving in nitre consumption effected. The evil of too much nitre, which is so fatal to the lead of the chambers, and so liable to occur by the Glover feed method, is entirely avoided.

An outline of a new method of introducing nitric acid to the chambers has recently been published. Under the auspices of the Ministry of Munitions one or two large manufacturers have experimented with an ammonia oxidation plant for this purpose and good results are said to have been achieved. Further details will be awaited with interest by the industry.

The acid plant described is driven entirely by electricity. Decentralised driving has been found to be the best in practice, though more expensive to instal. Compressed air is supplied to the automatic eggs at 70–80 lb. pressure from a belt-driven compressor, which is fitted with an automatic cut-off device enabling the pressure to be maintained within a few pounds of that desired. The entire plant takes about 35 horse-power. At present prices the cost of such a plant is certainly not more than the cost of a wooden one of the same chamber capacity. Based upon output, however, it is about 40% cheaper owing to greater capacity per cubic foot of chamber space.

De-arsenication by this method costs about 2s. per ton of acid less than by the ferrous sulphide process, and much less if gas heating is available.

The chamber and Glover acids are practically water-white. It may be said without fear of contradiction that Glover acid made by this process is cleaner than that produced by any other plant using pyrites fines in mechanical burners. All the

dust and arsenic usually present in Glover acid is concentrated in the wash acid, which only amounts to 3–5% of the total output. The actual amount varies with the nature of the ore employed.

#### Concentration.

The concentration of sulphuric acid is a wide subject and full justice cannot be done to it in the scope of this paper. It would not be complete, however, without some mention of the broad lines along which development has taken place.

The older methods of concentration in glass and platinum vessels have now been practically abandoned. Concentration of chamber acid from about 120° Tw. (sp. gr. 1.6) is still effected in open lead pans on a large scale. Lead, if good quality, is not materially attacked by acid of under 150 Tw. (sp. gr. 1.75) and this method is very cheap where waste heat is available. Evaporation losses are extremely low.

The most rational, as well as the most economical, process of chamber acid concentration is in the Glover tower, whereby the weak acid assists the denitration of the nitrous vitriol and at the same time supplies part of the water necessary for the formation of sulphuric acid. Naturally there is no loss in this method. It is not possible always to work up all the chamber acid produced on a set in this way, neither is it possible to concentrate to high strengths. External plant must therefore be provided.

The cascade system is one of the most popular in use at present. By this method the weak acid is fed through a series of vessels of fused silica, or acid-resisting material of a metallic nature, of which there are several in the market. These basins are arranged in step formation whereby the acid flows from one to the other in its approach towards the hottest part of the flue and gradual concentration takes place. The lower portion of the cascade is covered in with blocks of lava stone or similar material and the acid vapours carried off under vacuum through a series of towers suitably packed, down which a stream of water is run, or more simply through a coke filter, which causes the condensation of acid fog. The fuel consumption on a plant of this description is about 25% of the weight of concentrated acid produced. This system is well adapted to gas firing, which can be very nicely adjusted and dispenses with the unavoidable disarrangement caused by the cleaning of the fire.

The upkeep of cascade plants is a heavy item, broken basins, etc., causing the acid to get into the brickwork of the flues. Distortion begins, and unless bad joints, etc., are made good at once, cold air is drawn in, with loss of efficiency. Unless the acid is very clean, iron is deposited in the basins and develops crusts, causing the acid to spurt. If the basins are of fused silica the crusts cause breakages; if of metallic alloy they are liable to burn out. It is of importance, therefore, that a clean acid be employed if upkeep costs are to be kept within reason. Bottomley gives details of a cascade plant of fused silica with beaker type preheaters (this J., 1917, 577).

The concentration of waste acids from nitration processes such as the manufacture of T.N.T. presents special problems owing to the presence of impurities. These impurities consist principally of iron, nitric and nitrous acids, together with considerable amounts of organic matter. One method of dealing with this acid is as follows: The feed acid descends a small earthenware tower packed with rings up which the hot vapours from final concentration are passed. The purpose of this tower is to preheat and denitrate the acid. The vapours pass on to the condensing system, whilst the acid flows to the first of a series of lead pans or preheaters where preliminary concentration up to about 140° Tw. takes place. Then follows



the usual cascade system where the concentration takes place up to 95%, after which the acid falls into a large cast iron pan placed over the furnace. The purpose of this pan is to bring the acid up to 97% strength and allow of easy cleaning of the deposited ferric sulphate which is thrown out of solution at this strength. The vapours given off from this pan are led to the preheating tower. Owing to the presence of nitric acid all the vapour pipes must be of earthenware.

The extreme discomfort to the operator of concentrating waste acid containing nitric acid is avoided if the acid be first denitrated by passing down an earthenware tower up which a current of slightly superheated steam at about 60 lb. pressure is blown.

The fifty-second annual report of the Chief Alkali Inspector for the year 1915 gives some interesting details with regard to cascade systems. Output per basin and details of condensers are analysed and tabulated.

A very successful plant which is specially suitable for the working up of large quantities (40—50 tons per day) of waste acid is the Gaillard tower. This apparatus is constructed of Volvic lava rings, and is about 40—50 feet high and 6—10 feet in diameter, according to output. The acid is fed down the tower (which is unpacked) in the form of a spray and meets the gases from a coke producer. The acid fumes are led away from the top of the tower into a smaller one where considerable quantities of weak acid are condensed and sent back with the feed acid. Final condensation takes place in a coke filter. The gases are drawn through the system by a fan placed after the coke filter. Upkeep in this type of plant is costly, but the output is high per unit of ground space occupied.

E. H. Armstrong (J. Ind. Eng. Chem., 1917, 9, 386—388; this J., 1917, 501) gives details of a tower method of concentration using quartz as packing and an oil flame for heat. The disadvantage seems to lie in the disintegration of the quartz; an instance of 50% being removed is quoted.

The type of concentrator usually supplied with the chamber plant described is a modification of the Kessler system. It consists of a coke producer with a capacity of at least 24 hours' working at maximum output. The hot air from this producer enters a "saturex" or saturator, travelling in a horizontal direction against the acid flow. The gas is deflected on to the surface of the acid by barriers placed at right angles to its path. The height of acid can be controlled by a weir placed in the outlet spout to the cooler. The gas, laden with acid vapour, after leaving the saturator, passes upwards in a zig-zag course through six or eight plateaux down which the acid is fed. This recuperation preheats the feed acid and condenses the acid mist from the gas. The plateaux are provided with barriers to facilitate contact between acid and gas in a similar manner to the saturator. The gas leaves the recuperator at a temperature of about 120° C., traverses a coke condenser, where the remainder of the acid mist is absorbed, and then through a fan to the atmosphere.

The principal difference between this apparatus and the Kessler is in the plateaux. In the Kessler the recuperator is more like a fractionating column, the contact between gas and acid being made by serrated porcelain hoods. These have

the disadvantage of becoming blocked with iron salts, necessitating frequent cleaning if the acid is not very free from this impurity. The open type of plateaux employed in the plant described obviates this difficulty to a great extent.

The saturator and plateaux are constructed of vitrified earthenware blocks, the joints being made with special cement. The whole is covered with stout lead and presents a neat appearance. The coke consumption is about 19%. Care should be exercised that the volatile matter is low or the acid is liable to be discoloured.

#### DISCUSSION.

The CHAIRMAN remarked that it used to be said before the war that the production of sulphuric acid in a country might be taken as an indication of its stage of civilisation. That remark would be of sinister significance nowadays; but there could be no doubt of the great and increasing importance of the manufacture. He asked the author whether the type of burner described (the Moritz) could be modified so as to roast a form of sulphide in regard to which external heating was necessary.

Mr. H. ROYAL-DAWSON said that the point put by Prof. Cobb had also occurred to him with reference to the burning of zinc sulphide, and of refractory ores in general, and he would like to know the type of burner used for these. The largest amount of acid came from pyrites of course, but with regard to other materials he only knew of one successful burner—he thought it was the Harris furnace—which could be used for zinc sulphide. The problem had been studied on the Continent, and he thought it was for this country to take the matter up, by zinc manufacturers treating their ores for recovering the sulphur as sulphuric acid in ore roasting.

Mr. G. F. HUDSON said that in concentration the difficulty was not to work a system with fair efficiency when it was new; they nearly all worked very well for a time; but then either the material of construction began to fail, or the heat of gas and leakages began to break the system down, often after only two or three years. Had the plant described by the author of the paper had a sufficiently long trial to compare in that respect with cascade concentrators, Gaillard towers, and other systems? The difficulties began when the materials had become saturated with acid.

Mr. Moss replied that the matter mentioned by Prof. Cobb had been receiving a good deal of attention of late, but there were very serious difficulties to be contended with. The burning of low-grade zinc ores, with perhaps 25 to 30% of sulphur, required extraneous heating. The necessity for the employment of artificial heat complicated the matter from the chemical engineering standpoint very considerably. As to the short life of concentration plants, he agreed in general with Mr. Hudson, but the type he had described had an exceptionally long life. He knew one Kessler type plant which had been working for something like ten years, and he thought it was practically as good as ever. Lava stone was liable to disintegration, but the material he had spoken of was not so liable, provided it could be obtained without flaw. Gaillard towers also had direct contact with the acid and gas, and they suffered from decrepitation, but he thought this type had on the whole proved to be satisfactory.

## New York Section.

*Meeting held at Chemists' Club on Friday, January 18th, 1918.*

MR. JEROME ALEXANDER IN THE CHAIR.

### PRESENTATION OF PERKIN MEDAL TO DR. A. J. ROSSI.

After some introductory remarks by the Chairman.

Mr. F. A. J. FITZGERALD gave an account of Dr. Rossi and his work, in which he mentioned that Rossi was educated in France, and came to New York in 1859. For some years he was engaged in engineering and surveying work, and was subsequently employed as chemist to the Boonton Iron Works. It was here that Rossi first became interested in the subject of titanium, the ores used in the blast-furnaces being magnetites containing from 1 to 2.5% or more of titanic oxide, and he made a close study of the problems connected with the smelting of titaniferous iron ores. This work, however, bore no fruit at the time, and it was not until several years after he had left metallurgical practice that owing to a lawsuit in connection with the Boonton Works, he became recognised as an expert in that particular field.

In 1890 he was consulted in regard to the smelting of the titaniferous ores of the Adirondacks, and since that date he has devoted most of his time to titanium. During his investigation of these ore deposits he found that in two small blast-furnaces which had been run between 1840 and 1858, excellent iron had been produced by adding large quantities of lime and silica to the charge, producing a silico-titanate slag—a practice adopted successfully also at Stockton-on-Tees with ores containing up to 35%  $\text{TiO}_2$ . The objection to this process was the large quantity of slag produced, and Rossi made a series of experiments with the object of replacing silica by titanic oxide in normal blast-furnace slags. As a result of these experiments small blast furnaces were built to treat the ores in question, and the success of these paved the way for the commercial utilisation of these ores.

Rossi's work on the smelting of titaniferous iron ores had convinced him that titanium had a beneficial effect on iron and this led him to the belief that an alloy of iron and titanium would be of value in the manufacture of iron or steel. Experiments confirmed these views, and in 1899 electric furnaces were built at Niagara Falls, in which considerable quantities of ferro-titanium were manufactured and used for testing purposes. These tests bore out all his anticipations, but it was many years before he succeeded in convincing iron and steel makers of the fact, and even now the scepticism has not been entirely abolished.

In the early days of the use of titanium it was chiefly employed in rail steel, but recently it has been applied in a number of other directions. An instance of the beneficial effects of titanium is found in the results of 155 heats representing the production of 9000 tons of rail steel; when no titanium was used only 36% of the steel came within the specified limits, while with increasing amounts of titanium the following percentages passed:—0.053% Ti, 43%; 0.077% Ti, 81%; 0.10% Ti, 100%.

After a short congratulatory address, Dr. W. H. NICHOLS presented the Perkin Medal to Dr. Rossi.

Dr. Rossi, after expressing his appreciation of the honour conferred upon him, added some further details of his work, as follows:—

My experience at the Boonton blast-furnaces convinced me that the presence of titanium dioxide to an average of 1.50%  $\text{TiO}_2$  in the ores which we were smelting, and of some 2 to 2.50% of  $\text{TiO}_2$  in the slags, had had no effect on the working of the furnaces, or on the behaviour of the slags as to fluidity and fusibility, so much so that I even ignored its presence, considering it as so much additional silica in the ores. In 1894, at the New York Car Wheel Works, Buffalo, in a small furnace of a capacity of three to four tons a day, I melted, without admixture, titaniferous iron ores from the Adirondacks, containing 15 to 18%  $\text{TiO}_2$  and 55 to 56% metallic iron, and the slags as run from the furnace contained 25–30%  $\text{TiO}_2$ , and some 15 to 18% silica, with lime, alumina, and magnesia as bases, magnesia having been recognised by me as an essential constituent of the slags to ensure the best running. Within the last two years, Mr. Bachman has smelted similar titaniferous iron ores in one of the large blast furnaces at Port Henry, making some 15,000 tons of a superior pig iron with an economy of fuel per ton of pig metal smelted.

The titaniferous ores of the Adirondacks are magnetic, associated with ilmenite ( $\text{FeTiO}_3$ ). They are, like almost all titaniferous ores, low in phosphorus and sulphur, some as low as 0.017% P and 0.02% S, with 55 to 56% metallic iron and 1.50 to 2%  $\text{SiO}_2$ . The iron smelted from them is an ideal open-hearth steel. Tests made in many foundries of such pig metal added to ordinary pig iron in certain proportions have shown an increase of 29½% in strength over cast iron not treated and an increase of 4% over cast iron treated with nickel.

However much value titaniferous ores may have, alone or in mixture with other ores, as blast-furnace stock, their use for making alloys of iron or copper with titanium for the treatment of cast iron, steel, copper, or copper alloys has opened a new and more important field of applications in metallurgy.

In about 1900 I started making ferrotitanium at Niagara Falls in an electric arc furnace; the furnace itself, made of a masonry of graphitic materials, formed the cathode, and a carbon electrode, movable vertically in the cavity of the furnace, constituted the anode. A mixture of the titaniferous material and the carbon required for the reduction of both the titanium and iron oxides properly comminuted, was then charged gradually into the furnace as the current was turned on. When the relative proportions of the  $\text{TiO}_2$  and the iron oxide in the titaniferous material were such as to produce an alloy higher in titanium than desired, some scrap iron was charged with the mixture to dilute the alloy, so to speak. I made in this manner alloys containing from 10 to 25% Ti or more, but practice in steel and other works has shown that an alloy containing some 15% Ti is best suited for most purposes.

Whenever the presence of carbon in the ferro-carbon-titanium was considered objectionable for special applications, I reduced the titaniferous materials by the aluminium bath process. Instead of using a mixture of finely powdered aluminium and titaniferous materials as in the Goldschmidt's Thermit process, the aluminium I used for the reduction of the oxides was melted in the electric furnace and the titaniferous materials (omitting the carbon) charged directly into the bath of aluminium. In such cases I used preferably  $\text{TiO}_2$  as rutile, in order to avoid using the aluminium which would have been required for the reduction of the iron oxide of the ore, charging scrap iron with the rutile to dilute the alloy to the content of titanium desired. In this manner I made ferrotitanium containing only from 0.18 to 0.50% C, and by reducing the amount of scrap iron added, I was able to make ferrotitanium containing as much



as 80 to 85% Ti. Titanium is very hard and brittle so that as such it has no particular use. The alloys of titanium containing 75 to 85% Ti, or even less, are white like silver and scratch glass and even quartz deeply.

The aluminium bath process is of a more general application. If into the aluminium bath a tungstic ore containing iron oxide is charged, a ferro-tungsten is obtained free from carbon. I have made thus 85% tungsten alloy. By using manganese ores I was able to make in the electric furnace 85 to 88% ferromanganese free from carbon, and by using chromic iron, a ferrochrome containing about 80% Cr and free from carbon.

By adding to a bath of steel or to cast iron a ferroc carbon-titanium containing some 15% Ti the bath of metal is cleansed from dissolved or occluded gases such as oxygen and nitrogen and also of such oxides of iron generally present in steel, especially in Bessemer steel made by the pneumatic process. When air is blown through the molten pig iron, the titanium of the alloy combines with the oxygen to form  $TiO_2$ , with the nitrogen to form titanium nitride, and the iron oxide is reduced to iron by the titanium and the carbon; the titanium then combines with the oxygen to form  $TiO_2$ , and the slag, carrying with it the titanium dioxide and nitride, rises to the top of the ingot or ladle: the  $TiO_2$  renders the slag more fluid and fusible. A slag collected at the top of an ingot and containing 6.5%  $TiO_2$  had a melting point of 1290° C. By adding enough  $TiO_2$  to such a slag as to have in the slag some 13%  $TiO_2$ , its melting point was lowered to 1190° C.

For the deoxidation of copper and its alloys such as bronzes, brass, manganese or aluminium-bronzes I have made a copper-titanium containing any percentage of titanium, though 10 to 15% appears most suitable. The titanium of such an alloy acts, as in steel, as a scavenger of the copper bath. I have also made aluminium-titanium containing 45% Ti for seasoning aluminium-bronzes. These alloys are made in the same manner as ferroc carbon-titanium or ferro-titanium in the electric furnace. For copper-titanium,  $TiO_2$  and aluminium are charged into the furnace in a bath of copper. For aluminium-titanium, the  $TiO_2$  is charged directly into the bath of aluminium. The reaction of aluminium on oxides being exothermic, much less current is required for the reduction proper.

I have also carried out experiments in smelting titaniferous ores in admixture with phosphoric ores, even to the extent of obtaining a pig iron high in phosphorus. I used phosphoric ores containing as much as 1.50% to 2%  $P_2O_5$ , even adding apatite to the mixture so as to obtain a pig iron as high in phosphorus as possible. Contrary to what might have been expected, the pig metal containing 0.40% Ti and from 2.50 to 3.50% P had the strength and the rating of fair No. 1 or No. 2 pig iron. This suggested treating phosphoric pig metal, generally weak and close grained, with ferrotitanium so as to incorporate a certain amount of titanium in it. The results were very encouraging.

If a ferrotitanium, practically free from carbon, made by my aluminium bath method, preferably as high in titanium and low in iron as possible, is heated with hydrochloric acid, in which it dissolves readily, a fine violet solution of a ferrous-titanium chloride is obtained. The double chloride contained about 40 to 50%  $TiCl_3$ , the balance being ferrous chloride. I have been able to bleach successfully and without injury cotton, silk, and wool by digesting them in this diluted violet solution at boiling temperature. During the operation, especially as the temperature approached the boiling point,  $TiO_2$  was precipitated, thus showing that the chlorine must have been set free and combined with the ferrous chloride to form

ferric chloride, the chlorine generated acting in the nascent state without even appearing as free chlorine in the solution.

The fact that titanic oxide was precipitated from one of its compounds by these organic matters suggested to me the possibilities of precipitating this compound from others of its compounds by vegetable or animal organic matters, and the results were very remarkable. If to a solution of titanic sulphate,  $Ti(SO_4)_2$ , is added an aqueous extract of vegetable or animal substances (leaves, green leaves, sawdust, tannin, wood pulp, wood-pulp liquor, horse chestnuts, beans, etc.) and the liquid boiled for a short time,  $TiO_2$  is precipitated and when calcined is obtained as a soft, smooth, flour-like, pure white powder requiring no mechanical pulverisation. If the titanic sulphate solution is precipitated by ammonia or caustic soda and boiled, and the  $TiO_2$  calcined, the product is granular and buff-coloured.

This method of preparing the smooth, white oxide finds a direct application for the production of a white titanic pigment, a product which our company is to manufacture on a large industrial scale. Calcined titanic oxide possesses the property of covering when used as paint material alone or in mixture with other paint pigments. Its superiority on this score, when prepared with proper oils, over white lead or zinc white is remarkable, and in addition  $TiO_2$  pigment is not attacked by sulphurous gases and is stable under climatic and atmospheric influences.

Titanic oxide, obtained as a gelatinous titanic acid,  $TiO_2 \cdot 2H_2O$ , and dried at 100° C., has the property of fixing colouring matters, like that of alumina. It can be prepared by known methods but the one I have studied is much more economical, as I can obtain it from a waste product of some of our manufactures. In that state it dissolves readily in oxalic acid, yielding a titanium oxalate which can be used in the same manner and for the same purposes as potassium titanium oxalate, which is much more difficult and costly to make.

Some of my work has been in an entirely different direction. I have used for purposes of artificial production of cold, instead of liquefied gases such as sulphur dioxide, carbon dioxide, or ammonia, a binary liquid composed of one moderately volatile liquid containing in solution the vapours of a much more volatile liquid. In ice-making and refrigeration the volatile liquids are charged in a special vessel, the refrigerator being immersed in a bath of uncongealable liquid so-called, such as a strong solution of sodium or magnesium chloride, or glycerin; or alternatively the liquid is caused to circulate in a tank in which cans containing the water to be frozen are immersed or circulated through pipes in the cellars of the brewery or store-room. By relieving the pressure of the volatile liquid on itself, at ordinary temperature, by means of a double-acting pump run by any power (steam engine or other), the liquid boils and by its latent heat of evaporation cools the refrigerator contents. The vapours entering the pump are compressed in a condenser cooled by circulating water, liquefied again under this pressure and cooling, and combined again, reforming the original volatile liquid, which is returned by proper devices to the refrigerator, rendering the operation continuous.

One of the binary liquids I used for machines erected in breweries and cold storages was ether containing sulphur dioxide in solution. Ether will absorb its own weight of  $SO_2$ , showing but a very few pounds of pressure (5 to 6) per sq. in. at 90° F. By using such binary liquid instead of sulphur dioxide, for instance, when the mixed vapours enter the compressor to be compressed and discharged into the condenser, ether liquefies first, being cooled by the circulation of water



around the condenser, absorbing anew the sulphur dioxide and causing the pressure in the compressor and condenser to reach the figure it would have reached had sulphur dioxide alone been used, thus saving on the mechanical power required.

The other liquid I used was sulphur dioxide, which can absorb some 5 to 10% of its weight of carbon dioxide. In this case, the same advantages as with the other binary are secured but to a much greater extent. Carbon dioxide boils at about  $-100^{\circ}$  F., and the pressures required to liquefy its vapours are considerable.

*Meeting held at Chemists' Club on Friday,  
October 19th, 1917.*

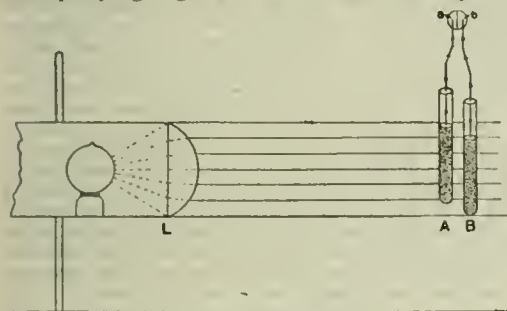
MR. JEROME ALEXANDER IN THE CHAIR.

### TECHNICAL APPLICATIONS OF NEPHELOMETRY.

BY PHILIP ADOLPH KOBÉ.

The method known as nephelometry is based upon the measurement of the brightness of the light reflected by a cloud—in other words, by the particles in suspension—very much as in an ultramicroscope. The intensity of the light reflected is a function of the quantity of suspended particles, when other conditions are constant.

The principle of the nephelometer can best be shown by a diagrammatic sketch, shown in the accompanying figure. Let A and B represent



tubes containing a precipitate in the form of a suspension, and L represent a strong light which throws its uniform beam on tubes A and B at right angles; then *a* and *b* will be the light in the eye-piece due to the reflections from the two suspensions. If tube A, for example, contained distilled water and the instrument were perfect, no light at *a* would be visible. As soon as the smallest amount of suspension is produced in the tube A, light is obtained in *a* in approximate proportion to the amount of suspended matter. This light *a* is never measured absolutely, but is always matched at *b*, which is that reflected by the precipitate from a standard solution—a known amount of the substance to be determined dissolved in a known volume.

The matching of the two lights could be done by changing the standard solution step by step until it would be exactly similar to the unknown. In practice this would be tedious, and therefore instruments were designed to eliminate this in whole or in part.

Work with the nephelometer should always be conducted in the dark. Probably the greatest source of error connected with nephelometry or colorimetry, however, is practically an instrumental one. Most diaphragms or openings at the top of the eye-pieces, even in the best instruments, are too large, but by putting a small diaphragm of the size of a pin-hole over the optical centre, equal illumination of both sides of the field can be obtained.

The chief requisite for making nephelometric

clouds or colloidal suspensions, and for keeping them as such for definite time, is that the substance be in a dilute solution, usually not stronger than 100 mgrms. per litre. Clouds produced by one part in 500,000 of liquid may be determined quantitatively. In some cases one part in 2,000,000 can be easily determined.

If the precipitate to be examined is highly coloured and remains in suspension, it is best determined colorimetrically; if slightly coloured, it is best determined nephelometrically. The precipitate must be colloidal, in the form of a suspension. Certain solutions of protective colloids can be used, such as egg albumin and soluble starch, which cause crystalloids and partly colloidal precipitates to remain in suspension long enough for the application of this method.

The number of possible applications of nephelometry is too large for consideration here, and therefore a few representative estimations have been chosen, viz., three inorganic and three organic determinations; the estimation respectively of ammonia, phosphorus, calcium, acetone, oils and fats, and, finally, proteins.

*Estimation of ammonia.* S. S. Graves has developed a sensitive nephelometric reagent for ammonia: 80 grms. of sodium chloride is dissolved in 130 c.c. of water and 100 c.c. of a saturated solution of mercuric chloride, and 70 c.c. of a saturated solution of lithium carbonate (1.0%) is added and the mixture filtered. By means of this reagent one part of ammonia can be detected in 160 million parts of water.

For every 10 c.c. of ammonium sulphate solution 15 c.c. of a 0.003% solution of starch will keep the suspension from agglutinating and settling for about an hour. The standard solution contains 9.43 mgrms. ammonium sulphate, which is equivalent to 2.43 mgrms. ammonia, or 2.00 mgrms. nitrogen per litre. When precipitated with 5 c.c. of reagent the resulting suspension is put into both cups of the instrument.

If the left cup is placed at any convenient height and the other cup moved up or down until the light in the eye piece, coming from both tubes, is equal, the height on the right is rarely, if ever, equal to the height set on the left side. The reading on the right side will be denoted as the standard reading. If in place of the standard on the right other solutions of ammonium sulphate of different strength are used a series of corresponding readings is obtained, and these are plotted in the form of a curve.

When the instrument changes so that re-standardisation is necessary, a nephelometric formula may be used, but it is simpler to adjust the height of the solution on the left side (the one used as a tare) so that the original reading for the standard is obtained, and therefore the original curve is applicable.

In making estimations of nitrogen, 0.1 gm. of the substance to be examined is heated in a Kjeldahl flask with 20 c.c. of concentrated sulphuric acid, and a little mercury or mercuric oxide as catalyst, diluted and made up to 500 c.c. To 5 c.c. of this solution is added 5 c.c. of N/1 sodium hydroxide and then, drop by drop, the same solution until it is neutral to litmus paper. The solution is then diluted to 200 c.c. with ammonia-free water, and a 10 c.c. portion is treated with 15 c.c. of 0.003% starch solution and precipitated with Graves' reagent (5 c.c.) and matched against 10 c.c. of standard solution (containing 2.0 mgrms. of nitrogen per litre) similarly treated. From the reading and the curve we can find out at once the percentage of nitrogen.

*Phosphorus.* The reagent developed by Pouget and Chouchak\* (a nitric acid solution of strychnine and molybdic acid) produces a very sensitive



reaction with phosphates, being capable of detecting one part of phosphorus in 20 million parts of water. As the precipitate is slightly yellow and remains in suspension for a long time, the authors have recommended it as a colorimetric reagent, but really the estimations were turbidimetric, i.e., they measured the absorbed light of the suspension.

On studying this reaction, Egerer and Kober found that it was not constant or quantitative, and that the reagent gradually became yellow and deteriorated, probably because of the action of nitric acid. On substituting hydrochloric for nitric acid, the solution not only remained practically colourless for an indefinite time, but was stable and gave quantitative and constant results.

To prepare the modified solution, 150 grms. of sodium molybdate is dissolved in 250 c.c. of water and 100 c.c. of hydrochloric acid (1:1) and 150 c.c. of strychnine sulphate solution (2.0%) added slowly with shaking. Protective action in addition to any produced by the strychnine does not seem to be necessary. The reagent is so sensitive that ordinary filter paper must not be used for filtering the reagent. A solution of potassium phosphate containing 0.003 mgrm. per litre shows the reaction very plainly, i.e., one part in 333 million.

For the determination of phosphorus in castiron, 2 grms. of borings is dissolved in 100 c.c. of 1:3 nitric acid by boiling. After cooling under the tap the solution is made up to 100 c.c. and 5 c.c. boiled for 2 minutes with 5 c.c. of concentrated sulphuric acid. This mixture is then diluted to 100 c.c. and filtered through a dry filter paper which has been washed with acid, and 25 c.c. is again diluted to 100 c.c. 10 c.c. is now treated with 35 c.c. of N/2 hydrochloric and precipitated with 5 c.c. of reagent. On matching in the instrument with a standard containing 5 mgrms. of  $\text{KH}_2\text{PO}_4$ , per litre the percentage of phosphorus can be obtained from a curve made similarly to the one described for ammonia.

**Calcium.** Lyman's nephelometric reagent for calcium is prepared by boiling 4 grms. of stearic acid and 0.5 c.c. of oleic acid with 400 c.c. of 95% alcohol, boiling, adding 20 grms. of ammonium carbonate in 100 c.c. of hot water, boiling, cooling, and adding 400 c.c. of 95% alcohol, 100 c.c. of water, 2 c.c. ammonia solution (sp. gr. 0.90), and finally filtering. A solution of calcium oxalate in nitric acid containing 0.2 mgrm. of calcium per litre (one part in 5 million) gives a decided reaction with this reagent.

For the estimation of calcium in milk, 10 c.c. is diluted to 100 c.c. with distilled water. Five c.c. of this mixture is then treated with 15 c.c. of 6.5% trichloroacetic acid, which precipitates the proteins, and filtered. The calcium is now precipitated as oxalate by McCrudden's method,\* redissolved, and the calcium determined nephelometrically after precipitation as soap. 20 c.c. of standard calcium oxalate solution (dissolved in nitric acid) containing 0.4 mgrm. of Ca is poured into 50 c.c. of Lyman's reagent and gently shaken. This cloud is used for matching an unknown treated in a similar manner. Here, as in the other estimations, only a few circumventions are necessary to eliminate interfering substances. In this precipitation oleic acid acts as a protective colloid; a small amount delays the agglutination for hours.

**Acetone.** Marriott has applied the extremely sensitive Scott-Wilson reagent for the nephelometric estimation of acetone. The acetone in each case may be distilled from the original solution into the reagent or may be aerated† at room temperature into sodium bisphosphate solution, and

then estimated nephelometrically. The composition of the reagent is given as: 10 grms. of mercuric cyanide, 180 grms. of sodium hydroxide, 1200 c.c. of water, to which 400 c.c. of 0.70% silver nitrate solution is slowly added and the mixture filtered. This reagent is sensitive to 1 part of acetone in 100 million. The standard and unknown solutions are precipitated by distilling into 50 c.c. of water and 15 c.c. of Marriott's reagent, 0.5 mgrm. of acetone, and finally making the solution or suspension up to 100 c.c. Standard solution if made with N/4 sulphuric acid so that it contains 0.5 mgrm. of acetone in 10 c.c. will keep for a few weeks at least. No additional protective colloid is added, as the organic nature of the complex is sufficient protection.

**Fats and oils.** Bloor has devised a nephelometric method in which the fat or oil is extracted with an alcohol-ether (3:1) mixture and then poured into water, when the fat or the oil separates in fine globules or suspensions. As little as 0.5 mgrm. of fat can easily be determined quantitatively, and a marked cloud is produced by 1 part in a million.

The usual standard and unknown are precipitated by running 5 c.c. of alcohol-ether solution containing 2.0 mgrms. of fat or oleic acid into 100 c.c. of water and after adding 10 c.c. of 1:4 hydrochloric acid and gently stirring or shaking and allowing to stand for 5 minutes, are read in the nephelometer.

Murlin and Riche have modified this method by pouring the fat solution into 0.05% gelatin solution which delays the coalescence of the fat globules. Woodman Gookin and Heath\* have worked out a similar method for essential oils. The solvent and extracting medium for essential oils is alcohol alone, and 5 c.c. of the standard solution—containing 100 mgrms. in 100 c.c. of 95% (redistilled) alcohol—is precipitated by pouring into 25 c.c. of water, or better 25 c.c. of hydrochloric acid.

By adding acid as used in the Bloor method, I found that one could use a much weaker standard than the authors recommended and still get suitable nephelometric clouds. They were able to estimate the oils of roses, peppermint, anise, and nutmeg nephelometrically with ease and accuracy.

**Proteins.** Nephelometry can be very well applied to protein estimations, a solution of sulphosalicylic acid being the reagent used (Kober, J. Amer. Chem. Soc., 1913, 35, 290). A solution containing 1.0 mgrm. of protein per litre gives a marked test.

In milk the fat is first removed by adding to the diluted milk (5 c.c. of milk in about 200 c.c. of water) 10 c.c. of N/10 sodium hydroxide, making up to 250 c.c. and shaking with ether. (For further details see J. Amer. Chem. Soc., 1913, 35, 1589).

## Sydney Section.

Meeting held at Sydney, on Wednesday, September 19th, 1917.

### TANNING IN ACID LIQUORS, WITH COMPARATIVE TESTS.

BY F. A. COOMBS AND W. H. MCGLYNN.

These experiments were undertaken in the hope of throwing some light on the value of acid in tan liquors. The experimental process, as related to hide-sections for comparative results, has been described previously (this J., 1917, 188 seq.). It will be noted that two experiments were made with acetic and one with sulphuric acid. Each experiment includes two tests and each test consists of two pairs suitable for com-

\* J. Biol. Chem., 1911, 10, 187.

† Folin, J. Biol. Chem., 1914, 18, 263.

\* J. Ind. Eng. Chem., 1916, 8, 128.

parative purposes. All the tan-liquors were prepared from wattle bark and a normal and acid tannage was used for each experiment.

We have endeavoured by a series of comparative tests to show the difference between the results obtained with normal wattle liquors and similar liquors with the addition of certain amounts of acetic acid. The hide sections were prepared for the tan-liquors in the way already described (*loc. cit.*). For the acid tannage in the first two experiments, the natural acidity of the wattle liquors was found by the lime-water test and was then made up to the required concentrations by addition of acetic acid. The difference between the acidity of the liquors in the normal and acid tannages will show the amount of acetic acid used for the latter.

The influence of acid on the quality of leather during the process of tanning varies to a considerable extent according to the acid used and its concentration at various stages. Generally it may be said that tanners use acid to plump or swell the hide, thereby obtaining a stouter and consequently a more valuable leather. Our problem was one of determining its influence on the quality of heavy leather.

Our experience teaches us that all skins in the pelt state respond quickly to the plumping action of dilute solutions of various acids, but they do not respond to this action after they have been tanned. Sheep pelts are tanned in weak solutions and fix the minimum amount of tannin, but the resulting leather successfully resists the plumping action of acids. A neutral pelt is soft and flabby, differing from the acid pelt, which is firmer and stouter. The physical difference between the acid and neutral pelt will be responsible for a difference between the qualities of their respective leathers, providing they are fixed in the condition described above and that tannin is the fixing agent.

It is generally recognised that a firm and stout leather is a desirable result in the production of sole leather and therefore within certain limits the best results from a monetary standpoint will be obtained by fixing the pelt in the acid condition. A soft, pliable leather will be the result when a neutral pelt is tanned in liquors containing the

#### *Acetic acid experiment. No. 1.*

##### *First test.*

Sections 2 and 5.			Sections 1 and 6.	
Normal wattle bark liquors.			Acid wattle bark liquors.	
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.
3	8°	2.3	8°	10
5	12°	2.8	12°	12
6	17°	3.1	17°	14
8	24°	5.1	24°	16
12	33°	7.2	33°	18
19	46°	9.5	46°	20
21	60°	9.9	60°	22

##### *Second test.*

Sections 4 and 7.		Sections 3 and 8.		
Normal wattle bark liquors.		Acid wattle bark liquors.		
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.
2	8°	3.1	8°	10
4	12°	4.3	12°	12
6	17°	5.3	17°	14
8	23°	7.1	23°	16
11	32°	9.0	32°	18
19	45°	7.7	45°	20
21	57°	9.0	57°	22

minimum amount of acid; but when a highly plumped acid pelt is placed in acid liquors capable of keeping the pelt in its swollen condition, a harsh and brittle leather is produced which, in extreme cases, will break like a biscuit. The wear by friction will always be greater on a brittle than on a pliable leather and the most prominent factor in this physical difference appears, to be one of elasticity.

For the first experiment a pelt which had been delimed on the surface with lactic acid was used. This pelt was placed in the first tan liquor containing acetic acid and the tannin penetrated a certain distance into the hide, fixing a portion of the fibres and producing a normal leather. At this stage about one-fifth is tanned and the other four-fifths of the hide has fixed a certain amount of acid. As the pelt moves forward through the various tan-liquors their acidity is gradually increased and the amount of acid fixed by the untanned portion of the hide also increased until the tannin has penetrated through the hide. Under normal Australian conditions the tannin takes from three to four weeks to penetrate into the centre of the hide and therefore all the plumping must be done before the end of the fourth week. The leather tanned in the acetic acid tan liquors (first experiment) had a dark streak of brittle fibre in the centre of the hide and this brittle portion would represent that part which was not tanned after the leather had been in the tan liquors for two weeks. At this stage we apparently had an acid concentration capable of swelling the untanned portion of the hide beyond that point at which harsh leather is produced, providing it is fixed in this abnormal condition.

#### *Acetic acid experiment. No. 2.*

##### *First test.*

Sections 2 and 5.		Sections 1 and 6.		
Normal wattle bark liquors.		Acid wattle bark liquors.		
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.
2	8°	3.2	8°	6.0
4	12°	4.3	12°	8.0
6	17°	4.3	17°	10.0
9	23°	6.1	23°	12.0
13	32°	6.2	32°	18.0
21	45°	0.4	45°	20.0
22	58°	10.2	58°	22.0

##### *Second test.*

Sections 4 and 7.		Sections 3 and 8.		
Normal wattle bark liquors.		Acid wattle bark liquors.		
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.
2	8°	3.5	8°	6.0
4	12°	4.0	12°	8.0
6	17°	5.0	17°	10.0
9	23°	6.7	23°	12.0
12	32°	8.8	32°	18.0
21	45°	9.0	45°	20.0
22	58°	9.8	58°	22.0

In dilute acid solutions the protein molecules of the hide combine with the acid to form a highly ionisable salt, and under certain conditions the concentration of diffusible ions in the hide is greater than the ionic concentration in the outer solution, which leaves the osmotic forces in the two phases in an unbalanced state and, as a result, the external solution is absorbed and the hide begins to swell. These forces are described, by



Procter and Wilson, for gelatin as an outward pull where the increase in volume is directly proportional to the pull. Now let the total elongation of the hide fibres due to any outward pull be represented by a straight line A B. If the soft flaccid neutral pelt be placed at A and the firm stout acid pelt at B, we will have a scale for the total elongation between the neutral pelt at zero and the acid pelt at the elastic limit. Suppose we now take a pelt swollen to 9° and place it in a tan liquor with the required acid concentration to keep it at that figure, then tannin would fix the pelt and give it a permanent set at 9° elongation.

When these sections were placed in tan-liquors the outer portions of the hide received a permanent set at a low figure, but as the leather was moved forward into liquors with higher acid concentrations, the figure for the permanent set would gradually increase. In this case it would appear as if the permanent set for the inner portion of the hide were too near the elastic limit for the production of good leather. Working on this theory we reduced the amount of acid during the early stages of the tanning process as shown in the second experiment, and the resulting leather was free from this fault, which was constant for all the acid leather from the first experiment.

Procter states that after the swelling has reached its definite value, it is unchanged at a constant temperature by further lapse of time, providing that no change takes place in the acid concentration of the outer solution. Thus the total elongation for a swollen pelt is directly related to this acid concentration, and if it is desirable to have a permanent set at any particular point on the scale for elongation, then the correct acid concentration must be maintained in the outer solution.

We also give results for leather swollen with sulphuric acid. The sections were treated in the usual manner before they reached the tanning process and then one-half was placed in a sulphuric acid solution for plumping purposes. It is not desirable to give a permanent set to the grain or superficial surface of the corium at a high elongation because this portion of the hide is the first to crack if the process tends to a brittle leather; therefore it is the usual custom to allow the tannin to penetrate the grain of the normal hide and then place it in the sulphuric acid solution.

Several experiments were carried out with pelt suspended in acid solutions of varied concentrations, and we finally decided to use an N/10 sulphuric acid solution for the first test in this experiment. The pelt was placed in a tan liquor 8° barkometer for 24 hours, at the end of which time the grain was permanently set. The leather then went into N/10 and N/15 sulphuric acid solution for first and second tests respectively, and was left overnight for a period of 17 hours. The remaining tanning process was the same for these sections as for the normal process. The sections were very firm when they came from the sulphuric acid solution, and the increase in weight was 1% on the lime-swollen pelt. They did not receive any lactic acid. The penetration of the tannin proceeded less quickly with the sulphuric acid than with the normal leathers and, while the acid process gave the stouter leather, or greater dimensions as represented by a straight line piercing the leather at right angles to the grain, it appeared to be the cause of a decrease in area. Thus any increase in this direction would not be directly proportional to the increase in volume.

When the partially tanned pelt was removed from the sulphuric acid solution and placed in the tan-liquors no attempt was made to keep up the acid concentration in the outer solution. The unstable collagen-sulphuric acid combination is

hydrolysed and the acid diffuses into the outer solution, where a portion displaces weak acids from those salts common to tan liquors. Under these conditions most of the sulphuric acid is removed from the leather at the end of the tanning process. Any sulphuric acid process, to be efficient, should not leave free sulphuric acid in the dry leather. This acid is used in quantities varying from 20 to 50 lb. for 50 hides, and the pelt swells to a considerable extent; but before the whole of the hide is permanently set a large portion of the acid will have been removed by the various tan liquors. With the sulphuric acid process, the amount of acid fixed by the hide reaches its maximum at the beginning of the tanning process, and if the swollen fibres are exposed to the tannin solution by cutting a piece off the hide, the cut surface appears to fix a large amount of tannin, and changes to a deep red. If, however, the conditions are such that the untanned portions of the fibre are gradually giving up acid to the outer solution before they are fixed by the tannin, the resulting leather is not a dark red colour providing sufficient acid diffuses out of the hide.

### *Sulphuric acid experiment.*

#### *First test.*

		Sections 2 and 5.		Sections 1 and 6.	
		Normal wattle bark liquors.		Acid wattle bark liquors.	
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.	
1	8°	2.5	8°	2.5	
3	12°	2.6	12°	2.6	
5	17°	3.5	17°	3.5	
8	23°	5.6	23°	5.6	
17	32°	6.2	32°	6.2	
21	45°	8.7	45°	8.7	
26	60°	10.6	60°	10.6	

#### *Second test.*

		Sections 4 and 7.		Sections 3 and 8.	
		Normal wattle bark liquors.		Acid wattle bark liquors.	
Days.	Barkometer.	Lime water.	Barkometer.	Lime water.	
1	8°	2.7	8°	2.7	
4	12°	3.0	12°	3.0	
5	17°	5.7	17°	5.7	
9	23°	5.6	23°	5.6	
17	32°	6.8	32°	6.8	
19	45°	9.0	45°	9.0	
23	60°	11.4	60°	11.4	

Procter and Wilson state that the rate of tanning will be a maximum for a given concentration of liquor when the potential differences are of opposite signs and the absolute value of each is a maximum. We have found that, after the second week, the acetic acid in the first experiment reached a certain concentration in the untanned portion of the pelt, and as a direct result an excess of tannin was fixed as indicated by the dark red streak in the centre of the hide. Under certain conditions the same dark red colour could be obtained with the sulphuric acid leather. To obtain these results the differences in potential must have reached its maximum for these experiments when the pelt had fixed the maximum amount of acid. As the difference in potential increases, the rate of penetration would appear to decrease, and it is probable that at a certain point, tannin is deposited on the fibre in sufficient quantities to form a barrier to further penetration.



We have seen sole leather with an untanned streak in the centre of the dark red leather and this was supposed to be due to some abnormal state in the sulphuric acid process. As the tannin diffuses into the hide it will choose the paths of least resistance and diffuse along the less dense spaces between the fibres. These spaces probably contain a weak solution of protein matter which is precipitated by the tannin, leaving what are known as air spaces in the dry leather. When the hide fibres swell under the influence of acid solutions, the outward pull may be directly proportional to the increase in the volume of the individual fibres; but in swelling they will tend to fill up any open spaces in the hide and retard the penetration of the tannin. If there are open spaces in the hide at this stage the outward pull (for a hide) would not be directly proportional to its increased volume as measured by external dimensions.

The amount of tannin fixed by a hide is proportional to the concentration of tannin in the liquors. In the normal tanning process the pelt is first placed in weak liquors. This was probably rendered necessary in the past because the hides contained a considerable amount of lime when they reached the first tan-liquors, and calcium tannate would be precipitated on the outer portion of the leather, with a tendency to retard the penetration of the tannin if strong liquors were used. The amount of tannin and calcium tannate deposited would then be at the maximum.

Strong liquors on a neutral or slightly acid pelt are supposed to give this undesirable result, which would be intensified with the higher percentages of acid as used in these experiments. There are several reasons for using weak liquors in the early stages of the tanning process, but if there is any value in the theory that an excess of tannin deposited on the fibres will retard penetration to an appreciable extent, then strong tan-liquors should not be used to fix the highly swollen sulphuric acid pelt in the early stages. A sound definition of "strong liquors" for the various stages in the process of tanning is impossible without another series of comparative tests to deal with this factor. Many tanners, more especially those who use bark and do their own leaching, would certainly start their hides in stronger liquors providing the loss was not excessive when pumping away tail-end liquors.

Our results for these experiments show that the amount of fixed tannin (see degree of tannage) increases with the acid processes, and from our water absorption tests we find that, as further amounts of tannin are fixed on the leather, greater resistance is offered to the penetration of water. The wear-resistance of leather will be proportional to the mass, other factors being equal, and as fixed tannin increases the mass this power of resistance will be proportional to the fixed tannin. If the combination between hide substance and tannin be a chemical one, then this portion of the leather is held together by cohesive forces, differing from the total solubles which are held in the leather by adhesive forces.

Both combined tannins in excess and water-solubles are capable of adversely affecting the results by imparting to the leather brittle qualities which will vary according to the tannins used. In a hot, dry climate leather containing large amounts of fixed tannins is inclined to become brittle or it loses some of its elastic properties. The same leather might give general satisfaction in Europe, where it retains that amount of water which is required to keep it in a pliable state. As far as sole leather is concerned, any brittle qualities which can be traced to any excess of combined tannins will not adversely affect the wearing qualities, providing the leather contains sufficient fatty matter and water.

The water-soluble matter in leather is brittle or plastic according to the amount of water it contains, and therefore varies with atmospheric changes, tending to a harsh, brittle leather in a dry climate and offering very little resistance to the penetration of water. Owing to its hygroscopic properties glucose is useful for keeping a leather mellow or pliable during exposure to a high temperature and low humidity, but these properties render it extremely unsuitable in any climate for sole leather which must, at certain times, resist the penetration of water to a degree not attainable with a leather containing glucose. Fatty matters make leather pliable and enable it to offer great resistance to the penetration of water. Fats, but not glucose, are the necessary constituents of a good sole leather.

Riethof\* states that sole leathers for the British and United States Governments are allowed to contain 25–26% of water-soluble matter. Temperature for extraction is not given. As both these Governments are advised by experts we assume that trained leather trade chemists are satisfied that this soluble matter contributes to a considerable extent to the wear resistance properties of sole leather. Water-solubles certainly increase the mass and may contribute to this desirable result mentioned above when they contain sufficient water to make them as plastic as cobbler's wax. There is very little evidence for and a good deal against these solubles having any value as a constituent of leather.

Six duplicate tests were made in these experiments and a total of twelve pairs for comparative purposes. The total solubles are higher for the acid processes in eleven out of the twelve pairs. If the combination between hide substance and tannin be perfectly stable under conditions as laid down by the extraction process, then the total solubles must be the residue from the tan-liquors held by capillary forces in the leather when it is hung up to dry. The amount of soluble matter would depend on the volume and concentration of the solubles in that tan-liquor which is retained by the leather under conditions mentioned above. The slight difference in the thickness between the acid and normal leather would not have any great influence on the amount of soluble matter removed when the leather is washed. A glance at the tanning process will show that the concentration of the liquor remaining in the leather should be approximately constant for each pair, so that we must now prove that the volume of the liquor retained by capillary forces was greater for the acid leather, or doubt must be felt as regards the stability of the combined hide substance and tannin.

#### Water absorption test.

Dry leather = 100.

Tannage.	Acid.	Normal.	Acid.	Normal.
Number .....	3	7	8	4
Acetic acid No. 1 ..	62.5	74.4	81.1	103%
Acetic acid No. 2 ..	99	110	130	135%
Sulphuric acid No. 3 ..	79.3	127	100.4	102%

The volume of the liquors will always be proportional to the interspaces of the leather. The hide sections increase in weight during the tanning process, but this increase is not equal to the amount of combined tannin and total solubles, so the difference must be due to water expelled from the leather. We find that a greater percentage of water has been expelled from the acid

\* Hide and Leather.



sections, and if the interspaces in the leather have decreased to a corresponding amount we would expect to find it confirmed by the water-absorption test. The amount of water absorbed is lower for the acid sections and therefore we assume that the volume of liquor held by the leather was proportionately lower for the acid sections and consequently the total solubles should be lower. The percentage of non-tans in the total solubles is lower for the acid leather in nine out of the twelve single tests, and in this as in previous experiments we have noted the small percentage of non-tans in various leathers. Such a result may be due to the non-tans decomposing much more quickly than the tannin or it may be due to the hydrolysis of the leather.

We have shown the soluble tannins as a percentage on the total tannins in the leather and in eleven out of the twelve the results are higher for the acid tests. If it be possible to remove the combined tannin with water, then these results seem to indicate that as the fixed tannin increases, the stability of the combined hide substance and tannin decreases.

The sulphuric acid plumped leather (first test) contained 0.57% of free sulphuric acid as shown by the Procter and Searle method of analysis. This is a bad result from a practical standpoint but we could probably have removed all the free sulphuric acid with sodium acetate or calcium carbonate used in conjunction with the drum process. As the concentration of the acid is

*Gain in weight during tanning process. Pelt weight=100.*

Tannage.	Section Nos.							
	A.	N.	A.	N.	A.	N.	A.	N.
	1	5	6	2	3	7	8	4
Acetic acid No. 1 .....	12.3	11.4	12	11.6	14.4	12.8	13.9	13
Acetic acid No. 2 .....	10.1	10.2	11.6	9.9	9.5	10.5	11.5	10.7
Sulphuric acid No. 3 .....	14.2	12.7	12.2	12	16	14.2	19.6	17

*Acetic acid experiment. No. 1.*

Section Nos. ....	Test 1.				Test 2.			
	1	5	6	2	3	7	8	4
	A.	N.	A.	N.	A.	N.	A.	N.
Water .....	14.00	14.00	14.00	14.00	14.00	14.00	14.00	14.00
Total solubles .....	17.31	15.87	19.11	16.89	19.14	17.27	18.72	16.75
Non-tans .....	2.34	2.44	2.33	2.11	2.16	2.16	2.39	2.31
Tannin .....	14.97	13.43	16.78	14.78	16.98	15.11	16.33	14.44
Hide substance .....	42.39	43.59	42.33	44.21	40.16	43.55	38.64	42.05
Combined tannin .....	26.3	26.54	24.56	24.9	26.4	25.18	28.64	27.20
Degree of tannage .....	62.04	60.88	58.02	56.32	65.25	57.82	74.12	64.68
Pelt weight $\times 100$ .....	115.6	114.3	118.1	118.5	111.1	113.4	124.3	126.4
Green weight .....								
Leather weight $\times 100$ .....	78.79	74.03	79.18	74.2	71.92	68.57	68.32	64.49
Green weight .....								
Leather weight $\times 100$ .....	68.17	64.78	67.04	62.61	64.73	60.46	55.0	51.0
Pelt weight .....								
Hide substance in pelt .....	28.89	28.24	28.38	27.68	26.26	26.33	20.93	21.44
Fats .....	1.23	1.35	0.93	0.98	0.22	0.27	1.03	1.32
Soluble tannin $\times 100$ .....	36.27	33.6	40.59	37.24	39.14	37.5	36.31	34.68
Total tannins .....								

*Acetic acid experiment. No. 2.*

Section Nos. ....	Test 1.				Test 2.			
	1	5	6	2	3	7	8	4
	A.	N.	A.	N.	A.	N.	A.	N.
Water .....	14.00	14.00	14.00	14.00	14.00	14.00	14.00	14.00
Total solubles .....	15.48	14.83	14.86	14.07	12.63	13.27	14.92	14.69
Non-tans .....	1.95	1.9	1.88	1.58	1.68	1.75	1.94	2.34
Tannin .....	13.53	12.93	12.98	12.49	10.95	11.52	12.98	12.35
Hide substance .....	44.12	45.56	44.78	46.06	45.73	46.0	44.48	45.94
Combined tannin .....	26.40	25.61	26.36	25.87	27.64	26.73	26.6	25.37
Degree of tannage .....	59.84	56.21	58.86	56.16	60.44	58.11	63.03	55.22
Pelt weight $\times 100$ .....	122.1	116.6	112.5	123.0	109.7	118.7	147.8	132.6
Green weight .....								
Leather weight $\times 100$ .....	77.32	71.62	71.62	72.89	60.17	61.03	66.28	58.65
Green weight .....								
Leather weight $\times 100$ .....	63.3	61.3	60.54	59.26	54.83	51.37	45.38	43.87
Pelt weight .....								
Hide substance in pelt .....	27.93	27.96	26.94	27.29	25.14	23.62	20.17	19.4
Fats .....	1.46	1.04	1.03	1.05	0.21	0.2	0.53	0.35
Soluble tannin $\times 100$ .....	33.8	33.55	32.99	32.46	28.38	30.12	32.8	32.72
Total tannin .....								

*Sulphuric acid experiment.*

Section Nos. ....	Test 1.				Test 2.			
	1	5	6	2	3	7	8	4
Tannage .....	A.	N.	A.	N.	A.	N.	A.	N.
Water .....	14.00	14.00	14.00	14.00	14.00	14.00	14.00	14.00
Total solubles .....	19.73	17.07	20.26	16.85	16.94	14.20	19.53	17.10
Non-tans .....	2.72	2.02	2.63	2.55	1.54	1.53	2.27	1.55
Tannin .....	17.01	15.05	17.63	14.30	15.40	12.67	17.26	15.55
Hide substance .....	40.16	43.32	40.01	43.78	41.44	46.05	37.73	42.24
Combined tannin .....	26.11	25.61	25.70	25.37	27.62	25.75	28.74	26.66
Degree of tannage .....	65.01	59.12	64.18	57.94	66.65	55.91	76.17	63.41
Pelt weight $\times 100$ .....	118.7	119.0	110.9	118.8	123.8	121.2	138.2	142.0
Green weight .....								
Leather weight $\times 100$ .....	85.66	77.22	84.88	75.73	76.81	68.52	77.86	71.08
Green weight .....								
Leather weight $\times 100$ .....	72.17	64.9	72.61	63.74	62.14	56.55	56.33	50.04
Pelt weight .....								
Hide substance in pelt .....	27.89	27.57	27.01	27.5	25.64	25.95	20.91	20.54
Fats .....	2.48	2.35	1.58	1.84	0.58	0.65	1.5	1.36
Soluble tannin $\times 100$ .....	39.45	37.01	40.69	36.05	35.8	32.9	37.52	36.84
Total tannin .....								

*Tensile strength test.*

Experiment.	Tannage.	Section	Original dimensions.			Stress in pounds.		Total elongation.	Moisture.
			Breadth.	Thickness.	Area.	Total.	Per sq. in.	%	%
Acetic acid No. 1	Normal.	4 D	1.531	0.25	0.3827	1820	4755	36	13.62
	Acid.	8 D	1.62	0.252	0.4082	1350	3307	38	13.9
Acetic acid No. 1	Normal.	7 B	1.551	0.214	0.3319	1240	3736	41	14.1
	Acid.	3 B	1.541	0.234	0.3605	1000	2773	37.5	14.01
Acetic acid No. 1	Normal.	7 D	1.543	0.207	0.3194	1220	3819	38.8	14.1
	Acid.	3 D	1.536	0.217	0.3333	1030	3090	33.3	14.01
Acetic acid No. 2	Normal.	4 X	1.642	0.212	0.3481	540	1551	38	13.72
	Acid.	8 X	1.645	0.234	0.3849	610	1584	34	14.24
Acetic acid No. 2	Normal.	7 X	1.55	0.157	0.2433	680	2794	43	14.13
	Acid.	3 X	1.65	0.165	0.2722	600	2204	37.5	14.37
Acetic acid No. 2	Normal.	7 L	1.55	0.172	0.2666	670	2513	38.8	14.13
	Acid.	3 L	1.556	0.171	0.266	660	2481	37.5	14.37
Sulphuric acid No. 3	Normal.	7 T	1.559	0.249	0.3881	1150	2963	45.8	14.19
	Acid.	3 T	1.528	0.233	0.356	820	2303	26	14.5
Sulphuric acid No. 3	Normal.	7 H	1.651	0.252	0.3933	1060	2695	43	14.19
	Acid.	3 H	1.525	0.280	0.427	940	2201	21	14.5

increased in the pelt the resulting leather becomes harder and more rigid, the elasticity decreases, the amount of fixed tannin increases, and also the resistance to the penetration of water. From the commercial standpoint it is a desirable process, giving greater weight and stouter leather, but under conditions obtaining in a hot dry climate it is probable that the resistance to wear decreases.

The actual tanning process probably ceases when the fibre is permanently set, a result obtained with a liquor containing a low concentration of tannin. Any increase of tannin obtained after that point is reached can only act as a filling agent and the good results obtained from chrome sole leather show that there are better filling agents than the vegetable tannins. A considerable amount of leather is struck through in the pits and then placed in the drum to be filled up with extract, etc. The weak point in this process is the filling agent. The tanner's process is a correct one but the material with which he is supplied is not good. What is wanted is a substance insoluble in water and one that will remain plastic when the leather is dry.

Our work confirms some of the previous results obtained by J. R. Blockey.

**Yorkshire Section.**

*Meeting held at Leeds on Friday, February 4th, 1918.*

PROF. J. W. COBB IN THE CHAIR.

**LOSS OF INDICAN DURING AIR-DRYING OF INDIGO LEAF.**

BY PROF. E. R. WATSON, M.A., D.Sc.

This investigation was undertaken in connection with an examination of the chemical nature of the substances in solution in seeth-water (A. G. Perkin, Chem. Soc. Trans., 1916, 109, 210 *et seq.*).

The percentage of indican in the samples of leaf examined was determined by boiling the aqueous extract with isatin and hydrochloric acid and weighing the indirubin produced, according to the method worked out by Gaunt, Thomas, and Bloxam, under Prof. A. G. Perkin's directions (this Journal, 1907, 1178).

The method of extraction described by Orchard-



son, Wood, and Bloxam (this J., 1907, 7) did not prove efficient for fresh leaf, probably because the unbroken epidermis of the fresh leaf is more or less impermeable to water. It was found that all the indican was extracted if boiling water was poured over the leaves and the water then boiled with the leaves for one minute. Nothing less than this was sufficient, as the following figures show:—

*Indirubin from 10 grms. of fresh leaf.*

	Sample 1.	Sample 3.	Sample 4.
Extraction according to method of Orchardson, Wood, and Bloxam ( <i>loc. cit.</i> ) .....	0.0190	—	—
Leaves steeped in boiling water for 1 minute .....	—	0.0877 } 0.0817 }	0.0843
Boiling water poured over leaves and water boiled with leaves for $\frac{1}{2}$ minute .....	—	—	0.0945
Bitto, boiling for 1 minute ..	0.1008	0.1057	0.0970

The method of extraction adopted was as follows:—Ten grms. of leaf was placed in an Erlenmeyer flask of 250 c.c. capacity and about 75 c.c. of boiling water poured over them. The water in the flask was at once brought to the boil and kept at the boil for one minute. The water was quickly drained from the leaves and filtered and the leaves were treated with successive quantities of water at 60° C., each quantity being allowed to stand with the leaves for one minute, then drained off, filtered, and added to the extract until its volume was made up to 250 c.c. This extract was treated with the same quantity of isatin and hydrochloric acid as prescribed by Gaunt, Thomas, and Bloxam (*loc. cit.*), for 5 grms. of dried leaf. (It was found that fresh leaf lost about two-thirds of its weight on air-drying.) For the sake of uniformity all samples of leaf, both fresh and air-dried, were extracted in this way.

The first sample of leaf (kindly supplied by Mr. R. S. Finlow, Bengal Agricultural Department) was collected in Behar, packed very loosely in a glass-stoppered bottle, and sent by post to Dacca for analysis, so that analysis was made two days after the leaves were collected. The second, third, and fourth samples were plucked at different times from plants grown in Dacca and the fresh leaf was analysed within an hour of plucking. For air-drying the leaflets (separated from the stalks—all analyses were for the separated leaflets) were spread in a thin layer on sheets of filter-paper, covered by a fine cotton net, and allowed to stand in a N. verandah until they were practically constant in weight. This took 3–4 days. The results obtained are tabulated below.

*Indirubin obtained from 10 grms. of leaf (weighed in fresh condition).*

	Leaf extracted whilst fresh.	Leaf extracted after air-drying.
Sample 1 ..	0.1008	0.0568 } 0.0515 }
Sample 2 ..	0.0825 } 0.0832 }	0.0610 } 0.0593 }
Sample 3 ..	0.1057	0.0763 } 0.0732 }

In each case air-drying has caused a very considerable loss of indican. Samples 2 and 3 lost 27.5 and 29.3% of indican respectively. Sample 1 lost much more. This was the sample which had stood for 2 days in a glass-stoppered bottle before examination.

If leaf is packed closely and allowed to stand, some fermentation takes place and all the indican may disappear. In this case a good deal of indigo-blue is deposited in the leaves. From

this it was expected that protracted drying in thicker layers would cause a greater loss of indican; and this was found to be the case.

	Leaf extracted whilst fresh.	Extracted after air-drying in 3–4 days.	Extracted after protracted air-drying in thicker layers.
Sample 3..	0.1057	0.0763 } 0.0732 }	0.0597

It was also found that rapid drying in a steam-oven completely destroyed the indican.

The results obtained are of some general interest as they indicate that any drying or standing of the leaf before extraction will considerably reduce the yield of indigo. In earlier days the leaf was sometimes dried before extraction; *c.g.*, in Crookes' "Dyeing and Calico Printing," 1874 edition, pp. 448 and 451, there is a quotation (Twist) to the effect that in Guzerat the leaves were dried in the sun; and in Bancroft's "Philosophy of Permanent Colours," Vol. I, p. 173, it is stated that in some cases the stems, and leaves are put into the steeping-vat at once, in others after being more or less dried. It has been stated that in the Madras Presidency it was the custom to dry the leaves before steeping, but apparently it is not the practice there to-day. Gaunt, Thomas, and Bloxam (*loc. cit.*, pp. 1180–1181) remark that probably there is considerable loss of indican during air-drying, but so far as the writer is aware the present note is the first record of any reliable investigation of the point. The loss of indigo-yielding glucoside which may occur if the leaf is not put into the steeping-vats immediately is evidently very considerable, and obviously arrangements should be made to avoid any delay. In the cane-sugar industry it has long been recognised that commercial success depends on getting the cane into the factory without delay, as otherwise some inversion of the cane-sugar occurs. Apparently delay is equally serious to the indigo-planter, yet the present note records the first quantitative investigation as to whether there is any loss if the leaf is allowed to stand or dry before putting into the steeping-vat.

Colour Chemistry and Dyeing Department,  
Leeds University.

## Communication.

### SEPARATION OF SECONDARY ARYLAMINES FROM PRIMARY AMINES.

BY TUDOR WILLIAMS PRICE, B.A., M.Sc., A.I.C.

When secondary arylamines are prepared in the usual way by heating a primary amine with an alcohol in the presence of a condensing agent such as sulphuric acid or hydrochloric acid, the product obtained is always contaminated with unchanged primary amine and also with tertiary amine. The isolation of the secondary amine by fractional distillation of the crude product is tedious and also unsatisfactory owing to the closeness of the boiling points of the primary, secondary, and tertiary amines. The isolation of the secondary amine by conversion into the nitrosamine and reduction of the latter is expensive and more-over produces low yields, so is inapplicable except on a purely laboratory scale.

During the course of an investigation into the conditions affecting the production of ethyl-*o*-toluidine, it was found that a very satisfactory and economical separation of the secondary amine from *o*-toluidine could be made by means of

sulphuric acid. After the method had been worked out, it was discovered that a similar method had been used by Gnehm and Blumer (this J., 1899, 18, 129) in the separation of methyl-*o*-toluidine from *o*-toluidine. Few details are given by these authors and consequently it was thought that it would be of interest and utility to publish the details worked out by the present author.

Primary aromatic amines combine with concentrated sulphuric acid to form solid sulphates which are insoluble in the free base. Secondary and tertiary amines, on the other hand, under the same conditions form syrupy sulphates which dissolve in the free base. It is found that an economical method of separation of these substances can be based on this difference in behaviour.

If a mixture of primary, secondary, and tertiary amines is treated with the requisite quantity of 96% sulphuric acid to combine with the primary amine present, the distribution of the sulphuric acid is influenced by two factors, namely, the relative strengths of the bases present and the degree of insolubility of their sulphates. Experiments have shown that the latter factor predominates and that practically the whole of the primary amine is precipitated as sulphate. A small proportion only of the sulphuric acid combines with the alkylated amines but the sulphates of these compounds remain dissolved in the excess of the amines, and consequently the primary sulphate can be removed by filtration.

*Experimental.* The sulphuric acid used was of sp. gr. 1.84 (168° Tw.). The ethyl-*o*-toluidine was made by heating *o*-toluidine and ethyl alcohol with sulphuric acid in an autoclave. The crude product was once distilled to remove alcohol and impurities of high boiling point.

The amount of *o*-toluidine present in ethyl-*o*-toluidine was determined by means of the dye formed with R-salt (sodium- $\beta$ -naphthol-3.6-disulphonate), observing the precautions recommended by Reverdin and de la Harpe (Ber., 1889, 22, 1004).

*Separation of ethyl-*o*-toluidine from *o*-toluidine.* The crude ethyl-*o*-toluidine generally contains 10–15% of unchanged *o*-toluidine and the amine used in the majority of these experiments contained this percentage of *o*-toluidine.

A preliminary experiment, using the theoretical amount of sulphuric acid required to combine with all the *o*-toluidine, showed that the amount of primary base was reduced from 10% to 7%.

A series of experiments was then carried out, in which the proportion of sulphuric acid to the mixed bases was gradually increased until it was greatly in excess of the amount required to combine with all the *o*-toluidine present. Great heat is developed during the combination, and after cooling to ordinary temperature the mixture was filtered. Filtration was tedious and the sulphate could not be obtained free from base. Washing with ether removed the adhering base from the sulphate. The results are shown in Table I.

These results show that even with a 30% excess of sulphuric acid a complete separation was not obtained, and that an excess of about 15% was necessary to reduce the primary base content from 10% to 1–2%. Later, working with much larger quantities it was found that only a very slight excess of sulphuric acid was required and that an excess larger than about 3–5% produced some syrupy sulphate of the secondary amine, which made filtration almost impossible.

*Freezing out of sulphate.* The first series of experiments showed that a complete separation of the primary and secondary amines is not obtained even when an amount of sulphuric acid greatly in excess of that required to combine with all the primary amine is added. This might be due to the slight solubility of the *o*-toluidine sulphate in the secondary amine at room temperature. Accordingly 150 grms. of ethyl-*o*-toluidine containing 10% *o*-toluidine was treated with 8 grms. of 96% sulphuric acid; in one case the mixture was cooled to room temperature and in the other to –13° C. by means of a freezing mixture. The sulphate was filtered in each case and the amount of primary amine left in the mixture determined. In the first case, 102 grms. of filtrate was obtained containing 1.6% of *o*-toluidine; the sulphate yielded 39 grms. of amine containing 24.8% of *o*-toluidine. When the mixture was cooled to –13° C. and filtered, the filtrate weighed 103 grms. and contained 1.8% of *o*-toluidine; the sulphate yielded 39 grms. of amine containing 22.3% of *o*-toluidine.

These experiments show that there is no difference in the separation when the mixture is cooled to –13° C. or to room temperature and that the incompleteness of the separation is not due to a slight solubility of the *o*-toluidine sulphate in ethyl-*o*-toluidine.

The probable explanation of the incompleteness of the separation is that although the bulk of the sulphuric acid combines with the *o*-toluidine, owing to the insolubility of its sulphate, yet a certain amount combines with the ethyl-*o*-toluidine. If this be true, then successive treatments with sulphuric acid should diminish the amount of *o*-toluidine left in the mixture until it becomes a negligible quantity. To confirm this view some ethyl-*o*-toluidine containing a high percentage of *o*-toluidine was treated as described below.

100 grms. of ethyl-*o*-toluidine containing 16% of *o*-toluidine was treated with 8 grms. of 96% sulphuric acid. After filtration 50 grms. of ethyl-*o*-toluidine containing 6.9% of *o*-toluidine was obtained. The 50 grms. of ethyl-*o*-toluidine was treated with 2 grms. of 96% sulphuric acid and filtered. The filtrate weighed 36 grms. and contained 1.3% of *o*-toluidine.

In another experiment 2800 grms. of ethyl-*o*-toluidine containing 12% of primary base was treated with 180 grms. of 96% sulphuric acid and put through a filter press after cooling to room temperature. 1850 grms. of ethyl-*o*-toluidine containing 2.9% of primary base was obtained.

TABLE I.

Effect of excess of sulphuric acid upon the separation of ethyl-*o*-toluidine from a mixture containing 10% *o*-toluidine.

No. of Expt.	Mixed bases, wt. in grms.	Sulphuric acid.		Filtrate.		Base obtained from sulphate.	
		Wt. in grms.	Excess.	Wt. in grms.	Primary base.	Wt. in grms.	Primary base.
1.....	100	5.0	% 9	71	% 3.3	22	% 26.4
2.....	150	8.0	14	102	1.6	39	24.8
3.....	100	6.0	30	69	1.4	13*	49.6

\* Sulphate washed with ether before neutralising.



1800 grms. of the filtrate from the last experiment was treated with 30 grms. of 96% sulphuric acid and filtered. 1625 grms. of ethyl-*o*-toluidine containing 0.6% of *o*-toluidine was obtained. These experiments confirm the statement that successive treatments with sulphuric acid are necessary owing to its distribution between the two amines.

*Large scale experiments.* The separation of ethyl-*o*-toluidine from *o*-toluidine having been successfully performed on a laboratory scale, it was decided to apply it on a semi-manufacturing scale. For this purpose filtration of the sulphate is too slow and a centrifuge was used. In order not to clog the centrifuge by the syrupy sulphate of the secondary base, the amount of sulphuric acid used was exactly sufficient to combine with all the *o*-toluidine present. 187.3 lb. of ethyl-*o*-toluidine containing 15.6% of *o*-toluidine was treated with 14 lb. of sulphuric acid. After standing overnight it was filtered and 114 lb. of ethyl-*o*-toluidine containing 2.7% of *o*-toluidine was obtained. This contained 70% of the secondary amine originally present. In a second experiment, 184.5 lb. of ethyl-*o*-toluidine containing 4.1% of primary amine was treated with 3 lb. 11 oz. of sulphuric acid. After passing through the centrifuge 152.5 lb. of ethyl-*o*-toluidine containing 1% of primary amine was obtained. This is a yield of 85% of the secondary amine originally present.

These results show that the method can be applied to large scale working equally with laboratory experiments. The yield of secondary amine after purification is always better the lower the original content of primary base. This is due to the secondary base being retained by the sulphate of the primary base on the filter or centrifuge. To obtain a greater yield the sulphate can be washed with an anhydrous solvent, such as benzene or alcohol, in which the sulphate is not appreciably soluble. By distillation the solvent and secondary amine can be recovered separately. This was done with the sulphate from the last two experiments. The combined sulphates as taken from the centrifuge weighed 96 lb. The theoretical weight should be 56.5 lb. calculated on the sulphuric acid used. Hence 39.5 lb. of secondary amine was retained by the sulphate. The sulphate was accordingly churned up with 100 lb. of benzene in a closed vessel and then centrifuged. The benzene was distilled and the residue above 100° C. weighed 40.5 lb. and contained 2.0% of *o*-toluidine. The sulphate was air-dried for several days and weighed 55 lb. The above figures show that an almost theoretical yield of ethyl-*o*-toluidine can be obtained when the sulphate is washed free of adhering secondary amine by an anhydrous solvent.

For a continuous process the best solvent is absolute alcohol, since then the sulphate need not be dried but can be utilised at once for a fresh autoclave charge, after analysis. This was done and the resulting crude ethyl-*o*-toluidine was of the same quality as when obtained from *o*-toluidine, alcohol, and sulphuric acid.

The method outlined above is therefore an economical and satisfactory method of separating ethyl-*o*-toluidine, since the *o*-toluidine sulphate,

and the alcohol used in washing it, can be used for a fresh autoclave charge and no non-essential material is used at all.

The above method of removing a primary amine is applicable to other primary amines as is shown by the following experiments.

*Separation of mono-methylaniline from aniline.* 100 grms. of monomethylaniline containing 10% of aniline was treated with 5 grms. of 96% sulphuric acid and filtered. 80 grms. of monomethylaniline containing 1.8% aniline was obtained. The yield is 87% of the monomethylaniline originally present.

*Separation of dimethylaniline from aniline.* 100 grms. of dimethylaniline containing 12% aniline was treated with 6 grms. of 96% sulphuric acid and filtered; the yield was 75 grms. of dimethylaniline containing 0.4% of aniline. This is equivalent to 83% of the dimethylaniline originally present.

*Separation of a mixture of mono- and di-methylaniline from aniline.* 100 grms. of a mixture of mono- and dimethylaniline containing 5% of aniline was treated with 2.5 grms. of 96% sulphuric acid and filtered; yield = 84 grms. of mixed amines containing 0.1% of aniline. This is equivalent to a yield of 88% of the mono- and dimethylaniline originally present.

*Separation of ethylaniline from aniline.* 50 grms. of ethylaniline containing 11.3% of aniline was treated with 2.8 grms. of sulphuric acid and filtered. 36 grms. of ethylaniline containing 0.42% of aniline was recovered. This is equivalent to a yield of 80.6% of the secondary amine originally present.

*Separation of diethylaniline from aniline.* 50 grms. of diethylaniline containing 7.8% of aniline was treated with 2 grms. of sulphuric acid and filtered. 38 grms. of diethylaniline, containing 0.15% of aniline was recovered. The yield is therefore 82.3% of the tertiary amine originally present.

The above results show that the sulphate method of removing a primary amine is applicable to a mixture of primary and secondary or primary and tertiary, or primary, secondary, and tertiary amines.

#### SUMMARY.

Experiments have been carried out on the separation of primary arylamines from secondary and tertiary amines by means of sulphuric acid. The mixture is treated with a quantity of sulphuric acid sufficient to combine with all the primary amine and filtered. If desired a second treatment may be given.

An increased yield can be obtained by washing the sulphate of the primary amine free from secondary or tertiary amine by means of an anhydrous solvent. If desired, the sulphate can be used for a fresh autoclave charge.

In conclusion, the author wishes to express his thanks to Messrs. Nobel's Explosives Co., Ltd., and to Mr. Wm. Rintoul, Manager of the Research Section, for the facilities offered for carrying out this work and permission to publish the results.

Research Laboratory,  
Ardeer Factory,  
Stevenston.

## Edinburgh Section.

Meeting held at Edinburgh on Tuesday, January 15th, 1918.

MR. D. B. DOTT IN THE CHAIR.

### A CONVENIENT ARRANGEMENT FOR ILLUMINATING A CHEMICAL BALANCE.

BY B. D. PORRITT, M.Sc.(LOND.), F.I.C.

In arranging the illumination of a balance, in addition to securing satisfactory lighting of the graduated beam, pans, and pointer, care must be taken to avoid any unequal expansion of the balance arms due to heat, or disturbance of equilibrium arising from air currents. For these reasons the source of light should be placed symmetrically to the beam at the top of the balance case and should be as extensive as possible in order to reduce heat radiation to a minimum and to distribute uniformly such as is unavoidable.

The type of electric lamp in which the light source consists of a metallic filament suspended along a narrow tube seemed to be free from the defects of the ordinary type, since the heat evolved would be slight and largely dissipated, and further, owing to the shape of the lamp, it could be easily arranged so that any slight heating effect should be uniformly distributed over the beam of the balance.

In the arrangement here described a "tubolite" metallic filament lamp of 16 candle-power, 8½ inches long, is employed, enclosed in a semi-circular aluminium reflector which serves both to direct the light through the glass top into the balance case and to screen the light source from the view of the person weighing. Both these fittings are standard articles in the electrical trade.

In order to ensure the lamp and reflector always being placed symmetrically to and slightly in front of the balance arm, two upright brass springs are attached to the wood work of the balance case and into circular counter-sunk holes in these are sprung the bosses at each end of the reflector.

The lamp and fittings can therefore be removed for cleaning or other reasons by merely loosening the springs, and, to facilitate this operation and to protect the latter from injury, they are each screened by a standard of polished ebonite or hard wood. By this device the balance and its source of illumination are self-contained, and the case may be altered in position at will by merely lengthening the flexible lead or by making connection to another point of the electrical supply.

Experiments indicate that the increase of temperature inside the balance case under normal conditions of weighing by such artificial illumination should not amount to more than 2°–3° C. Such a variation, when uniform, should not affect the accuracy of an ordinary analytical balance giving trustworthy results over the range of temperature variation experienced in ordinary laboratory practice.

Evidence has also been adduced that no disturbance of equilibrium due to uneven heating of the balance arms or air currents need be feared when employing this form of illumination.

In conclusion, I have to express my indebtedness to Mr. A. C. D. Smith and Mr. D. McKinnon, of the Engineering and Vulcanite Departments respectively, for their co-operation in designing and making the device which forms the subject of the present communication.

Research Laboratory,  
Castle Mills,  
Edinburgh.

## DETERMINATION OF NITROGEN CONTENT OF RUBBER.

BY MATTHEW HOWIE.

Some time ago it was necessary to determine the nitrogen content of a very considerable number of samples of plantation rubber. The nature of the nitrogen compounds present in rubber is still uncertain, but it was thought that they would be fairly easily decomposed into ammonia, and this conjecture proved later to be correct. The method adopted was Wilfarth's copper sulphate modification of the Kjeldahl process, which gave complete satisfaction.

It is very generally held that in Kjeldahl estimations, of whatever kind, the end point or the point at which digestion with sulphuric acid may cease is when the solution becomes clear. The factor which should decide the completion of any determination, however, is not the point at which the solution becomes transparent but that at which the nitrogen compounds present have been completely converted into ammonia. Owing to the large proportion of hydrocarbons present in rubber, it was found that in practically every case at least 6 hours was necessary to clarify the solution, and it was decided to carry out an investigation with a view to determining whether or not the time of heating could be curtailed.

In each determination about 1 grm. of rubber was digested with 30 c.c. of sulphuric acid, 7 grms. of potassium sulphate, and about 1 grm. of anhydrous copper sulphate. Six separate estimations were carried out in which after 1, 2, 3, 4, 5, and 6 hours respectively, the heating was discontinued and the nitrogen figure determined in the usual way. The mixture digested for 6 hours had become quite transparent although not colourless; the others still contained varying proportions of suspended carbon. The results obtained are given in the following table.

Time of heating. Hours.	Plantation sheet.		Manihot rubber.
	Sample I. Nitrogen %.	Sample II. Nitrogen %.	Nitrogen %.
1	0.19	0.31	0.68
2	0.20	0.30	0.70
3	0.23	0.32	0.72
4	0.24	0.33	0.72
5	0.23	0.32	0.73
6	0.24	0.32	0.72

These results show that, for rubber at any rate, it is not necessary to clear the solution, and that from 3–4 hours is quite long enough to get the maximum nitrogen figure.

Laboratory,  
Castle Mills,  
Edinburgh.

## London Section.

### CORROSION OF LEAD ROOFING.

(See this J., 1918, 39 T.)

#### DISCUSSION.

MR. F. SOUTHERDEN writes as follows:—In May last I examined two samples of a yellowish-white incrustation formed by the corrosion of lead roofing at Axminster Church. The material, like that analysed by Prof. Brame, had the characters of commercial white lead, though of poor colour.



On ignition, the loss of weight was 13.6% and 13.2% respectively on the two samples, as compared with 13.7% required by the formula,  $2\text{PbCO}_3, \text{Pb}(\text{OH})_2$ . The lead had been laid on oak boards, and both the oak and lead of the south aisle, from which the samples were taken, had been renewed as recently as 1909. On turning up the sheets in several places the oak boarding was found to be very thickly coated with the deposit, 3.6 oz. (exclusive of flakes left on the lead sheet) being taken from an area of 6 in. by 6 in.; that is, nearly 7 lb. of lead per sq. yd. had been destroyed in a period of 8 years. Perforation of the lead was observed at three points. It is worthy of special note that the lead of the nave roof, laid upon deal in 1833, is in quite good condition. The danger of laying lead upon oak is, I understand, generally appreciated by architects, being attributed to the tannin present in the wood, and experiment shows that a solution of ordinary tannin exerts a solvent action on clean lead. For the dates given I am indebted to Mr. Harbottle Reed, F.R.I.B.A., the architect consulted, who also took the samples.

*Meeting held at Burlington House on Monday, March 4th, 1918.*

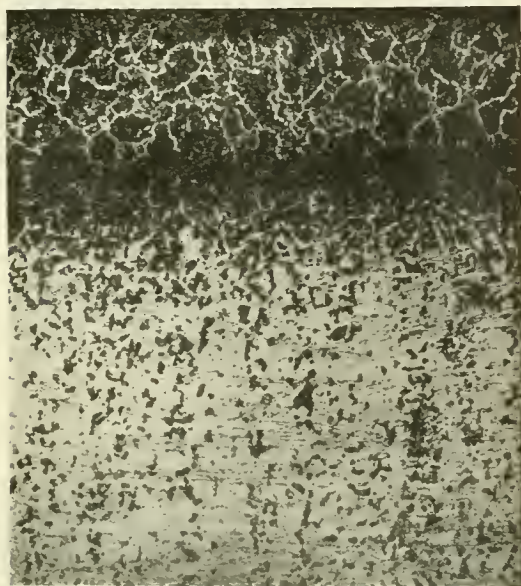
DR. C. A. KEANE IN THE CHAIR.

### SOME REACTIONS OF ACETYLENE.

BY W. R. HODGKINSON, C.B.E.

It is well known that acetylene polymerises, mainly to benzene, when heated in a closed or partially closed vessel at  $120^\circ$ – $150^\circ$  C. At higher temperatures and pressure above normal, and especially at varying pressures, acetylene decomposes into carbon and hydrogen.

The gas can, however, be passed, at a moderate speed, through a glass or silica tube heated to  $600^\circ$ – $800^\circ$  C. either alone or diluted with an inert gas as nitrogen or hydrogen chloride or coal gas, for a moderate time (20–60 minutes) before any serious deposition of carbon occurs. The nature of the glass has a decided influence on the rate of decomposition. Silica has less effect than soda-glass.



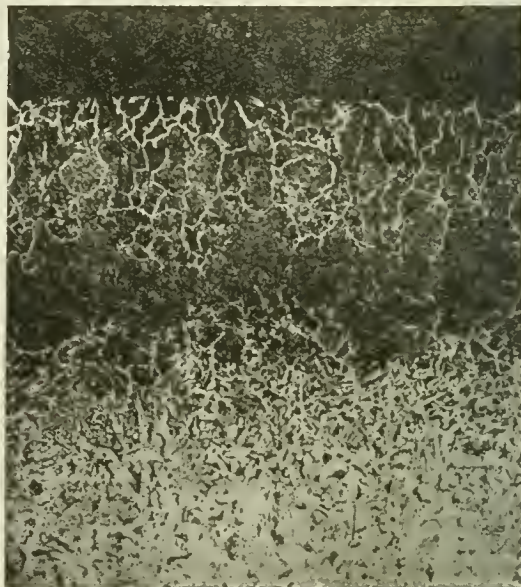
I.  
1 hour.  $1050^\circ$  C.

Various metals were heated to temperatures from  $100^\circ$  to  $1060^\circ$  C.\* in a stream of acetylene for like time periods. Cadmium, zinc, lead, tin, copper, aluminium, and others showed no particular action at temperatures up to their melting points. No change was found in the physical state of the metals, and no carbon was taken up by them, and the carbon deposited burnt off without residue.

Iron, nickel, cobalt, and to a lesser extent manganese, tungsten, platinum, and palladium, almost in this order, showed a decided reactivity when heated, in some cases to very moderate temperatures (*e.g.*,  $200^\circ$  C.), in acetylene, either pure or diluted.

Under favourable conditions of temperature and supply of the gas, the metal, if in moderately small pieces as wire, frequently glowed, the glow passing along the whole mass of metal employed. Carbon deposited on the metal and, after attaining a moderate thickness, the layer peeled off and another formed.

In the case of iron the strength of the metal was retained—in fact somewhat increased; in the case of nickel and cobalt (and to a lesser extent some



II.  
2 hours.  $1050^\circ$  C.

of the metals before mentioned) a great physical change was observed. They became quite brittle, breaking up on attempting to bend them, and also showed very decided corrosion and pitting. This action (with nickel) commenced at comparatively low temperatures, *e.g.*,  $200^\circ$  C., and became rapid at  $600^\circ$  C.

In the case of iron, a more or less deep carburisation resulted. With nickel and cobalt carburisation was also observed.

The most remarkable part of this action of acetylene is that as the carbon of the acetylene enters the metals (Fe, Ni, Co), some metal enters into and is contained in the soot-like deposit around the metal. This extrusion or volatilisation of the metal was found to occur to a maximum extent with nickel, followed in order by cobalt and iron. A nickel wire (No. 14) was in fact entirely dispersed or disseminated into the sooty

\* Temperatures were controlled by melting points of metals or by Rh-Pt pyrometer.



deposit by heating for a few hours in the gas stream.

The amount of metal contained in this black deposit was found to vary considerably in different experiments. The cause of variation is not yet quite determined.

In some cases as much as 15% of nickel was obtained, in others about 5%. With iron 3% and more has been observed. Dilution of the acetylene was found not to stop the wandering of the metal, but simply to reduce the rate a little.

The carburising of iron by heating in acetylene is very rapid, especially at temperatures above 800° C., and many observations indicate that it reaches a maximum just below 1000° C. The type of carburisation is quite peculiar, the carbon showing distinct diffusion into the metal. The accompanying photomicrographs are of iron carburised by heating in acetylene for different periods at 1050° C.

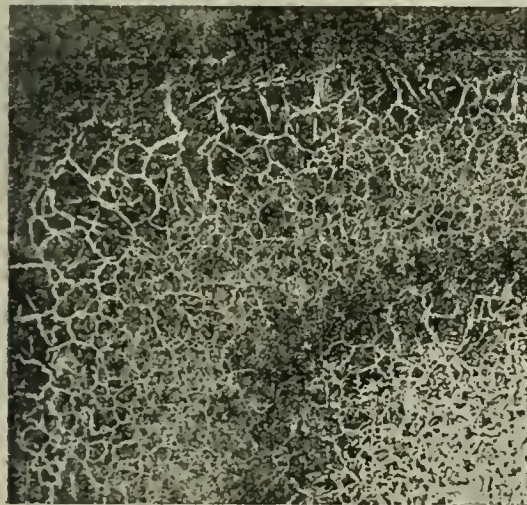
Ammonia was used to dilute acetylene in some experiments on metals because it prevented to a remarkable degree the deposition of carbon from acetylene by heat, and also because the flame of the mixture was nearly smokeless.

In this gas mixture, iron, nickel, and cobalt were found to be more rapidly carburised than by acetylene alone, and the amount of extraneous carbon, or soot, reduced.

It was imagined that the cause of the more rapid absorption of carbon by the metals might be due to the simultaneous formation of a nitride and possibly its further reaction with the acetylene.

Iron (or other metal) was heated in ammonia to 800° C. At something below this temperature fine iron wire was quickly converted into nitride,  $\text{Fe}_3\text{N}$ .

Ferrous nitride heated in acetylene lost its nitrogen and the iron became highly carburised.



3 hours. 1050° C.

### III.

Unfortunately, the physical change on conversion into nitride was not altered by the subsequent heating with acetylene. The iron remained brittle. On heating to bright redness and quenching, the steely structure of a highly carburised iron was obtained.

Further experiments showed that when ferrous nitride was heated in acetylene, ammonia was formed, but the matter was not followed up quantitatively.

Amines, as aniline, etc., have been found to act as very rapid carburising agents on iron and nickel.

Many benzenic compounds (benzene, toluene, phenol, aniline, naphthalene, and others), when passed as vapour over nickel or iron, were found to act in a manner somewhat similar to acetylene—that is, a deposition of soot formed on the metal and a quantity of the metal was found to have wandered into this soot. In some cases the amount of nickel in the “soot” (benzene, aniline, phenol) was greater for the same time and temperature of heating than with acetylene.

Iron and other metals were observed to act in a similar manner, but not to the same degree as nickel.

### DISCUSSION.

Mr. WALTER REID remarked that iron burners would not work with acetylene even when they were chilled; apparently from the experiments made by Dr. Hodgkinson the destruction of the iron was due to volatilisation. The experiments also appeared to explain why acetylene black always contained iron.

Mr. GARDNER asked if the author had considered the possibility of carbon monoxide being present in the acetylene in the case of nickel. A very small quantity would cause vaporisation.

Dr. M. O. FORSTER believed that the gas given off from the tube would be found to contain cyanogen; it might be possible to produce aniline.

Dr. R. LESSING mentioned that iron was invariably found in coal tar and pitch. In most cases this was locked up in the free carbon. The ash in retort carbon also contained iron, but this was partly due to mechanical admixture of coke dust containing iron in the ash. The carbon or soot deposited during thermal decomposition of hydrocarbons, e.g. coal gas, always contained minute traces of iron, which could not be due to mechanical impurities. Whether this decomposition of iron was due to reactions of acetylene or of some hydrocarbons which were present in coal gas in much larger proportion than acetylene was difficult to say.

Prof. HODGKINSON replied that there was no carbon monoxide present as there had been no oxygen in the tube. It was possible that there were carbides of iron and nickel which vaporised slightly and then decomposed into metal and carbon. Cyanogen was present in the gases from the tube.

### ACID-RESISTING IRON AND ITS USES IN CHEMICAL PLANT.

BY S. J. TUNGAY.

For many years chemists and metallurgists sought some form of metal which could be adapted to the requirements of the chemical industry, for the purpose of resisting the corrosive action of acids, but it is only in comparatively recent years that great developments have taken place in the introduction of forms of acid-resisting iron, which are suitable for the manufacture of chemical plant.

It was long recognised that the replacement of brittle, friable, or uncertain materials, ordinarily used for the construction of chemical plant, such as glass, earthenware, porcelain, pottery, or the more recently developed forms of fused quartz or silica, by a metal of the nature of cast iron, would present great attraction alike to the chemist, the chemical engineer, and the chemical manufacturer, provided that such metal could be made to resist acids. Many of the difficulties against which the chemical engineer has long had to contend were occasioned by the limitations in the character of materials that could be used for the construction of plant, owing to the highly corrosive nature of the various reagents and chemical substances for which the plant was to be used. Once a metal of reasonable strength and of reasonably certain behaviour for dealing with acids could be



obtained, a large number of objections and difficulties in the way of construction of plant were easily overcome. The obvious utility, therefore, of a suitable metal in the form of iron which would withstand the corrosive action of sulphuric acid or nitric acid, or a mixture of the two, was of such profound interest to the chemical industry as to open up a fertile field for research, particularly in view of the vast possibilities of such a product on a commercial scale.

Various iron alloys have been known for many years which are more or less resistant to the corrosive action of such acids as sulphuric, nitric, and hydrochloric acids, and long since it has been proved in the laboratory that a pure form of iron could be rendered resistant to either sulphuric or nitric acid by the addition of a suitable proportion of silicon, chromium, or other elements. The development and application of such metals upon a commercial or industrial basis was not, however, approached until some twenty years ago. In Paris, Jouve was successfully making castings of small beakers, pans, and evaporating dishes in a form of passive iron which consisted largely of a refined cast iron with a high silicon content, but he was compelled to confine his operations chiefly to small castings and small vessels. Later, in Germany, "Neutrалеisen," so-called, was produced by more than one metallurgical house for the purpose of making castings to withstand nitric or sulphuric acid, but its use was both transitory and fraught with much discouragement, to the end that one industrial concern decided to abandon its manufacture, whilst another found itself surrounded with continued difficulties and disaster except as far as concerned the manufacture of small castings.

The composition of acid-resisting iron has been in some quarters guarded with much jealousy and surrounded with secrecy, notwithstanding the fact that to the competent chemist or metallurgist the secret of its composition could not long be maintained. There is perhaps something to be said after all in favour of guarding a metal alloy as a secret compound, in view of the fact that under the present condition of the British patent law it has been found that a patent covering a metal alloy comprising two or more known metals, cannot be held as valid.

In more recent years the development of the electric furnace has proved to be a valuable aid to the satisfactory commercial production of such alloys as ferro-silicon and ferro-chromium, and consequently these and other ferro-alloys are now produced both of good quality and in good quantities in many countries, chiefly perhaps in Norway, Sweden, Italy, Russia, and very extensively in America.

In America, Kowalke has made considerable research to determine the resistance to corrosion of alloys of iron and silicon, and I am able to quote some very remarkable figures as follows:—

#### *Loss in 10% sulphuric acid.*

Si %.	Area, sq. cm.	Cumulative loss, %.		
		51 hrs.	75 hrs.	141 hrs.
2.5	15.7	1.6	4.1	6.5
3.3	21.9	64.9	87.5	92.8
7.4	11.5	31.0	48.9	62.0
9.9	12.7	10.7	19.8	23.1
11.1	13.0	5.0	8.8	13.1
12.4	9.2	1.2	2.1	2.4
13.6	16.6	0.2	0.6	0.9
14.8	18.0	0.03	0.05	0.05
16.1	10.2	0.03	0.04	0.04
17.3	11.9	0.03	0.03	0.03
19.8	9.6	0.13	0.2	0.25

#### *Loss in 10% nitric acid.*

Si %.	Area, sq. cm.	Cumulative loss, %.		
		115 hrs.	166 hrs.	14 days.
2.5	8.4	20.8	48.0	53.5
3.3	11.8	13.3	21.6	22.1
7.4	11.2	8.9	11.1	11.4
9.9	7.3	5.9	8.7	8.9
11.1	10.4	2.4	3.5	3.6
12.4	13.1	2.3	4.6	4.6
13.6	11.7	0.05	0.1	0.1
14.8	6.3	0.013	0.02	0.02
16.1	11.4	0.006	0.003	0.0015
17.3	11.2	0.006	0.007	0.007
18.5	8.9	0.037	0.044	0.046
19.8	13.8	0.01	0.01	0.01

#### *Loss in 10% hydrochloric acid.*

Si %.	Area, sq. cm.	Cumulative loss, %.		
		7 days.	16 days.	21 days.
2.5	8.4	2.4	10.0	15.7
3.3	11.8	9.71	38.7	60.8
7.4	11.2	5.2	19.0	27.3
9.9	7.3	7.1	16.4	21.1
11.1	10.4	4.9	8.1	9.4
12.4	13.1	1.4	2.7	4.0
13.6	11.7	0.18	0.51	0.67
14.8	6.3	0.12	0.31	0.42
16.1	11.4	0.12	0.27	0.36
17.3	11.2	0.31	0.40	0.41
18.5	8.9	0.03	0.03	0.04
19.8	10.8	0.1	0.28	0.28

Certain figures in these tests are worthy of special remark. It is particularly noteworthy that silicon present in a lesser quantity than 12% does not promote a satisfactory resistance to corrosion, whilst when the silicon content reaches 19% or upwards the acid-resisting quality of the alloy again falls. Once, however, the character of these alloys was determined, the satisfactory production of them upon a commercial scale was found by the earlier undertakings to be a somewhat speculative industry, as it easily occurs, except with most careful working of the furnaces, that much of the silicon which should enter the alloy, is lost in the form of "graphite," and the alloy produced is ultimately found to be so deficient in silicon as to be no more resistant to corrosion than ordinary cast iron.

In addition to the serious difficulties presented by the considerable shrinkage of these non-corrosive iron castings during cooling, which amounts to slightly over  $\frac{1}{4}$ -inch per foot in each direction as compared with a shrinkage of about  $\frac{3}{32}$ -in. per foot in the case of ordinary cast iron, the presence of graphite in any considerable quantity causes disaster. The carbon and phosphorus content of the iron must also be low, otherwise during the process of cooling these compounds may tend to separate out and form eutectics, the separated compounds resulting in pellets which are found in small blow-holes or cavities in the thickness of the metal, and this notwithstanding that the alloy may have been carefully and correctly mixed in the first instance.

Some tests recently made at the Manchester School of Technology show these eutectics to consist largely of phosphorus and silicon. Whilst a normal average quality of pig iron may show an analysis of say, Si 3.45, S 0.03, P 0.64, Mn 0.66, combined carbon 0.27, graphite 3.48, Fe 91.47%, to produce an acid-resisting or non-corrosive iron it is necessary to get rid of what may be termed the 10% of "impurities" and make the alloy from as pure a form of high grade iron as possible.

These difficulties have largely been overcome by suitable furnaces and by improved manipulation in mixing the metal, with the result that in present commercial practice it is found quite possible to produce a satisfactory acid-resisting iron: but its application for the manufacture of vessels and plant of large capacity suitable for the requirements of the chemical industry, had up to recent years on all sides resulted in failure and disappointment, and often much loss.

It is due to the energies of British chemical engineers and metallurgists that great manufacturing difficulties have been surmounted, and that we are now enabled to produce a large variety of vessels and plant suitable for the manufacture of nitric and sulphuric acids, and suitable for chemical processes involving the use of such acids for the advancement of chemical progress and industry. Lasting credit is due to men like Jouve, Lennox, Pearce, and Marsden of Manchester, who each sought to overcome the manufacturing difficulties of making large castings for construction of plant from these different alloys. This has been followed by more recent work by Dr. Rossi in Italy, and others in the United States.

Whilst, as I have already stated, there may be no secret as regards the composition of the alloy, its satisfactory manipulation for producing reliable vessels and castings is a question of craftsmanship of the highest order. These methods of manipulation vary at the different works producing acid-resisting iron castings, and are for the most part somewhat jealously guarded.

I exhibited some specimens of acid-resisting iron castings at the Chemical Engineering Exhibition in London in 1913, which provoked much favourable comment, notably some parts of nitric acid condensation plant and vessels for the concentration of sulphuric acid. As a result, chemical manufacturers rapidly displayed an interest in plant constructed of "Ironac" acid-resisting iron.

Following are the results of some corrosion tests made in the course of practical work, with test pieces from large vessels made of "Ironac" acid-resisting iron, being chiefly test pieces from works plant of large capacity.

#### Corrosion tests on "Ironac" acid-resisting iron.

Acid.	Initial weight, grams.	Loss after boiling at 150° C. for :		
		24 hrs.	48 hrs.	72 hrs.
Sulphuric acid 65% .....	112.648	Nil.	Nil.	Nil.
Sulphuric acid 20% .....	115.207	Nil.	Nil.	Nil.
Nitric acid 90% .....	113.392	0.011	0.038	0.074
Nitric acid 30% .....	110.620	0.013	0.016	0.016

#### Loss after boiling in mixed acids as follows :—

Mixed acid.	—	Original weight, grms.	Loss in grms.
65% H <sub>2</sub> SO <sub>4</sub> 2% HNO <sub>3</sub> 33% water	24 hrs. at 150° C.	(1) 303.0	Nil
		(2) 385.59	Nil
		(3) 15.903	Nil
	15 hrs. at 150° C.	(1) 302.9	0.1
		(2) 385.59	Nil
		(3) 15.903	Nil
	15.5 hrs. at 150° C.	(1) 302.6	0.3
		(2) 385.5	0.09
		(3) 15.903	0.08
	12.5 at 150° C. to 260° C.	(1) 302.6	Nil
		(2) 385.5	Nil
		(3) 15.903	0.08

After each experiment the specimens were boiled out with water several times and then dried on the hot plate at a temperature of about 400° C.

The above tests are of more value to the chemical engineer or works chemist, inasmuch as they do not relate to special test pieces prepared for the laboratory.

One authority has given the following physical constants of acid-resisting iron as compared with cast iron :—

	Ordinary cast iron.	Acid-resisting iron.
Density .....	7.3	6.8
Tensile strength (tons per sq. in.)	9—10	6—7
Melting point .....	1150° C.	1200° C.
Hardness .....	24	35
Heat conductivity .....	10	8
Electrical resistance .....	8	16
Contraction per ft. in casting ..	3/32	9/32
Crushing strength, 1 in. cubes	40 tons	34 tons

In actual practice I have been able to maintain nearly all these figures, although the results vary somewhat according to the variation of the alloys.

Since the outbreak of war, acid-resisting iron has played no small part in the construction of plant for the production of propellants and high explosives. For the condensation of nitric acid, this metal was found a great boon, as plant of large capacity had to be rapidly erected at short notice, and it was found practicable to instal many large nitric acid plants of acid-resisting iron very rapidly—a result which could not have been effected by using pottery owing to the excessive length of time required for its manufacture. In addition, it was found that acid-resisting iron possessed a much higher efficiency owing to its heat conductivity being greater than that of earthenware. The heat-transmitting power of acid-resisting iron has been calculated to be nearly 10 times the heat-conductivity of stoneware or quartz, consequently parts of plant intended for conducting heat may be made of considerably smaller dimensions than would be the case if such parts were made of stoneware. The heat-transmitting power is about twice that of lead. A condenser built of acid-resisting iron condensed a charge of nitric acid in 16 hours, which was usually found to take 36 hours with a similar condenser built in pottery.

A further wide application of acid-resisting iron has been its use in connection with plant for concentration of sulphuric acid—a very large number of installations having been built up of cascade type as well as modifications of Kessler and other types of plant for rectification of sulphuric acid and re-concentration of waste acids from explosives manufacture. Its use is becoming daily of wider application, and whilst primarily it has been adopted as a metal to resist sulphuric and nitric acids, it is becoming more and more useful for other acids such as hydrochloric, acetic, citric, etc.

In spite of the success already attained, there are still factors of difficulty to be overcome. All alloys of low silicon content, say under 10%, are attacked very readily by sulphuric, hydrochloric, acetic, and citric acids. These alloys are not exceedingly brittle, but alloys containing from 16% to 18% silicon, whilst they are exceedingly resistant to sulphuric, hydrochloric, nitric, acetic, and citric acids, are extremely brittle, and are so very hard that it is impossible to machine them in any other way than by grinding with high-speed abrasive wheels. These points are decided drawbacks in many cases against the utilisation of acid-resisting iron, and experiments are still being continued to improve the tensile strength of the metal and produce an alloy that will be both malleable and machinable.

Notwithstanding the specific drawbacks already mentioned, it is possible to provide parts with



flanges and faces carefully ground on the surface to a very high and accurate finish, so that pipes of large and small diameters may be carefully jointed together without leakage.

Certain definite points must be held in mind in designing parts of plant or apparatus for manufacture in these acid-resisting irons. All joints in pipe work and vessels should be formed by means of flanges or spigot and socket, since the metal is too hard to screw or to take a screwed thread, and it is preferable so to design pipes and cored castings, that the use of chaplets can be avoided as far as possible. Another point in design is that all sharp corners must as far as possible be avoided, owing to the considerable shrinkage in cooling which has already been mentioned, otherwise sharp corners will become a source of weakness, and the vessel is very likely to sustain a fracture through strains set up by shrinkage in cooling. For the same reason all vessels such as boiling pans, receivers, stills, should be made curved at the bottom, as large flat surfaces present difficulties through contraction of the castings.

Few of these acid-resisting irons can be recommended for vessels which are required to be worked at high internal pressures, and it is not usually considered safe to use pressure vessels for any internal pressure higher than 50 lb. per square inch, excepting in cases where the vessels are of quite small diameter.

As already mentioned, the melting point of acid-resisting iron is considerably higher than that of ordinary cast iron, and this for many purposes is a decided advantage. In a statement made last year before the American Institute of Chemical Engineers, a case was mentioned regarding some nitric acid works equipped almost entirely with plant of acid-resisting iron. The works sustained a serious fire after being in operation for some 8 months, during which time no corrosion or breakages occurred with the plant, and whilst the fire destroyed the building, the acid-resisting plant was found to be unharmed, and 80% of the material was recovered and is still in use. It is questionable whether similar apparatus constructed of stoneware or other ceramic material could have withstood so unexpected a test.

Apart from the actual construction of chemical plant, these alloys have a wide application, such as in manufacturing anodes in connection with electro-metallurgical processes. After the outbreak of war, it was found impossible to obtain magnetite anodes, since these were chiefly made in Germany, and a substitute was found in acid-resisting iron. Whilst the material is not absolutely unacted upon when used as an anode in copper sulphate solution, many times its original weight of copper can be deposited before the anode shows serious signs of corrosion. The mechanical strength of the acid-resisting iron is a further decided advantage, although a little higher electrical energy is required than with anodes made of magnetite. Some experiments made in 1915 at the University of Illinois\* disclosed remarkable properties, which proved the metal to be superior to many other metals for certain electro-magnetic machinery, and, with the addition of aluminium, the magnetic properties of the metal were greatly improved and increased toughness resulted.

#### DISCUSSION.

Capt. GOODWIN said that some 10 or 15 different makes of acid-resisting irons showed curious differences in composition. The average silicon content was about 15% in the United States and yet one sample would contain about 0.3% manganese and another 2½%. He was able to confirm what Mr. Tungay had said as to hydrochloric acid,

particularly hot acid, and also to some extent as to chlorine. As far as he knew there was only one acid-resisting iron which would withstand their action satisfactorily and that had a high silicon content, viz., 16 to 20%, together with a small percentage of rare metal, the name of which he was not at liberty to mention. The skin of silicon iron castings was different from the body, and while usually the action on the skin was not greater than on the body, yet some materials such as hot ammonium chloride attacked the skin more rapidly. Further research was also necessary regarding the action of acetic acid. He believed that most castings of acid-resisting iron were pickled before they were sent out to see if acid would attack them or not. That suggested the necessity for investigation from the electrochemical point of view. In the design of plant he thought there had been too great a tendency to copy designs of apparatus usually adopted when stoneware or silica was used. Flanges in the pipes were frequently used, but were a source of weakness owing to the unequal contraction on cooling, and he suggested the use of conical buttress joints. Apparatus had also been put on the market for which acid-resisting iron was economically unsuitable, e.g., nitric acid retorts and fans, which latter, owing to considerations of strength and speed, would not give more than one inch draught. The future belonged to all kinds of acid-resisting metals, lacquers, and varnishes, and not only to iron. An acid-resisting aluminium would be extremely useful and was already on the American market.

Mr. SWINDIN said that he regarded these irons as glasses rather than as metals in the ordinary sense of the term, owing to their extreme brittleness. If in using acid-resisting iron manufacturers never made anything very large, kept it always in the form of a pipe, never made anything very flat, and kept away from flanges wherever possible, they would have very little trouble. The hardness was a nuisance. The use of these irons was bound to increase because of the rise of a new industry—the concentration of weak nitric acid produced from the fixation of atmospheric nitrogen. The physical constants of these alloys as given by the author were too generous to be accepted. The tensile stress to which the metal could be subjected meant nothing. Some lucky test piece might be satisfactory in this respect, but no one would rely upon the test. The next advance in the manufacture of these alloys must be in the direction of toughness.

Mr. GARDNER said that the figures given for corrosion were of little value since the area exposed was not given.

Mr. W. L. OAKDEN asked if acid-resisting iron had been tried in the construction of ordinary sulphuric acid chambers in place of lead, and how it compared as regards cost. Had this material been used in the Glover tower for the ordinary sulphuric acid process? The inlet pipe of this tower was soon subject to corrosion.

Mr. J. W. HINCULEY suggested to the author that when he compared ordinary cast iron with acid-resisting iron he should choose a good chemical iron. The one selected was an agricultural implement iron. The silicon and the graphite were far too high and the combined carbon was too low, whilst the proportions of phosphorus, manganese, and sulphur were bad. Referring to the author's table, he particularly drew attention to the shrinkage of 3/32 in. in the case of cast iron, and 9/32 in. in the case of acid-resisting iron. The shrinkage in the case of cast iron was not all contraction: it was the difference between contraction on cooling and expansion due to the separation of graphite. If the cast iron had no graphite the shrinkage would be the same as that of the acid-resisting iron. The best acid-resisting irons were those free from graphite,

\* Univ. Illinois Exp. Stat., Bull. 95.



and manufacturers should really make it their business to produce such a material. To do this it was necessary to melt the materials in the electric furnace, and the kind of furnace must be chosen with a view to keeping carbon away from the material. The question of machining was not so hopeless as some might think. It was possible to drill and machine some brands fairly satisfactorily if it were done carefully and the tools properly chosen.

Mr. H. M. RIDGE asked if the author had observed any action due to the presence of carbides; were these separated out in the high-silicon metal and, if so, were they appreciably attacked by acids when the vessels were in use? The author had not referred to the use of acid-resisting iron or steel for cutlery; it was well known that chromium was used for that purpose.

Mr. C. H. ALLDREE pointed out that the results given in the paper were chiefly with rather strong acids. He had found that whereas with sulphuric acid above 20%, or even above 10%, the metal would stand very well, when the percentage was lower, say from 2 to  $\frac{1}{2}$ %, there was difficulty. In the case of a pump for pumping weak acid, the parts were destroyed almost as quickly as ordinary cast iron and particularly the piston, which had been ground and freed from skin. Further, one portion would be acted upon more rapidly than another. He had not found any material that would withstand hydrochloric acid.

Mr. C. O. BANNISTER said that although it was simple to make materials that would withstand the action of strong acids, one primary cause of failure was porosity of the casting. Sometimes this porosity was quite minute and was only discernible under the microscope. Another difficulty was graphite, and in spite of what Mr. Hinchley had said he was afraid this would never be got rid of. The best thing to do was to get it in a finely divided state and evenly distributed throughout the casting. The sulphur and phosphorus should be kept to the lowest possible limit. As to the divergence in the amount of manganese, he had found castings varying from 0.5 to 2% manganese behave perfectly well.

Mr. WALTER REID said that one method of removing the stresses which sometimes gave rise to fracture when a strain came upon flanges and similar parts of castings, was to treat the portion in which the strain existed to the heat of an oxy-acetylene blowpipe; after cooling the metal was very much stronger. It was also possible that heat treatment such as had been found so useful with steel, applied after casting, would result in the metal being very much tougher and more resistant to shock than it was at present.

Mr. TUNGAY replied that the actions of hydrochloric acid and chlorine were being investigated. The output of this iron had recently been largely confined to nitric and sulphuric acid plants, and the question of hydrochloric acid had not been of paramount importance. The skin of the casting was more easily attacked than the body, as had been suggested. The skin consisted generally of free particles of iron which adhered in the cooling, and on cooling it seemed to detach itself partially in the form of a scale and was rapidly attacked and cleaned off in pickling. He could not agree with Mr. Swindin that this iron was of the nature of a glass, as it would stand very much heavier treatment and altogether had much greater capabilities. In reply to Mr. Hinchley, he said that the cast iron taken for comparison was normal quality cast iron, taken out of Ryland's list.

Mr. HINCHLEY pointed out that that was pig iron.

Mr. TUNGAY agreed that the analysis was that of ordinary pig iron. In reply to Mr. Ridge, he said that carbides had separated out of the metal on cooling

and it was the separation of the carbides that at times caused great trouble. He had used low strength acids on some acid-resisting iron and had found quite satisfactory results. Porosity was one of the greatest difficulties and the endeavour was to keep the graphite as low as possible.

## Nottingham Section.

*Meeting held at University College, Nottingham, on Wednesday, February 20th, 1918.*

DR. R. M. CAVEN IN THE CHAIR.

### THE ANALYSIS OF ALUMINIUM ALLOYS.

BY BERNARD COLLITT AND WILLIAM REGAN, B.S.C.

The use of alloys of aluminium in the construction of aircraft and other engines of warfare necessitates very numerous analyses of the alloys, and requires, moreover, that the results of such analyses be available in the shortest possible time after the receipt of the samples.

During the past two or three years, we have been called upon to examine a considerable number of these light alloys, and have naturally been much interested in two important papers on the subject which have recently appeared by W. H. Withey\* and J. H. Stansbie† respectively.

Our work was carried out several months before the publication of Mr. Stansbie's paper, and we find ourselves in agreement with him on the following points:—

1. That for rapid working it is advisable to "open out" the alloys with a solution of caustic soda for the determination of most of the alloying metals.

2. That on filtering the resulting alkaline solution, practically all the aluminium and zinc are to be found in the filtrate, whilst copper, iron, nickel, magnesium, and all the manganese except a trace, are left as a residue on the filter paper.

3. That a small amount of aluminium accompanies the copper, iron, etc., so that the iron cannot be estimated gravimetrically by precipitation with ammonia.

The alloys which we have examined may be divided into three classes or groups, as follows:—

*Class I.*—Alloys containing between 10 and 15% of copper, with or without about 1% of manganese.

*Class II.*—Alloys containing 10 to 20% of zinc, up to 5% of copper, and small amounts of any of the following metals: iron, nickel, manganese, and magnesium.

*Class III.*—Alloys containing up to 5% of copper and one or more of the following metals in amounts not usually exceeding  $1\frac{1}{2}$  or 2%: iron, nickel, manganese, zinc, magnesium.

Lead and tin were not present in appreciable amounts in the alloys which came under our notice.

In Class I. alloys, we prefer to estimate copper by a volumetric method, and in Classes II. and III. to determine it gravimetrically. We have avoided the separation and estimation of copper by electrolysis, as we consider that the methods outlined below are quite as accurate, and are more rapid, especially when dealing with large numbers of samples.

#### *Alloys of Class I.*

In order to comply with certain specifications, these alloys must contain copper and manganese

\* This J., 1916, 472.

† This J., 1917, 802.



within stated limits, and the chemical analysis is therefore usually confined to the determination of these two metals.

**Copper.**—One gram of fine drillings of the alloy is weighed out into a 400 c.c. tall beaker and 30 c.c. of solution of sodium hydroxide (15%) is added, or the alloy is covered with 100 c.c. of distilled water, and about 5 grms. of sodium peroxide added in small portions. The beaker is kept covered by a clock glass until violent reaction has ceased. The sides of the beaker are then washed down with a little water and the solution boiled until the alloy is thoroughly decomposed. After dilution with hot water to about 250 c.c. the solution is again boiled for a short time and then the precipitate of metallic copper and manganese quickly settles out and can be filtered off and washed with hot water. The filtrate is rejected; the residue is dissolved on the filter into a 300 c.c. conical flask by means of hot nitric acid (sp.gr. 1.18) and the filter paper washed free from copper and manganese with hot water and hot dilute nitric acid. The solution of copper is now evaporated to very small bulk, diluted slightly, 5 c.c. of ammonia solution (sp.gr. 0.90) is added, and the contents of the flask boiled for at least one minute. After cooling, 6 c.c. of glacial acetic acid is added, then 50 c.c. of sodium fluoride solution, followed by 10 to 15 c.c. of solution of potassium iodide (25 grms. in 100 c.c.). The solution is now ready for titration with standard solution of sodium thiosulphate, starch indicator being added when the colour of the solution shows that very little free iodine remains.

The standard solution of sodium thiosulphate is prepared by dissolving 400 grms. of the pure salt in distilled water and diluting to ten litres; 1 c.c. equals approximately 0.01 gm. Cu. The solution is standardised against the purest electrolytic copper. Particular attention is directed to the use of sodium fluoride, as suggested by Fraser,\* in order to eliminate the interfering effect of any iron present in the solution to be titrated. The sodium fluoride solution is made up by saturating distilled water with the salt, filtering, and diluting 500 c.c. of the filtrate with distilled water to one litre.

In connection with the analysis, or rather with the sampling of these alloys containing 10 to 15% of copper, it should be noted that they are subject to liquation when in the molten condition, and unless suitable precautions are taken, castings poured from the same crucible or melting pot may contain considerably different percentages of copper, as shown by the following figures:—

Melt S.		Melt H.B.3.	
Casting No.	% Cu.	Casting No.	% Cu.
1. ....	12.52	1. ....	12.29
2. ....	12.80	3. ....	12.54
3. ....	13.03	5. ....	12.79
4. ....	13.20		

Melt II.C.		Melt II.D.	
Casting No.	% Cu.	Casting No.	% Cu.
1. ....	11.71	1. ....	11.21
2. ....	11.61	2. ....	11.54
3. ....	12.06	3. ....	11.51
4. ....	12.11	4. ....	12.41
5. ....	12.86	5. ....	12.46
6. ....	12.86	6. ....	12.86
7. ....	12.94	7. ....	13.87
8. ....	13.04	8. ....	13.06

\* This Journal, 1915, 462.

**Manganese.**—Two of the volumetric methods, suitably modified, used for the determination of this element in steel, were tried, and gave excellent results, both as regards accuracy and rapidity. The second method, which involves no separation or filtration, is particularly speedy.

(1) One gm. of the alloy is weighed out into a 300 c.c. conical flask and 25 c.c. of solution of sodium hydroxide (15%) is added. The mixture is heated until the alloy is completely decomposed, then cooled, nitric acid (sp.gr. 1.42) is added until the solution is just acid, and then 35 c.c. of the same acid in excess. The mixture is boiled until everything is in solution, diluted with 35 c.c. of water and a little sodium bismuthate added. After boiling, the permanganate colour (or the precipitated manganese dioxide) is destroyed with a few drops of sulphurous acid solution, and all nitrous fumes boiled off. The solution is then cooled, and when quite cold, and after adding 1 gm. of sodium bismuthate, is shaken well, allowed to stand for a few minutes, filtered through asbestos or through an alundum crucible into a large conical flask, washed with dilute nitric acid, and titrated with standard solution of sodium arsenite, approximately  $N/10$ . It is advisable to standardise the arsenite solution against standard permanganate in a solution of the same volume and containing approximately the same amount of copper as the alloy solution to be titrated.

It is probable that the preliminary oxidation with bismuthate and subsequent reduction might be omitted altogether without affecting the accuracy of the results.

(2) One gm. of the alloy is weighed out into a 250 c.c. graduated flask of resistance glass, 25 c.c. of a solution of sodium hydroxide added, and the alloy decomposed as in method (1). After cooling, 90 c.c. of nitric acid (sp.gr. 1.18) is added, and solution completed by boiling, which is continued until all nitrous fumes are expelled. The solution is well cooled, diluted to the mark with distilled water, and mixed thoroughly. Into a 300 c.c. conical flask 50 c.c. (0.2 gm. alloy) is pipetted off, 25 c.c. of silver nitrate solution (2 grms. per litre) added, and the contents heated to boiling. Five c.c. of solution of ammonium persulphate (15%) is added, the flask immersed in boiling water for one minute, cooled, 80 c.c. of cold distilled water added, and the solution titrated with standard solution of sodium arsenite. The sodium arsenite solution should be of such strength that 1 c.c. = 0.01% Mn when working upon 0.2 gm. of alloy.

In cases where it is certain that the alloy is quite homogeneous, the determination may be shortened by weighing 0.2 gm. of alloy direct into a 300 c.c. conical flask, instead of weighing out 1 gm. of the alloy, dissolving, and taking an aliquot part of the solution.

#### *Alloys in classes II. and III.*

**Silicon** is estimated in the usual way by evaporating the solution of the metal with sulphuric acid, and in the filtrate from silica the copper is precipitated as sulphide, filtered out, washed, ignited, and weighed as oxide.

**Silicon.**—Two grms. of alloy is weighed out into a 500 c.c. tall beaker, and a mixture of 30 c.c. of hydrochloric acid (sp.gr. 1.16) and 10 c.c. of nitric acid (sp.gr. 1.42) is added very carefully, as the reaction with the alloy is violent. When all the mixture of acids has been added and the violence of the reaction has subsided, 30 c.c. of sulphuric acid (sp.gr. 1.84) is added and the solution of the alloy evaporated until the sulphates of metals have separated out and copious fumes of sulphur trioxide are evolved. The beaker and contents are allowed to cool, and about 200 c.c. of hot water is added and the separated sulphates dissolved by boiling. The residue is collected on



an ashless filter paper, thoroughly washed with hot water, ignited, and weighed as silica or as a mixture of silica and graphitic silicon.

**Copper.**—The filtrate and washings, about 350 c.c. in volume, are collected in a 600 c.c. beaker, heated almost to boiling, and the copper precipitated by the addition of two or three grms. of sodium thiosulphate crystals. On boiling, the precipitate of copper sulphide rapidly coagulates and can be filtered out very quickly, washed with hot water, ignited, and weighed as copper oxide.

Copper sulphide precipitated in the manner indicated is filtered out and washed with the greatest ease, and on ignition in an open muffle furnace is quickly converted into oxide. The method is not, however, recommended for alloys containing 10% and more of copper, as the results are not so good, probably owing to the fact that the conversion of sulphide to oxide is not completed so rapidly or thoroughly when igniting a comparatively large amount of precipitate. The following figures show what agreement may be expected when determining copper in this way; the volumetric results were obtained by the method described below, and used occasionally for comparative purposes.

Sample No.	Copper, %. Gravimetric, weighed as CuO.	Copper, %. Volumetric iodine titration.
13300 .....	2.37 2.36	2.44
	2.37 2.39	2.36
14127 .....	1.46	1.87
	1.92	1.94
4-C-17 .....	5.58	5.65
	5.63	5.65
20-C-17 .....	3.11	—
	3.11	—

**Copper (alternative method), iron, and magnesium.**

—Two grms. of the alloy is weighed out into a 500 c.c. beaker, dissolved in 100 c.c. of solution of sodium hydroxide (15%), diluted to about 300 c.c. with hot water and boiled, the residue of copper, iron, magnesium, etc., filtered out, washed with hot water, and the filter paper and residue returned to the original beaker; 10 c.c. hydrochloric acid (sp.gr. 1.16) and 10 c.c. nitric acid (sp.gr. 1.42) are then added, the mixture heated to complete solution of the metals, boiled, diluted, and filtered, and the precipitate washed with hot water and hot dilute hydrochloric acid. The bulk of filtrate and washings is kept as small as possible at this stage to prevent loss of time on subsequent evaporation. Ten c.c. of sulphuric acid (sp.gr. 1.84) is added and the solution evaporated until fumes of sulphur trioxide are evolved in order to drive off the other acids. The beaker and contents are allowed to cool, about 200 c.c. of hot water added, and the copper precipitated with a rapid stream of hydrogen sulphide, filtered, and washed with hydrogen sulphide water. The filtrate is reserved for determination of iron and magnesium. The copper sulphide on the filter paper is dissolved with hot nitric acid (sp.gr. 1.18) and washed alternately with hot water and hot nitric acid (sp.gr. 1.18), collecting the solution and washings in a 300 c.c. conical flask. The solution of copper is boiled down to very small bulk and the copper determined by titration with standard solution of thiosulphate exactly as described for alloys of Class I.

**Iron.**—The filtrate from the copper sulphide precipitation is boiled to free it from hydrogen sulphide, and a few drops of strong nitric acid added to oxidise the iron present. Three grms. of ammonium chloride is added, and a slight excess of ammonia solution (sp.gr. 0.90). The solution is now boiled; it is important that any aluminium which escaped solution in the treatment with

sodium hydroxide, but was dissolved in the acid mixture along with copper, iron, etc., be completely precipitated at this stage. The iron and aluminium hydroxides are filtered off and well washed with hot water, the filtrate and washings being reserved for the determination of magnesium. The residue of hydroxides, with the filter paper, is returned to the beaker in which precipitation took place, and 20 c.c. of hydrochloric acid (sp.gr. 1.16) added; the contents of the beaker are heated, diluted, and filtered into a 300 c.c. conical flask, and the residue of filter paper well washed with hot water and hot dilute hydrochloric acid. The acid solution of iron is then carefully reduced with solution of stannous chloride, excess of this reagent destroyed by a solution of mercuric chloride, the solution cooled thoroughly, diluted to a volume of 600 c.c. and titrated with standard N/10 or N/20 permanganate solution in the presence of phosphoric acid and manganese sulphate.

As it is necessary to separate iron before proceeding to the determination of magnesium, we prefer this process for estimating iron; in the case, however, of alloys which contain no magnesium, or in which its determination is not required, the following more rapid method for iron gives results which are sufficiently accurate for most purposes.

The alloy is decomposed in caustic soda solution as previously described, the copper, iron, nickel, etc., filtered out, washed, and dissolved in hydrochloric and nitric acids, and the solution diluted and filtered. Enough solution of ammonia (sp.gr. 0.90) is now added to precipitate the iron and any aluminium present, and to form the dark blue solution of cuprammonium salt. This solution is boiled for a short time only, the iron and aluminium hydroxides filtered off, washed, dissolved, and the iron reduced and titrated as previously described.

**Magnesium.**—To the filtrate from the iron and aluminium hydroxides, which should be practically neutral, 2.5 c.c. of ammonia solution (sp. gr. 0.90) is added, the solution heated to about 70° C. and then saturated with hydrogen sulphide. The precipitated sulphides are filtered off and washed with water containing ammonium sulphide. The filtrate is acidified and reduced by evaporation; nitric acid is then added and the volume of solution further reduced by boiling down to small bulk. Any precipitated sulphur is filtered out and the residue washed with hot water. Twenty c.c. of solution of ammonium phosphate is next added, the solution neutralised with ammonia and then, with constant stirring, more of the ammonia solution (sp.gr. 0.90) is gradually added until it forms one-third of the volume of the contents of the beaker. The solution is stirred at intervals, allowed to stand 12 hours, filtered, the precipitate washed with dilute ammonia, the filter and precipitate dried, ignited gently at first and afterwards strongly, and weighed as  $Mg_2P_2O_7$ .

**Manganese** is determined exactly as described for alloys of Class I.

**Nickel.**—We consider that it is unnecessary to separate copper before precipitating the nickel with dimethylglyoxime.

Two grams of the alloy is decomposed as directed for determination of copper, iron, and magnesium, and the residual copper, iron, nickel, etc., filtered off, and washed. The filter and residue are returned to the original beaker, and the metals dissolved in 20 c.c. of hydrochloric acid (sp. gr. 1.16) and 5 c.c. of nitric acid (sp.gr. 1.42); the solution is boiled, diluted, and filtered, and the precipitate washed with hot water and hot dilute hydrochloric acid. Six grms. of tartaric acid is added, and the solution diluted to 400 c.c., and made slightly alkaline with ammonia solution (sp.gr. 0.90) and then faintly acid with hydrochloric acid. Thirty c.c. of alcoholic solution of



dimethylglyoxime (1%) is added, and then ammonia solution, drop by drop, with constant stirring, until the solution is faintly alkaline. The beaker is allowed to stand for one hour in a warm place, the solution filtered through a tared alundum or Gooch crucible, and the residue dried for 1½ hours at 103° C., cooled in a desiccator, and weighed. The nickel dimethylglyoxime contains 20.31% nickel.

**Zinc.**—For alloys of Class II., we have used the method described by Withey in the paper already referred to. For alloys of Class III. we decompose the alloy by means of solution of caustic soda, dilute, boil, filter, and in the filtrate precipitate the zinc as sulphide. This is filtered out, washed, dissolved in hydrochloric acid, and the zinc finally precipitated as zinc ammonium phosphate, which is ignited and weighed as zinc pyrophosphate.

#### Analyses of aluminium alloys.

	I.	II.	III. (a)	III. (b)	III. (c)	III. (d)
	%	%	%	%	%	%
Silicon .....	—	0.44	0.40	0.37	0.54	0.005
Copper .....	12.13	0.79	1.97	2.37	5.63	1.91
Iron .....	0.44	0.52	0.58	0.57	1.56	0.474
Manganese ..	0.68	Nil	—	—	—	—
Nickel .....	—	Nil	0.66	1.44	0.78	1.13
Zinc .....	—	12.03	—	—	—	—
Magnesium ..	—	Nil	1.71	1.32	0.64	1.52
Aluminium ..	—	86.22	95.58	93.93	90.85	94.665
(by diff.)						

The alloys of Class III. are usually made by first preparing a rich alloy of copper, nickel, and aluminium, and adding this to the requisite weight of aluminium to produce an alloy of the desired composition. The magnesium is not included in the rich alloy (or hardener), but is added to the finished diluted alloy shortly before pouring. As it is necessary to analyse the rich alloy at times, the following methods of estimating the copper, nickel, and iron present are described.

**Silicon and iron.**—The latter element cannot conveniently be determined by treating the alloy with caustic soda solution, filtering out copper, nickel, iron, etc., and then precipitating the iron with ammonia, reducing with stannous chloride, and titrating, as the iron is accompanied by so much aluminium that the precipitate obtained on the addition of ammonia is far too large to filter and wash. The procedure followed is to dissolve 2 or 3 grms. of "hardener" in a mixture of hydrochloric and nitric acids in a 400 c.c. beaker; and when solution is complete, dilute with a little water, cool, and add 15 c.c. of sulphuric acid (sp.gr. 1.84). The solution is evaporated until fumes of sulphur trioxide are evolved, cooled, diluted, heated until all the sulphates are in solution, filtered, and the precipitate washed with hot water, dried, ignited, and weighed as SiO<sub>2</sub>. The filtrate is heated to about 70° C., hydrogen sulphide is passed in until all the copper is precipitated, the liquid is filtered, the residue being rejected, and the hydrogen sulphide boiled off from the filtrate, when 5 c.c. of nitric acid (sp.gr. 1.42) and 50 c.c. of a solution of ammonium chloride (1 in 4) are added. The solution is boiled down to about 300 c.c. and a solution of sodium hydroxide (50%) added until all the precipitated aluminium hydroxide redissolves. The liquid is heated to boiling, the precipitate allowed to settle for a short time only, filtered, and washed once or twice with hot water. The filter and residue are replaced in the original beaker, digested with 25 c.c. of hydrochloric acid (sp.gr. 1.16) until all ferric hydroxide is in solution, diluted, filtered into a 300 c.c. conical flask, and the iron reduced with stannous chloride and titrated as usual with standard N/10 solution of permanganate.

**Copper.**—0.5 gm. of alloy is weighed into a 300 c.c. conical flask, dissolved in nitric acid (sp.gr. 1.18) and copper determined by the iodide method, titrating with standard solution of sodium thiosulphate.

**Nickel.**—0.3 gm. of alloy is weighed into a 400 c.c. beaker, 50 c.c. of solution of sodium hydroxide (15%) added, the liquid is boiled, diluted with hot water, allowed to settle, and filtered, rejecting the filtrate. The filter and residue are transferred to the original beaker, and the residue dissolved in nitric acid (sp.gr. 1.18), diluted with a little hot water, and filtered into a 450 c.c. beaker, and the precipitate washed with hot water. To the filtrate a solution of ammonia (sp.gr. 0.90) is added until a very slight permanent precipitate is formed, then 15 c.c. of saturated solution of sulphur dioxide is at once added, the mixture heated to boiling, and a slight excess of decinormal solution of ammonium thiocyanate added. After boiling for ten minutes, with constant stirring, the mixture is allowed to stand for an hour, when the cuprous thiocyanate is filtered off and washed with hot water. The residue and filter paper are rejected. To the filtrate in a 1000 c.c. conical flask, 2 grms. of tartaric acid and 2 c.c. of sulphuric acid (sp.gr. 1.84), are added, a solution of ammonia (sp.gr. 0.90) added until neutral, and sulphuric acid (3%) until faintly acid. A solution of ammonia (sp.gr. 0.90) is now added to exact neutrality and 15 c.c. of 2N ammonia solution added. The liquid is cooled thoroughly and titrated with N/10 solutions of potassium cyanide and silver nitrate, using 2 c.c. of 2% solution of potassium iodide as indicator.

If the copper be estimated first, the amount of N/10 solution of ammonium thiocyanate required to precipitate all the copper and leave a slight excess of the reagent can be easily calculated.

A typical analysis of one of these rich alloys is Cu 30.2%, Fe 1.59%, Ni 16.63%.

#### DISCUSSION.

Mr. H. DROOP RICHMOND asked, in connection with the first class of alloys containing aluminium and copper, whether any use had been made of the large difference which existed between the density of aluminium and that of copper as the basis of a method for control work. Having had to examine a large number of solders with the object of classifying them, he had found that the specific gravity enabled the percentage of lead and tin to be calculated with an accuracy of about 1%. In the case of solders containing antimony, the method did not apply and it was necessary in such cases to estimate the antimony. If a similar method were employed for the examination of the copper-aluminium alloys, it should be possible to get much greater accuracy than in the examination of solders. Was it desirable in the estimation of aluminium and iron by precipitation of the metals as hydroxides by the addition of ammonia, to neutralise the solution exactly by the employment of an indicator, or was the addition of excess of ammonia and its subsequent removal by boiling equally satisfactory?

Dr. E. B. R. PRIDEAUX asked whether any attempt had been made to devise a rapid test for the examination of these alloys, depending upon their magnetic properties. He did not know whether these alloys possessed magnetic properties, but from the fact that Heusler's alloy, which contained aluminium, copper, and manganese, was distinctly magnetic, he considered it a possibility. He further suggested that the amount of hydrogen evolved on treating the alloy with alkali and then the alkali-insoluble matter with acid might be made to yield a "mean hydrogen equivalent" from which the composition of the alloy could be deduced.

Mr. T. H. ADAMS expressed his disappointment



that a method for the direct determination of aluminium had not been forthcoming. In his opinion the figure obtained by difference was not satisfactory. The most useful method he knew consisted in precipitating the aluminium as phosphate after removing the other metals, but he confessed that owing to the small quantity of the sample on which one had to work on account of the bulky nature of the precipitate, the results were not reliable to within, say, 0.2%. The authors stated that they estimated zinc by precipitation as sulphide from alkaline solution. A much better method was to precipitate in a solution faintly acid with sulphuric acid and containing a little sulphurous acid. The presence of the sulphurous acid caused a co-precipitation of sulphur in such a form that the whole precipitate was almost granular and could be readily filtered and washed with cold water.

Mr. J. M. WILKIE said that he was somewhat astonished that the electrolytic methods had been neglected by the authors on account of the superior rapidity of the chemical ones, and recalled the work of Dr. H. J. S. Sand on the rapid determination of metals by the method of graded potential and rapidly rotating electrodes. With regard to the use of sodium fluoride in the iodometric determination of copper to avoid interference by ferric salts, it was evident that the action could only be accounted for by the non-ionisation of the complex sodium ferric fluoride. This non-ionisation of the soluble fluorides was very interesting from the theoretical standpoint; it was necessary to bear in mind that phosphoric acid possessed a somewhat similar action, so much so that in the iodometric determination of ferric salts a much higher concentration of hydrochloric or sulphuric acid was required in the presence of appreciable amounts of phosphoric acid to produce any coloration. The effect of adding phosphoric acid to the coloured solution of ferric chloride was quite marked, for the solution became colourless. The rapidity of the thiosulphate method for the precipitation of copper sulphide with the elimination of hydrogen sulphide, and the physical condition of the copper precipitate obtained were so marked features that it had always surprised him that the method was not more widely used. That the authors adhered to the stannous chloride-mercuric chloride method for the reduction, and subsequent titration with permanganate for the determination of iron, showed that in their opinion this early volumetric method had not been displaced by more recently devised processes. With regard to the determination of ferrous iron by titration with permanganate in the presence of hydrochloric acid, Jones and Jeffrey some years ago claimed that in the presence of Reinhardt's solution (manganese sulphate, sulphuric acid, and phosphoric acid) accurate results were dependent on the rate of addition of the permanganate to the ferrous iron solution. However, later workers had not entirely confirmed this. The iodometric relations of ferrous and ferric salts possessed peculiar features of interest. The authors, as already mentioned, used sodium fluoride when it was desired to determine copper iodometrically, but it was also possible to determine ferric salts iodometrically in acid solution with considerable accuracy in the presence of very varying concentrations of free acid, and finally ferrous salts could be determined iodometrically in the presence of ferric salts by a very convenient method devised by Romijn. The ferrous material was dissolved in *N*/1 hydrochloric acid and sodium pyrophosphate added and then excess of standard iodine. After standing a few minutes the excess was determined by means of standard thiosulphate solution. This method of Romijn was exceedingly useful for the determination of ferric iron in organic materials.

Mr. GRISLEY stated that the estimation of copper in copper pyrites was satisfactorily accomplished by titrating an ammoniacal solution with potassium cyanide, and suggested this method for use in connection with the estimation of the copper present in aluminium alloys.

Mr. COLLITT, in reply, said that they had had no need to use such a mechanical method as the specific gravity determination owing to the rapidity and accuracy with which the iodometric determination of copper could be carried out; it was possible for one operator to perform 30 or more estimations in duplicate during the course of a working day. For a similar reason he had made no attempts along the lines suggested by Dr. Prideaux. For control analysis he did not consider that electrochemical methods for copper and nickel were of any advantage owing to the accuracy and rapidity of the chemical ones, but he added that the one case in which the electrochemical method would probably supersede the chemical one was for the estimation of zinc. He considered that the volumetric estimation of iron employing the stannous chloride method of reduction, was entirely satisfactory, and he stated that it was the standard method in use at the present time in America and Germany for iron ore analysis, and further that probably 75% of the iron ores of the world were valued on the basis of the results obtained by this method.

*Meeting held at University College on Wednesday, March 20th, 1918.*

DR. R. M. CAVEN IN THE CHAIR.

### THE PREPARATION OF "STANDARD" PLANTATION RUBBER: A NEW METHOD OF COAGULATION.

BY M. BARROWCLIFF, F.I.C.

Doubtless owing to conditions imposed by the war, the question of the preparation of a standardised plantation rubber has not, apparently, during the last three years, continued to receive the attention that was formerly being paid to it. That little, if any, real advance has been made towards the solution of the problem is shown by the heavy premium which still persists in favour of Brazilian rubber. At the present time (Feb., 1918), the respective prices of "Fine hard Para" and plantation rubber are 2s. 8d. and 2s. 3d. per lb. When it is remembered that Para rubber shows, on washing and drying, a loss greater by about 12% than does plantation rubber, the premium in its favour is seen to be equivalent to 9d. per lb., or 33%.

The reason for the obvious partiality shown by the manufacturer for Para is that plantation rubber, even of the highest grades, varies in quality, the variations being considerably greater than those found in different samples of fine hard Para. Much experimental work, dealing with the causes of this variability, has been published, notably by B. J. Eaton and J. Grantham. It was found that it is almost solely in respect of the time of heating required to ensure correct vulcanisation, that is, to afford a vulcanised rubber having the best mechanical properties, that the variations occur. Samples of plantation rubber, prepared by the different methods in general use, were found to require for the correct "cure" widely varying times of heating, at a constant temperature. Further, rubber made by the same process, on different estates, and at different times on the same estate, was found also to vary greatly in this respect. When, however, the proper time of heating had been found and applied, relatively



small differences were noted in the mechanical properties possessed by the vulcanised samples.\*

The fundamental fact established by Eaton and Grantham is that the time factor for correct vulcanisation varies inversely according to the degree to which the rubber coagulum has been allowed to remain, subsequent to coagulation, unworked, and in contact with its serum. The longer this period the shorter the length of heating required to produce a correct cure.†

Bearing this in mind, also the fact that the present custom is to machine the rubber on the day following coagulation, the influences exerted by the various factors introduced, in practice, into the manufacture of rubber from latex can be accurately foretold. The greater the extent to which the coagulum is machined, and the more completely the serum is consequently washed out, the slower the rate of "cure" of the rubber produced. This "crêpe" rubber, as now marketed, is the slowest to vulcanise. "Sheet"

the latex (the usual method) is to retard markedly the rate of cure, as will be seen from figures given below. Little difference is occasioned, however, between acid-coagulated rubbers, by variations in the amount of acid that has been used, at all events within the limits met with in practice.

As is obvious from the foregoing, to produce a uniform rubber without some radical change in method would involve almost insuperable difficulties. One type of rubber only could be permitted; whilst present-day smoke houses, drying rooms, and much machinery, would have to be "scrapped," in order that the necessary standardised mechanical treatment could be introduced. The way out of this difficulty is clearly indicated by Eaton and Grantham's further work.

It was found that the change which takes place in the rubber coagulum and causes the diminution in the time of heating required for vulcanisation is completed in the course of a few days. This is shown by the following figures\* :—

	Optimum time of cure, 2½ hrs.	Breaking load, kilos. per sq. mm. (a).	Elongation of break, (original length = 100) (b).	Product (a × b).
<i>Pressed slab.</i>				
Crêped 1st day after coagulation .....	2½	1.14	1012	1153
" 4th day .....	1½	1.50	1038	1557
" 7th day .....	1½—1¼	1.50	976	1464
" 10th day .....	1¼	1.51	1032	1558
<i>Coagulum.</i>				
Crêped 1st day after coagulation ....	2½	1.25	997	1246
" 4th day .....	2	1.59	988	1570
" 7th day .....	1½	1.37	975	1335
" 10th day .....	1¼	1.38	1001	1381

rubber, which receives a much less drastic machining and retains a considerable quantity of serum, that drains out relatively slowly, cures in a much shorter time, whilst "slab," which receives no mechanical treatment and in which 12% to 15% of moisture is allowed permanently to remain, vulcanises still more quickly.

In the preparation of "smoked" rubber two variables are concerned: (a) the action of the smoke products; (b) the temperature of drying. The former retards the rate of cure, whilst the latter, within the limits attained in practice, accelerates it. Hence "smoked" sheet may cure either faster or slower than similar unsmoked sheet, depending on the balance of these factors in the earlier stages of drying.

In the case of unsmoked rubber the rate of drying similarly affects the vulcanisation time factor. The same plantation drying room may produce, on different days, according to the temperature and atmospheric humidity, rubber having different rates of "cure," even if the moisture content of the wet rubber happened, on each occasion, to be the same.

The amount of serum left in "sheet" rubber depends on the machining it has been given. Eaton found that the more concentrated the latex the more rapid is the rate of cure of the "sheet" afforded by it. The coagulum from the undiluted latex being thicker and harder, more serum is retained in it after the "rolling" process; the rate of drying, consequently, is slower than in the case of the thinner sheet afforded by the diluted latex, affecting, in the way expected, the optimum time required for the "cure."‡ Similarly, "differential" rolls and rolls of equal size and speed will produce sheet rubber requiring differing times of "cure," other factors being constant. The effect of employing acetic acid to coagulate

In each case the latex was diluted to the same degree and coagulated with acetic acid. Although the maximum effect appears to be attained about the 7th day, it is seen that there is little change in the rate of vulcanisation after three days, when the vulcanised rubber possesses the best mechanical properties. The slower progress of the change in the coagulum, as compared with the slab, is probably due to putrefactive changes having occurred. To obtain the best results, therefore, the coagulum should be left unworked for three days. It is then immaterial to what extent the rubber is worked, in what form it is finished, or how quickly or slowly it is dried, the time of "cure" not being affected to any significant extent.

If, however, coagulation is carried out in the usual way with acetic acid in open vessels, the rubber coagulum undergoes changes, if kept such a length of time, which render the method unsuitable. Putrefactive action sets in, accompanied by darkening of the coagulum, whilst spot diseases, which have been shown to cause an extraordinary retardation of "cure," are likely to develop during the subsequent treatment.

A new procedure is required, therefore, and seems to be furnished by the method introduced by M. Maude and W. S. Crosse, known as the M.C.T. process.† It is based on the fact that in closed vessels, latex coagulates completely without the addition of acid and without at the same time undergoing putrefactive changes. As now carried out in practice, large cement tanks provided with heavy water-sealed covers are used. These are filled with the bulked latex, leaving the smallest practicable air space. The covers are affixed and sealed and left undisturbed until the following day, when coagulation is found to be complete. The resulting coagulum is converted into "crêpe" in the usual manner.

Addition may be made to each 100 gallons of

\* Agric. Bull. Fed. Malay States, Feb., 1915.

† This Journal, 1916, 723.

‡ Ibid. 1917, 1221.

\* Agric. Bull. Fed. Malay States, Oct., 1916.

† Patented in the Fed. Malay States, Ceylon, etc.

latex of a quantity of a soluble calcium salt, *e.g.*, calcium acetate or sulphate (gypsum), corresponding to 4 oz. of calcium. The effect of this is to accelerate the coagulation. A beneficial effect on the properties of the rubber may also be produced; but evidence on this point is not yet available. In this connection there merits notice the fact that whereas the soils of the Amazon region are well supplied with lime those of the Malay Peninsula are remarkably deficient in this respect. Further,

a pressure of one or two atmospheres, which has the effect of keeping in solution the carbon dioxide of which the bubbles consist. For this purpose it is necessary to use iron pressure vessels provided with partitioned removable aluminium linings.

The following vulcanisation results afforded by rubber prepared by this method are contained in a report by Mr. B. J. Eaton to Mr. Maude, then manager of the Cicely Rubber Estates (April, 1917):—

*Crêpe rubber.*

Source of rubber.	Optimum time of cure.	Breaking load, kilos. per sq. mm. (a).	Elongation at break (original length = 100) (b).	Product (a × b).
<i>Cicely Estate.</i>	hrs.			
Latex from old trees .....	2½	1.37	990	1356
" " " .....	2½	1.30	955	1293
" " " .....	2½	1.32	985	1300
" " " .....	2½	1.34	990	1326
Undiluted latex .....	2½	1.44	989	1424
" " " .....	2½	1.44	990	1425
Bulked latex (old and young trees) ..	2½	1.43	1020	1458
" " " .....	2½	1.39	1008	1401
Undiluted latex .....	2½	1.52	1007	1530
The same, diluted 1:1 .....	2½	1.33	1029	1368
Latex from young trees .....	2½	1.34	1005	1346
" " " .....	2½	1.35	1009	1362
" " " .....	2½	1.41	993	1400
Ratanui Estate latex .....	2½	1.38	1000	1380
" " " .....	2½	1.40	979	1370

\* Soaked before drying in sodium bisulphite solution to produce a pale crêpe.

the Para method of coagulation favours the retention of calcium compounds by the rubber, whilst acid coagulation tends to remove them.

For comparison are given the following standard figures, afforded by good specimens of plantation rubber and fine hard Para.

Source of rubber.	Optimum time of cure.	Breaking load, kilos. per sq. mm. (a).	Elongation at break (original length = 100) (b).	Product (a × b).
<i>Unsmoked sheet.</i>				
<i>Cicely Estate.</i>	hrs.			
Undiluted latex .....	1½	1.34	1028	1377
Diluted latex (1:1) .....	2½	1.41	999	1408
Ratanui Estate latex .....	2½	1.38	960	1224
<i>Smoked sheet.</i>				
<i>Cicely Estate.</i>				
Undiluted latex .....	2	1.51	1014	1531
" " " .....	2½	1.45	979	1419
Diluted latex (1:1) .....	2½	1.38	934	1358
Ratanui Estate latex .....	2½	1.47	967	1421
<i>Slab rubber.</i>				
<i>Cicely Estate.</i>				
Undiluted latex .....	1½	1.50	960	1440
" " " .....	1	1.45	1005	1457
" " " .....	1	1.47	978	1437
Diluted latex (1:1) .....	1½	1.50	962	1443
Ratanui Estate latex .....	1½	1.45	978	1418
" " " .....	1½	1.30	940	1222

Source of rubber.	Optimum time of cure, hrs.	Breaking load, kilos. per sq. mm. (a).	Elongation at break (original length = 100) (b).	Product (a × b).
Slab .....	hrs.			
" " " .....	1½	1.51	1032	1558
Sheet (unsmoked) .....	2½	1.52	960	1473
Sheet (smoked) .....	2½	1.46	973	1420
Thin crêpe .....	3½	1.30	930	1209
Fine hard Para .....	2½	1.52	1034	1571

These points may be not without bearing on the causes of such differences as may exist between Para and plantation rubber.

For the manufacture of "sheet" the tanks are suitably partitioned. "Sheet" rubber so made contains usually a number of small bubbles. This can be obviated, if desired, by coagulating under

Regarding these results, Eaton states: "All the samples possess excellent tensile properties, equal to the best samples of plantation sheet and crêpe, and prove that the process is capable of producing rubber of excellent quality." It is to be noted that all the above rubber was manufactured within 24 hours of coagulation. This



accounts for the difference in the rate of cure shown by the three types, slab, sheet, and crêpe.

The uniformity of the samples of crêpe rubber is remarkable, considering that they represent the produce of old and of young trees; of pure latex and latex diluted with water; of rubber made on different days. The rate of cure is seen to be more rapid than in the case of the standard sample of crêpe, a result attributed by Eaton to the absence of acid.

The above figures are given in order to show that this method of coagulation affords, at least, rubber equal in all respects to the best now being produced by acid coagulation, and of greater uniformity. The point upon which stress requires to be laid, however, is that it is a process, and seemingly the only practical one, by which the coagulum may be allowed to mature for the length of time essential for eliminating the differences that otherwise result from unstandardised subsequent treatment.

For the production of "standard" rubber the bulked latex from a day's tapping requires to be placed in the coagulating tanks, as described above, and allowed to remain undisturbed for a fixed time, which, it is suggested, should be three days. Under these conditions no putrefaction occurs and the coagulum remains perfectly white, except for a slight surface darkening.

Judging by the considerations advanced above the rubber so produced, whatever the source, or whether finished as crêpe or sheet, would show no variations in the time of heating required under standard conditions for correct cure, whilst the vulcanised material would possess the maximum attainable tensile strength. The cure would be substantially quicker than that of Para under similar conditions, but this is an advantage as it enables the manufacturer, whilst heating for the same length of time, to vulcanise at a lower temperature, which is all to the benefit of the finished material.

The result of the general adoption of the above described *modus operandi* would be the production of a plantation raw rubber as uniform in its vulcanising properties as fine hard Para, and one affording at least an equally satisfactory vulcanised product.

## Sydney Section.

Meeting held on Wednesday, November 21st, 1917.

### ON THE RELATION BETWEEN THE DEGREE OF SUPERSATURATION, THE REFRACTIVE INDEX, AND THE TEMPERATURE OF SUGAR SOLUTIONS.

BY E. V. MILLER AND F. P. WORLEY, M.A., D.SC.

In the industrial operation of crystallising sugar from solution, by evaporation in a vacuum pan, the temperature of evaporation is intentionally varied at different stages of the process; but notwithstanding this variation the solution must of course be kept always in a supersaturated condition, otherwise the process of crystallisation would be stopped or reversed. It would be of assistance to the operator to know at any moment the degree of supersaturation of the solution in which the crystals of sugar were forming or growing. In the absence of any direct method of expeditiously determining the degree of supersaturation, an indirect method has been devised. An instrument, to be described in a subsequent paper, has been constructed by one of us which will at any

time show at a glance the refractive index of the liquid portion of the contents of the vacuum pan. The temperature is easily ascertained. From these data the supersaturation may be inferred, provided certain relations existing between temperature, refractive index, and degree of supersaturation of sugar solutions are known.

#### Theoretical.

It is necessary to state what is meant by "degree of supersaturation," since this may be defined in a variety of ways. In making a choice among these, regard should be had to the forces operative in inducing crystallisation, and in determining its rate. These are (1) the excess of osmotic pressure over the solution pressure, *i.e.*, its excess over the osmotic pressure of the saturated solution, and (2) the viscous resistance to the movement of the molecules of sugar in the solution. Knowledge of the magnitude of these two forces in a crystallising solution would probably carry the operation of crystallisation completely out of the rule-of-thumb stage in which it exists at present: so that in reality the mere knowledge of the degree of supersaturation existing in a solution is desirable only in so far as it gives some indication of the first mentioned of these forces. In defining, therefore, a scale of supersaturation we should aim at having its degrees as nearly as may be proportional to equal increments of the force which operates in favour of crystallisation. A method of specifying the supersaturation which has considerable vogue is deficient in this important respect. It is a mere numerical co-efficient, having unit value when the supersaturation is zero. It gives no indication of the excess osmotic pressure.

Recent work on the variation of osmotic pressure with concentration, in concentrated solutions, has shown that at constant temperature the pressure varies approximately in proportion to the ratio solute: solvent. In accordance with this law we define  $s$ , the degree of supersaturation, as follows:

Starting with a quantity of saturated solution containing 100 grms. of water, imagine  $s$  grammes of sugar to be dissolved in it, the initial and final temperatures being the same. Then  $s$  will be the degree of supersaturation existing in the solution.

If  $P$  is the percentage of sugar in a saturated solution at a given temperature, and  $S$  the quantity of sugar per 100 of water in the same solution, the relation between these systems of evaluation is given by

$$P = \frac{100S}{100 + S}$$

from which we find

$$S = \frac{100P}{100 - P}$$

or, since  $P$  is a function of the temperature, say  $F(t)$ , which has already been determined,

$$S = \frac{100F(t)}{100 - F(t)}$$

In a solution supersaturated to  $s$  degrees the sugar per 100 of water will amount to  $S + s$ , and the percentage of sugar,  $p$ , in it will be given by

$$p = \frac{100(S + s)}{100 + S + s} = \frac{100 + F(t)(100/s - 1)}{1 + 100/s - F(t)/100} \dots \dots \dots (1)$$

It is well known that at a given temperature the refractive index of a sugar solution varies with the concentration. Assuming the fixed temperature to be  $0^\circ \text{C}$ . we may write

$$r = \theta_0(p)$$

where  $r$  is the refractive index,  $p$  the percentage of sugar in solution, and  $\theta_0$  the function for solutions at  $0^\circ \text{C}$ . It is equally well known that the refractive index of a solution of a given concen-

tration decreases with rise of temperature; and it may be that the rate of variation per degree is not the same for different concentrations of solution: for instance, it is not likely to be the same for zero concentration (pure water) as for a concentrated solution. The quantity, then, to be subtracted from  $\theta_0(p)$  of the last equation in order to give the refractive index, not only of any strength of solution but for any temperature above  $0^\circ\text{C}$ ., will be a function of both the temperature and the concentration of solution, and may be written  $\phi_0(t, p)$ , where  $t$  is the temperature and  $\phi_0$  the function; the suffix indicates that it is appropriate to the  $\theta$  function when the latter relates to  $0^\circ\text{C}$ . For brevity this function will be called the temperature coefficient. We have, then, for any concentration and any temperature,

$$r = \theta_0(p) - \phi_0(t, p)$$

The use of this equation would involve a knowledge of the refractive indices of highly concentrated solutions at  $0^\circ\text{C}$ ., and of their temperature coefficients starting from the same point. It is found, however, impossible to work with concentrated solutions at low temperatures owing to their tendency to crystallise, but they may readily be manipulated through a range of temperature,  $30^\circ$  to  $75^\circ\text{C}$ ., which is important industrially. Within that range, and within the industrially important range of concentrations, 72% to 81% of sugar, they have a temperature coefficient, say  $\phi(t, p)$ , which, it is found, can be expressed (keeping within errors of experiment) in a simpler way than could that of the larger range  $\phi_0(t, p)$  which goes down to zero temperature and zero concentration. This simplified temperature coefficient involves a change in the other function which we will now call  $\theta$ . By properly defining  $\phi$  and  $\theta$ ,  $r$  may be expressed by these functions just as well as by the more complex functions  $\phi_0$  and  $\theta_0$ .

In the equation

$$r = \theta(p) - \phi(t, p) \dots\dots\dots (2)$$

$\phi(t, p)$  is the temperature coefficient of the refractive index correct (within errors of experiment) over a range of temperature  $30^\circ\text{C}$ . to  $75^\circ\text{C}$ . and a range of concentration 72% to 81%, while  $\theta(p)$  is a function of the concentration such that (2) will give the refractive index, as found by experiment, for any solution at any temperature within the range specified.

Since, as we have seen (eq. 1),  $p$  may be expressed in terms of  $t$  and  $s$ , it is obvious, from eq. 2, that  $r$  also may be expressed in the same terms. Of the three quantities in eq. 2, the refractive index and the temperature can readily be ascertained at any time at the vacuum pan, whence the remaining quantity, the supersaturation, may be known. But, for this, it is of course necessary to know the functions  $F(t)$ ,  $\theta(p)$ , and  $\phi(t, p)$ . The first of these has been carefully determined by Herzfeld, hence the object of this investigation was to ascertain the functions  $\theta$  and  $\phi$ .

To determine  $\phi$  it was necessary to ascertain the refractive indices of a solution of known concentration at various temperatures, and then to deal in the same way with solutions of other concentrations covering the range above referred to. From this series of observations  $\phi(t, p)$  was determined. In order to find  $\theta$  it was necessary first to ascertain the refractive index of a solution of known concentration,  $p$ , at a definite temperature, the temperature chosen being about the middle of the range for which the temperature coefficient for that concentration of solution had been ascertained. If  $r_1$  is the index thus found we have, from (2)

$$\theta(p_1) = r_1 + \phi(t, p_1) \dots\dots\dots (3)$$

This gives us one value of  $\theta(p)$ , viz., that for  $p_1$ . By making similar determinations with other concentrations the function  $\theta(p)$  was found.

It was thought that one set of readings for each

solution of different concentration should furnish data to derive both the functions. But it was found to be impossible to get thick solutions so homogeneous that a single sample placed in the refractometer could be relied upon accurately to represent the bulk. This did not matter so far as the determination of the temperature coefficient was concerned, for the results showed that the influence of concentration was trifling in comparison with the influence of temperature, and an error of even  $\frac{1}{2}\%$  in the concentration would not be serious. But in determining the  $\theta$  function accurate knowledge of the concentration was required. The investigation was therefore divided into two parts: (1) the determination of the temperature coefficients by placing in the refractometer one sample of each solution, and taking readings of its index at different temperatures; and (2) the determination of  $\theta(p)$  by placing in succession in the refractometer, maintained at constant temperature, several samples of a solution of known strength, taking the mean of the index readings to be the true index, and repeating this process with solutions of various strengths.

#### Experimental.

An Abbé refractometer with jacketed prisms was used, the readings of which refer to the D radiation. The prisms were maintained at the required temperature by the circulation through their jackets of water from a thermostat.

*The thermostat.*—For a full description of this apparatus reference may be made to a paper by one of us on "Studies of the Processes Operative in Solutions, XXII.," Proc. Roy. Soc., A. 87, 1912.

As several alterations had to be made to fit the apparatus for the higher temperatures required in this investigation a brief description will be of use. The thermostat consisted of a lagged cylindrical tank holding about 60 litres of water, fitted with a stirrer and a rotary pump to circulate the water anywhere outside the vessel. Both these were driven by electric motor. This outside circuit consisted of the prism jackets of the refractometer and a glass bottle with inlet and outlet tubes so arranged that the circulation could be viewed. A thermometer was placed in the tank for convenience, though the temperatures recorded were those of the thermometer in the prism jackets. The heat required to maintain the temperature of the water was provided by an electric radiator lamp, taking 250 watts, immersed in the water. At the higher temperatures the supply of heat was augmented

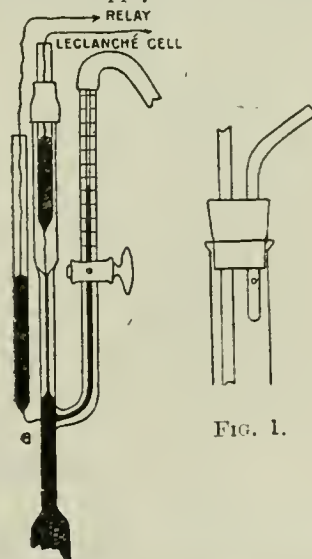


FIG. 1.

FIG. 2.



by a Bunsen burner applied to a spiral copper tube through which water from the tank was caused to circulate by means of the pump. The regulation of the temperature was effected by the automatic making and breaking of the electric circuit containing the radiator lamp at intervals the length of which was controlled by the mercury regulator shown in Fig. 2, the lower part of which was immersed in the water of the tank. The side tube of the regulator is graduated with a temperature scale, so that when the level of the mercury is adjusted to a definite degree on this scale, the other level of the mercury will be at a convenient height in the finer capillary tube when the water in the tank has reached that temperature. Fine adjustment can then be made by raising or lowering the platinum wire in the capillary. This form of regulator can be rapidly and accurately set so as to act at any required temperature. The platinum wire in the capillary, and the other one sealed into the tube at *c*, were connected through a Leclanché cell and a Post Office relay of high resistance. As less than a milliampère goes through this circuit the surface of the mercury in the capillary does not foul. The relay does not directly put the radiator lamp, which takes its current from the mains, in and out of action, but does so indirectly by means of another circuit in which a second relay, and certain branches and resistances, are arranged so as to prevent sparking in the P.O. relay, the final result being that when the mercury touches the platinum wire the radiator lamp goes out, and when it recedes from the wire the lamp lights up. The action of the regulating system is very reliable; it keeps a steady temperature well within 0.05° C. at the highest temperatures employed. Paraffin wax was put on the water in the tank to prevent evaporation. When it was desired to change the temperature quickly, the tank was partially emptied and then filled again with cold water, or with boiling water, and the regulator re-adjusted.

*Preparation of the solutions.*—The sugar used was made at the Chelsea sugar refinery, Auckland, New Zealand, from specially pure material. The crystals were well formed, and after effective washing with water in a centrifugal to remove all traces of mother-liquor, and after a preliminary drying, they were ground and passed in succession through gauze sieves of 30 and 36 holes to the linear inch; the material passing through the former and caught by the latter was used for the investigation. Before making a solution a quantity of this selected sugar was dried *in vacuo* at 100° C. The amounts of sugar and water necessary to make a solution of a desired concentration were weighed into a round-bottom flask of 160 c.c. capacity fitted with a two-holed indiarubber stopper. Through one hole was inserted a thermometer, the bulb reaching nearly to the bottom of the flask, and through the other a bent glass tube sealed at its lower end, but having a small hole in its side, as shown in Fig. 1. The stopper having been inserted, the pressure in the flask was rapidly reduced to one or two inches of mercury, and the valve tube raised so as to close the flask and retain the vacuum. This method of closing the flask left no cavities in which solution could lodge. It was ascertained that the process of evacuation did not, in the time, remove an appreciable amount of vapour from the water which had been weighed in. The sugar was dissolved in the water by heating the flask in a water bath, or, in the case of a concentrated solution, in a brine bath, at the same time keeping it in motion. To dissolve the sugar the contents of the flask were maintained some ten or fifteen degrees above saturation temperature. The solution was always milky at first owing to minute air bubbles, but boiling for a few seconds removed these completely. Brisk boiling was induced by pouring cold water on the upper part of the flask.

From this point the processes for determining the  $\theta$  and  $\phi$  functions differed.

*Determination of temperature co-efficient.*—The flask was now removed from the bath and the contents well mixed by alternately inverting the flask and giving it a circular motion. Air was then admitted by means of the valve tube, and the flask again closed. The stirring was continued for a short time, care being taken to trap no air bubbles, for these would interfere with the indications of the refractometer. The thermometer was then withdrawn from the stopper and quickly replaced by a previously cleaned and dried wash-bottle exit tube, but with no constriction at the end. The valve tube was then pushed down and used as a mouth tube, the flask being thus fitted like a wash bottle. Some of the solution was ejected into a receptacle immediately beside the open refractometer prisms, which were being maintained at the required temperature by the circulation through their jackets of water from the thermostat, and when about a quarter of the solution had come through, the stream was directed for an instant on to the lower prism, which was then immediately closed. If no bubbles were observed in the sample between the prisms the rest of the solution was dispensed with, and readings taken.

*The readings.*—The first temperature for which readings were taken was usually in the vicinity of the saturation temperature, and was the highest desired for the particular solution in the refractometer. After sufficient length of time to insure that the solution had attained the required temperature, the readings were taken. As there were two observers, the personal error was eliminated as far as possible by each making a series of five adjustments of the refractometer field without knowing the readings, which were made and recorded by the other observer. The average of these readings was the value adopted. The temperature was then lowered and another set of readings taken. After the lowest temperature required had been reached, the temperature was raised and readings taken at one or more points near previously observed temperatures. If the rising-temperature and falling-temperature observations proved consistent, *i.e.*, fell on the same curve when plotted, this was taken as an indication of the absence of crystallisation, and that sufficient time had been allowed for the prisms and solution to attain the temperature of the water in the jacket.

*Determination of  $\theta(p)$  function.*—The procedure for the determination of  $\theta(p)$  was somewhat different. The difficulty of getting concordant results from different samples of the same solution necessitated special precautions being taken. After the solution had been prepared, the flask was placed in a wire frame which could be rotated in an air bath in such a way that the flask was inverted during each revolution of the spindle. The bath was maintained at a temperature a few degrees above the saturation temperature of the solution. After thorough mixing in this way the flask was taken out of the bath, air admitted to it, the thermometer withdrawn and replaced by a wash-bottle tube, rather long in the bent portion, and without constriction at the end. A small quantity of the solution was ejected by operator A into a receptacle beside the open prism, and the stream then directed for an instant on to the prism, which was immediately closed by operator B. In the meantime, operator A had kept the stream going very slowly until B had time to insert into the open end of the tube a previously prepared indiarubber plug. The flask was then replaced in the air bath until another sample was required. For receiving the sample the refractometer was placed so that the clear glass surface of the refracting prism was uppermost, and the sample

fell immediately on this; and not, as is usual, on the ground surface of the other prism. In this way the surface of the solution which was to form the interface between glass and solution was exposed to the air for the least possible time. After a set of readings by the two operators, as before described, the prisms were thoroughly cleaned and another sample from the flask placed in the instrument. The procedure was the same as before, except in one particular. Operator A removed the flask from the air bath and created a slight pressure in the flask by means of the mouth tube, whilst operator B cut about half an inch off the end of the exit tube, so as to expose a clean place to the issuing liquid. Each subsequent sample was taken in this way. All samples from one solution were brought to the same temperature in the refractometer before readings were taken. This temperature was usually ten to fifteen degrees below the saturation temperature.

**Corrections.**—The thermometer entering the prism jacket, on which the temperatures were read, was standardised by comparison with a standard thermometer. The two were placed with their bulbs together, the standard being vertical and the other horizontal as it is in the instrument, in a glass vessel through which water from the thermostat was made to circulate; a series of readings of both thermometers was taken.

The refractometer was tested several times during the course of the investigation by using the glass test plate supplied with the instrument. No variation was detected. No correction has been made for the varying temperatures of the glass prisms.

Since sugar and water were weighed into the flask at room temperature, while the samples for the refractometer were taken from the flask when it was a few degrees above the saturation temperature of the solution, the vacant space in the flask would then contain vapour which must have been taken from the solution. It is estimated that the maximum correction to the concentration on this account would not exceed 0.03% of sugar in solution.

A correction operating in the opposite direction is that for buoyancy of the air in weighing; the maximum effect here being 0.01%. Both these corrections have been neglected.

### Results.

As before mentioned the investigation was divided into two parts, the first of which had for aim the determination of the temperature coefficient of the refractive index. A sample of the results obtained in determining this function is shown in the following table:—

*Concentration of solution taken, 78.37% sugar.*

Temperature.	Index found.	Calculated.	Difference.
73.3° .....	1.47486	1.47489	0.00003 +
60.7° .....	1.47801	1.47793	8—
48.5° .....	1.48063	1.48066	3+
61.4° .....	1.47783	1.47778	5—
73.5° .....	1.47500	1.47507	7+

The last two readings were taken after the solution had reached its lowest temperature and had been re-heated.

On plotting the values found (column 2), the index of refraction was seen to be, within errors of experiment, a linear function of the temperature for this range of temperatures. The formula which best fits the results is

$$R = 1.49150 - 0.0002235t$$

where  $R$  is the refractive index for this particular concentration of solution, and  $t$  the temperature. The figures in column 3 are derived from this formula, and column 4 gives the differences between the calculated and experimental values.

Results for nine different concentrations of solution were tabulated in a similar way. In every case a linear function satisfactorily represented the relation between temperature and index, and an empirical formula was found for each strength of solution. The differences between the indices calculated from these formulæ and the values actually found were of the same order as those shown in the above example. Fifty-two observations, each one the mean of ten readings, as before explained, were taken in all; and in only two cases did the difference between the observed and calculated values reach the fourth decimal place, the larger of the two differences being 0.00024. We think this affords evidence that the Abbé refractometer is capable of a high degree of accuracy if used in the manner above described.

The relations between temperature and index of refraction for different strengths of solution, as found experimentally, are plotted in Fig. 3, where the numbers correspond to those in the first column of the table below. The lines drawn in the

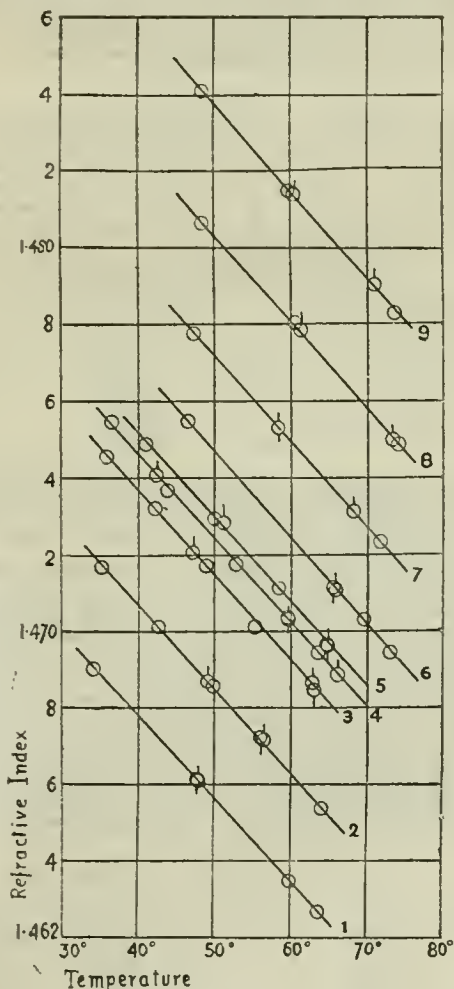


FIG. 3.

figure correspond to the empirical formulæ found. The circles with strokes pointing upwards indicate observations taken on re-heating the solution; the



circles without strokes, and those with strokes pointing downwards, indicate observations taken before re-heating.

To arrive at the temperature coefficient it is not necessary to know the constants in the empirical formulae, and indeed these constants could not be relied upon since the exact concentrations of the solutions were, for reasons already given, doubtful. The following table gives the approximate concentrations,  $p$ , of the nine solutions dealt with; and the "coefficient of  $t$ " found for them. The meaning of this last expression will be clear on referring to the last equation; it is the co-efficient of  $t$  for a particular strength of solution, not that for any strength  $-\phi(p, t)$ .

No.	$p$ .	Co-eff. of $t$ .
1.....	72.5	0.000216
2.....	73.5	218
3.....	74.8	2203
4.....	75.0	2183
5.....	75.4	218
6.....	76.3	2217
7.....	77.0	2197
8.....	78.4	2235
9.....	79.5	2252

On plotting this relation it is found to be linear, within errors of experiment, and to be given by coeff. of  $t=0.0001241+0.000001267p$  which gives, as the temperature coefficient of the refractive index,

$$\phi(p, t) = (0.0001241 + 0.000001267p)t \dots (4)$$

*Estimation of  $\theta(p)$ .*—Knowing the temperature coefficient from eq. 4 we can, with the help of eq. 3, find  $\theta(p)$  by carefully determining the refractive indices of a number of solutions of different concentration, each at a different temperature. The results obtained for one of these solutions are given in the following table:—

*Strength of solution, 74.55%.*

Temperature, °C.	Index found.
50.3 .....	1.47056
50.3 .....	1.47049
50.3 .....	1.47015
50.3 .....	1.47071
50.3 .....	1.47064
Mean	1.47051

The temperature coefficient at 50.3° C. is, from eq. 4, 0.0002185. The value of  $\theta(p)$  for  $p=74.55$  will, therefore, be given by  $\theta(p)=1.47051+(0.0002185 \times 50.3)=1.48150$ .

Each of the observations recorded in the above table is of a different sample of the solution, and is the mean of ten readings as already described. The discrepancy between the highest and lowest of these observations is 0.00056, a figure much beyond the instrumental or personal error, and arises from differences in the concentration of different samples which persisted in spite of the endeavours made to obtain a homogeneous solution. The following table gives the values of  $\theta(p)$  obtained for ten different solutions, the number of separate samples observed in the case of each solution, and the maximum discrepancy between observations:

$p$ .	$\theta(p)$ from exp.	No. of obs.	Max. discrep.	$\theta(p)$ from plot.	Diff.
72.51	1.47605	4	0.00061	1.47604	0.00001—
73.52	1.47858	4	26	1.47870	12+
74.55	1.48150	5	56	1.48141	9—
75.46	1.48378	5	60	1.48380	2+
75.27	1.48603	6	88	1.48593	10—
77.54	1.48953	5	113	1.48927	26—
77.55	1.48896	4	58	1.48930	34+
78.41	1.49147	4	37	1.49156	9+
79.47	1.49433	4	41	1.49435	2+
80.54	1.49729	5	98	1.49716	13—

The figures in the fifth column are calculated from the following empirical equation which was found to fit the results:—

$$\theta(p) = 1.28534 + 0.00263p \dots (5)$$

and the numbers in column 6 show the differences between the values derived, as before explained, from experimental results and those derived from the above equation.

In Fig. 4 the numbers in column 2 are plotted against percentage of sugar; the line represents the relation expressed by eq. 5.

The refractive index of any sugar solution between the before-mentioned limits of concentration and of temperature is, then, given by

$$\begin{aligned} r &= 1.28534 + 0.00263p - \\ &\quad (0.0001241 + 0.000001267p)t \\ &= 1.28534 - 0.0001241t + \\ &\quad (0.00263 - 0.000001267t)p \dots (6) \end{aligned}$$

where  $r$  is the refractive index,  $p$  the percentage of sugar in solution, and  $t$  the temperature in Centigrade degrees.

The following table will facilitate the computation of refractive indices from this equation. To find the refractive index of a sugar solution between 72% and 81% concentration at a given temperature, multiply the % concentration of the solution by the "coefficient of  $p$ " for that temperature and add the product to the figure in line with that temperature in the second column. For example, if we have a 75.6% solution at 41° C. the refractive index will be  $1.28026 + (75.6 \times 0.002578) = 1.4752$ . The decimal places have been extended for purposes of calculation, but in the result no figures after the fourth decimal are reliable.

*Table to be used with equation 6.*

Temp. °C.	Value of 1.28534 — 0.0001241t	Co-efficient of $p$ .
34 .....	1.28112	0.0025869
35 .....	1.28100	0.0025857
36 .....	1.28087	0.0025844
37 .....	1.28074	0.0025831
38 .....	1.28062	0.0025819
39 .....	1.28050	0.0025806
40 .....	1.28038	0.0025793
41 .....	1.28026	0.0025781
42 .....	1.28013	0.0025768
43 .....	1.28000	0.0025755
44 .....	1.27988	0.0025743
45 .....	1.27975	0.0025730
46 .....	1.27963	0.0025717
47 .....	1.27950	0.0025705
48 .....	1.27938	0.0025692
49 .....	1.27925	0.0025679
50 .....	1.27913	0.0025666
51 .....	1.27900	0.0025654
52 .....	1.27888	0.0025641
53 .....	1.27876	0.0025628
54 .....	1.27864	0.0025616
55 .....	1.27851	0.0025603
56 .....	1.27839	0.0025590
57 .....	1.27826	0.0025578
58 .....	1.27814	0.0025565
59 .....	1.27801	0.0025552
60 .....	1.27789	0.0025540
61 .....	1.27776	0.0025527
62 .....	1.27764	0.0025514
63 .....	1.27751	0.0025502
64 .....	1.27739	0.0025489
65 .....	1.27726	0.0025476
66 .....	1.27714	0.0025464
67 .....	1.27702	0.0025451
68 .....	1.27690	0.0025438
69 .....	1.27677	0.0025426
70 .....	1.27665	0.0025413
71 .....	1.27652	0.0025400
72 .....	1.27640	0.0025388
73 .....	1.27628	0.0025375
74 .....	1.27616	0.0025362

To express the refractive index in terms of temperature and supersaturation we have to replace

p in the last equation by its value as given in equation 1, and we then have

r = 1.28531 - 0.0001241t + 100 + F(t)(100/s - 1) / 1 + 100/s - F(t)/100 (0.00263 - 0.000001267t).. (7)

where F(t) = 64.1835 + 0.13477t + 0.0005307t^2 as found by a careful determination by Herzfeld (Z. ver. deuts. Zuckerind., 42, pp. 181, 232).

sugar in the saturated solutions: this table is calculated from the last equation. Its use may be illustrated as follows: If a sugar solution at 145° F. is found to have a refractive index of 1.4727, then it is supersaturated to the extent of 30°.

The research described in this paper was carried out at Auckland University College, Auckland, New Zealand.

% Sugar.		Refractive indices.											
° F.	s = 0.	s = 0.	s = 10.	s = 20.	s = 30.	s = 40.	s = 50.	s = 60.	s = 70.	s = 80.	s = 90.	s = 100.	s = 110.
110 ....	71.02	1.4628	1.4650	1.4660	1.4688	1.4706	1.4723	1.4739	1.4754	1.4769	1.4783	1.4796	1.4809
115 ....	71.53	4636	4656	4675	4693	4710	4727	4742	4757	4771	4785	4798	4810
120 ....	72.04	4643	4662	4681	4698	4715	4731	4746	4760	4774	4787	4800	4812
125 ....	72.56	4650	4669	4687	4704	4720	4735	4750	4764	4777	4790	4802	4813
130 ....	73.09	4658	4676	4693	4709	4725	4740	4754	4767	4780	4792	4804	4815
135 ....	73.63	4666	4683	4699	4715	4730	4744	4758	4771	4783	4795	4806	4817
140 ....	74.18	4674	4690	4706	4721	4735	4749	4762	4774	4786	4798	4809	4819
145 ....	74.74	4682	4697	4713	4727	4741	4754	4766	4778	4790	4801	4812	4822
150 ....	75.30	4690	4705	4720	4733	4746	4759	4771	4783	4794	4804	4815	4824
155 ....	75.87	4698	4713	4727	4740	4752	4764	4776	4787	4798	4808	4818	4827
160 ....	76.45	4707	4721	4734	4746	4758	4770	4781	4792	4802	4812	4821	4830
165 ....	77.04	4716	4729	4741	4753	4765	4776	4786	4796	4806	4816	4824	4833
170 ....	77.64	4725	4737	4749	4760	4771	4782	4792	4801	4811	4820	4828	4836

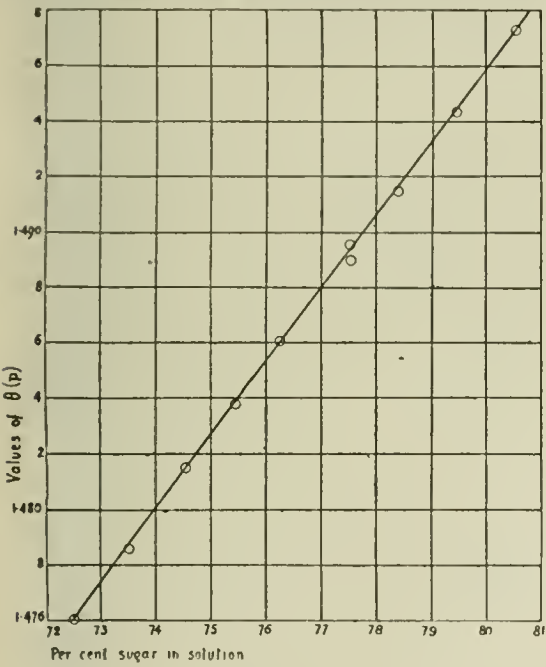


FIG. 4.

In eq. 7 are given the relations which it was the object of the investigation to determine.

Since the Fahrenheit scale is used in British sugar factories it is convenient to express the results in that scale. The relation between the scales is given by t = 5u/9, where t = centigrade degrees, and u = Fahrenheit degrees in excess of 32. Herzfeld's relation then becomes f(u) = 64.1835 + 0.07487u + 0.0001638u^2, and eq. 7 becomes

r = 1.28531 - 0.00006894u + 100 + f(u)(100/s - 1) / 1 + 100/s - f(u)/100 (0.00263 - 0.000000704u).

Appended is a table giving refractive indices for steps of 5° temperature and 10° in supersaturation, with an added column giving the percentage of

Communication.

THE MANUFACTURE OF NITRIC ACID FROM NITRE CAKE.

BY DR. J. GROSSMANN.

Dr. GROSSMANN writes as follows in reply to Captain Goodwin's criticism (this J., 1917, 1161) of his paper on the manufacture of nitric acid from nitre cake (this J., 1917, 1035):—

Captain Goodwin has failed to understand that in the working of the two patent processes which are described in my paper, no agitation or stirring is required; they are baking operations which can be carried out in any shallow muffle. The use of mechanical stirring apparatus would only add to the cost without improving the efficiency of my processes, as the latter give quantitative results without stirring.

I have dealt with nitre cake from different works, and so far as my experience goes it varies in its content of free sulphuric acid between 27 and 33%. Though difficult in laboratory experiments, it will be an easy matter on a large scale to adjust the mixture of nitre cake and sodium nitrate in such a manner as to yield a saltcake containing less than 2% of free acid. The assumption that the mixture containing coke might lead to fires is quite erroneous, as is the statement that there is no advantage in adding carbonaceous matter. The two processes, i.e., the one with and the other without the addition of carbonaceous matter, are designed to suit different purposes. The condensation of the nitrous gases can be effected in such manner as to add very little to the cost, certainly not more than a small fraction of what a mechanical furnace such as Captain Goodwin recommends would cost in material, working, and upkeep.

I agree that saltcake has not always been profitably saleable; but Captain Goodwin appears to have overlooked the fact that in the manufacture of hydrochloric acid from nitre cake nearly 3 tons of saltcake is produced against one ton of saltcake produced in the manufacture of nitric acid by my process, so that the manufacturer of hydrochloric acid would be far more seriously handicapped by a slump in saltcake. By the adoption of my process in nitric acid making, nitre cake would be produced only as an intermediate product



in like manner as it has always been an intermediate product in the manufacture of saltcake in connection with the Leblanc process, and thus cease to be a marketable commodity for the manufacture of hydrochloric acid.

It is quite fallacious to represent the reactions between nitre cake and sodium nitrate, and nitre cake and sodium chloride, as if they were technically analogous. Complete decomposition in a mixture of nitre cake and sodium nitrate takes place at a temperature below  $250^{\circ}\text{C}$ . and is assisted by the fact that sodium nitrate melts at  $330^{\circ}\text{C}$ ., whereas a mixture of nitre cake and sodium chloride requires a temperature of nearly  $500^{\circ}\text{C}$ . for its decomposition, *i.e.*, a temperature which is still  $350^{\circ}\text{C}$ . below the melting point of sodium chloride. It is therefore evident that sodium nitrate is technically more amenable to treatment with nitre cake than sodium chloride; and whilst the working costs are higher in the production of hydrochloric acid, the absolute profit, owing to the low intrinsic value of hydrochloric acid, is less than in the production of nitric acid. The present absurdly inflated price of hydrochloric acid is no legitimate basis on which to form estimates of profit.

I cannot agree with Captain Goodwin's remark "that my process might be remunerative only whilst the war lasted." I have shown that under any conditions processes for the manufacture of hydrochloric acid from nitre cake are technically

and commercially at a disadvantage; and it may be safely predicted that after the war, when competition will be the determining factor in manufacturing, any process which will enable the manufacturer to produce nitric acid cheaper will be found acceptable.

As regards present conditions I may say that my aim in working out my processes has been to stop the great waste of sodium sulphate and sulphuric acid which takes place when nitre cake is used in the place of weak acid; and the still greater waste where large quantities of nitre cake are lying useless. All this waste entails additional importation of large quantities of pyrites from Spain and consequent loss of gold, foodstuffs, and shipping room to this country—matters which will be serious for years after the war and which should be taken in hand in time by the Government and properly dealt with.

The rational utilisation of nitre cake is not only a chemical but an economic problem of national importance, and its solution will only lie in methods which effect a commensurate saving in the use of pyrites. Most of the methods used at the present time are makeshifts which in the fourth year of the war should be overhauled. But unless the Government recognises this there is no inducement in the present times to manufacturers to take up new processes on these lines, however profitable they might be.

## Birmingham Section.

Meeting held at Birmingham University on Thursday,  
February 14th, 1918.

DR. R. S. MORRELL IN THE CHAIR.

## STUDIES IN NITRIDE FORMATION.

BY E. B. MAXTED, PH.D., B.SC.

The general chemical inertness of free nitrogen under ordinary conditions lends a peculiar interest to the study of the methods which must be employed in order to induce nitrogen to combine directly with other elements. Broadly speaking, and omitting for the time being any consideration of a few nitride-forming elements such as lithium, nitrogen only becomes active at moderate temperatures in the presence of a catalyst. Two industrial reactions, at least, take advantage of this property, namely the synthesis of ammonia by the Haber process and the direct formation of cyanides by interaction of alkalis, carbon, and nitrogen, the catalyst usually employed being in each instance iron.

Nitrogen, however, acquires considerable chemical activity at high temperatures, possibly owing to its dissociation, the compounds formed being in some cases capable of stable existence throughout the entire range of temperature between the temperature of formation and room temperature, while in others the product is only obtained at the ordinary temperature by cooling sufficiently quickly through an intermediate temperature zone in which the compound is liable to decomposition. The commercial formation of nitric oxide offers an example of this method of working, it being found necessary, in order to obtain optimum yields of nitric oxide, to "freeze" the equilibrium by cooling quickly from the temperature of the high tension arc to 1000° C., below which temperature nitric oxide is able to exist in a state of false stability.

Nitric oxide is, however, peculiar in properties by reason of the slowness with which it decomposes even at high temperatures, and in the case of other nitrogen compounds a far more rapid form of cooling is necessary in order to recover at ordinary temperatures the compounds formed for instance at the temperature of the arc.

I propose to consider in the present paper firstly an interesting aspect of the formation of ammonia from mixtures of nitrogen and hydrogen. It is well known that by the action of a catalyst at temperatures varying from 400° to 800° C., small amounts of ammonia are obtained at atmospheric pressure; further, that the yield may be increased by working at an elevated pressure, the volume of ammonia formed being found to be, for small percentages, approximately proportional to the pressure.

It is known, however, that ammonia may also be formed, in a similar manner to nitric oxide, by subjecting a mixture of nitrogen and hydrogen to the action of induction sparks, and it was therefore thought interesting to investigate further the possibility of forming ammonia thermally.

The subject has been examined experimentally in the work about to be described, and it will be shown that yields of ammonia amounting to over one per cent, by volume for atmospheric pressure may be obtained thermally by sufficiently intense cooling of mixtures of hydrogen and nitrogen from very high temperatures to the temperature of the room. The result is of special interest if one considers that such a result is only obtainable otherwise below 350° C., a temperature at which ammonia formation, even in presence of active catalysts, does not take place with measurable velocity.

Before passing to a description of the methods employed I propose to deal very briefly with the

theoretical aspect of ammonia formation at high temperatures.

All the formulae which are derivable by thermodynamic treatment of the ammonia reaction depend on more or less small differences between the specific heats of nitrogen and hydrogen on the one hand and of ammonia on the other. It is only possible to determine the values of these specific heats, and of their temperature coefficients, at low or at the most at moderate temperatures and to extend by extrapolation these experimental results to higher temperatures, assuming that no latent factors which will cause an increase or decrease in the observed temperature coefficients appear with increasing temperature. It is known, for instance, that the specific heat of a gas may be expressed by the general relation,  $C = a + bT + cT^2 + dT^3$  etc., but the coefficients of powers of  $T$  higher than  $T^2$  are too small to be determined at temperatures for which the specific heat is capable of direct measurement. It is, however, just these higher powers of  $T$  which will become, with increasing temperature, more and more important factors in the determination of the equilibrium ammonia content. Assuming a linear temperature coefficient  $a + bT$  for nitrogen and hydrogen and a coefficient of the form  $a + bT + cT^2$  for ammonia, this being merely the mathematical expression of experimentally observed results, the following three alternative equations are obtained, in which—

$$K = \frac{P_{\text{NH}_3}}{P_{\text{N}_2}^{\frac{1}{2}} \times P_{\text{H}_2}^{\frac{3}{2}}}$$

- (1)  $\log_{10} K = \frac{2215}{T} - 3.625 \log_{10} T + 0.000307T + 0.29 \cdot 10^{-6}T^2 + 4.82$   
 (2)  $\log_{10} K = \frac{2098}{T} - 2.5088 \log_{10} T - 0.0001006T + 0.186 \cdot 10^{-6}T^2 + 2.1$   
 (3)  $\log_{10} K = \frac{2098}{T} - 2.806 \log_{10} T + 0.0000932T + 0.145 \cdot 10^{-6}T^2 + 2.8494$

The first two equations are due to Haber, equation (1) being based on Wüllner's and Wiedemann's values for the specific heat of ammonia and on Thomsen's figures for the heat of formation, while equation (2) is based on Haber's own measurements. The third equation has been derived by the author by independent calculation based principally on Haber's experimental results.

All three equations agree in that they all contain terms which cause the equilibrium content first to sink with increasing temperature, then to pass through a minimum and finally to increase once more, but the calculated position of the minimum and the rate of increase vary with the formula employed for the calculation. On extending these formulae, which all represent more or less correctly the ammonia equilibrium at temperatures usually employed for the synthesis of ammonia, to high temperatures, widely different results are obtained and the discrepancy between Wiedemann's, Wüllner's, and Thomsen's results for the specific heat and for the heat of formation of ammonia on the one hand, and of Haber's measurements on the other, suggest a careful experimental revision of these important quantities.

The following table gives the variation of  $K$  with temperature by each of the formulae quoted.

TABLE I.

Temperature, ° A.	Value of $K \times 10^4$ .		
	By eq. (1).	By eq. (2).	By eq. (3).
1000	5.7	5.7	5.8
2000	0.78	0.26	0.25
3000	3.03	0.28	0.03
4000	152.0	1.5	0.9



The formation of ammonia by the action of sparks on a mixture of nitrogen and hydrogen was first observed by Deville and later studied by Donkin, who showed that such a mixture sparked in an eudiometer over dilute acid gradually disappeared, the formation taking place in presence of sparks only and not by the action of a silent discharge. This method of synthesis appeared to confirm the possibility of forming ammonia thermally, but since it is difficult by means of spark discharge to heat the whole of a definite volume of gas uniformly to a known high temperature before suddenly cooling, and in view of the comparatively low yields—probably due to this—obtained by sparking in similar reactions, for instance in the synthesis of nitric oxide from nitrogen and oxygen, it was not thought to constitute a promising method for the production of maximum quantities of ammonia. I considered it therefore preferable to investigate the formation of ammonia firstly by rapidly cooling mixtures of nitrogen and hydrogen injected into flames, and secondly by rapidly cooling such gas mixtures from the temperature of the high tension arc.

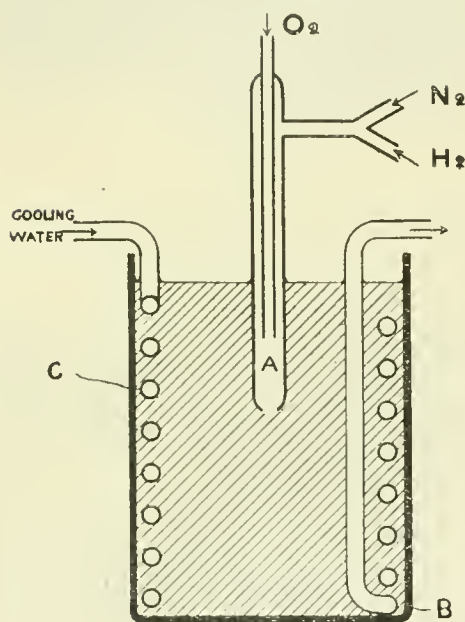


FIG. 1.

Accordingly, working by the first of these two methods, the thermal formation of ammonia has been studied, firstly for not extremely high temperatures, by rapidly cooling mixtures of nitrogen and hydrogen from the temperature of the oxy-hydrogen flame down to room temperature. The method of heating and suddenly cooling con-

sisted in injecting the mixture into an oxy-hydrogen flame burning under water, or, in a second series of experiments, in a water-cooled silica tube, the amount of ammonia being estimated in some cases by direct titration and in others colorimetrically by Nessler's solution. The reaction mixture contained, therefore, in addition to nitrogen and hydrogen, a volume of steam depending on the proportion of the mixture of nitrogen and hydrogen injected into the flame.

It was noted further that the yield of ammonia recovered at ordinary temperature was considerably greater when direct cooling with water was employed than when the flame was cooled by contact with a water-cooled quartz tube.

The apparatus used for the first series of experiments is illustrated in Fig. 1. It consisted of a silica combustion chamber, A, 6.5 mm. in diameter and 7.5 cm. in length, completely immersed in about 900 c.c. of distilled water contained in a glass vessel, B, fitted with an efficient cooling coil, C, through which a rapid current of cooling water was passed, the vessel having a capacity of slightly more than one litre. Into the quartz reaction chamber, nitrogen, hydrogen, and oxygen, at rates determined by velocity meters, were injected and ignited, the flame gases passing out of the open end of the combustion chamber and into the water, by the action of which the ammonia, recovered from the flame by sudden cooling, was retained. In some cases the water was replaced by dilute acid, without, however, any appreciable difference in the results, provided that the water was kept cool. The amount of oxygen passed into the reaction chamber was in every case regulated in such a way as to leave uncombined hydrogen equal to three times the volume of the nitrogen passed in.

In order to obtain a comparison of the results obtained at various temperatures and at varying partial pressures, *i.e.*, by the increasing dilution of the oxy-hydrogen flame with a mixture of nitrogen and hydrogen, advantage was taken of the known fact that for small percentages of ammonia, the volume formed at a given temperature is directly proportional to the pressure, so that the volume of ammonia which would be formed at the temperature of the flame but at atmospheric pressure may be calculated by dividing the value observed by the corresponding partial pressure of the nitrogen-hydrogen-ammonia in the nitrogen-hydrogen-ammonia-steam reaction mixture. In this way it becomes possible easily to compare the relative amounts of ammonia formed at different temperatures and at varying partial pressures. In this series of experiments the ammonia determination was made colorimetrically by Nessler's reagent in the resulting solution in the cooling water, care being taken to use for cooling the flame distilled water free from ammonia. In those cases in which dilute acid was used for cooling, this was made up by dissolving 40 c.c. of ammonia-free N/10 sulphuric acid in each litre of distilled water.

Table II. summarises the results obtained by direct cooling with water or dilute acid.

TABLE II.

Expt. No.	N.	H.	O.	Duration of expt. (mins.).	Vol. % of NH <sub>3</sub> in N/H mixture.	Partial pressure of system N-H-NH <sub>3</sub> in flame.	Estimated flame temp., ° Abs.	Vol. % of NH <sub>3</sub> formed at absolute pressure of 1 atm.
1.	60	220	20	15	0.00235	0.86	1280	0.0027
2.	60	260	40	15	0.005	0.75	1500	0.0067
3.	60	360	90	15	0.013	0.57	1830	0.023
4.	30	270	90	15	0.018	0.4	2120	0.045
5.	20	240	90	15	0.0205	0.31	2260	0.066
6.	20	300	120	10	0.0366	0.25	2350	0.146
7.	20	460	200	5	0.07	0.167	2470	0.42
8.	10	430	200	5	0.112	0.091	2580	1.23

The columns headed N, H, and O represent the velocity in litres per hour of the nitrogen, hydrogen and oxygen passed into the reaction chamber. For experiments Nos. 2, 3, 4, and 5 the cooling agent was water, otherwise dilute sulphuric acid was used.

It will be noticed that the percentage of ammonia by volume in the nitrogen-hydrogen mixture recovered by suddenly cooling (reduced for uniformity to 1 atmosphere pressure) rises from 0.0027 to 1.23 as the reaction temperature approaches that of the oxy-hydrogen flame. The theoretical temperature of the oxy-hydrogen flame calculated by equating the integrated product of the heat capacity of the gases involved, expressed as a function of temperature, multiplied by the temperature, against the heat of formation of water, works out, if dissociation effects be taken into consideration, at something over 3000° C., but in practice, since the complete combination of oxygen and hydrogen is not absolutely instantaneous, radiation phenomena will interfere not only with the rate of fall of the maximum temperature but also with the value of this maximum temperature itself, at any rate as far as the gross maximum temperature of the flame is concerned.

In the absence of sufficient data to calculate the effect of such radiation, I have inserted in the column headed "Flame temperature" values obtained by interpolation from Fery's experimental measurements of the temperature of the oxy-hydrogen and hydrogen-air flames.

#### *Experiments with indirect cooling.*

The reaction chamber taken for this series of experiments consisted of a vertical quartz tube 90 cm. long and 8 mm. in internal diameter. This was cooled by making it the inside tube of a long Liebig condenser. The flame gases, which were in this case cooled only by contact with the cold walls of the silica tube, were allowed to pass from the bottom of the tube through a known quantity of  $N/10$  acid, which was afterwards titrated back with alkali in the usual manner. As was to be expected from the less rapid cooling, the results obtained were at high temperatures less than those obtained by directly leading the flame into cold water. At lower temperatures the divergence became less marked, until, at the lowest temperature studied, approximately equal values for the yield of ammonia were observed. The results obtained by indirect cooling in the above manner are summarised in Table III.

TABLE III.

No. of expt.	Duration in mins.	Partial pressure of $N-H-NH_3$ in flame.	Vol. % of $NH_3$ in $N-H$ .	Estimated flame temp., ° Abs.	Vol. % of $NH_3$ formed at absolute pressure of 1 atm.
1.	30	0.86	0.0026	1280	0.003
2.	30	0.57	0.0068	1830	0.012
3.	30	0.25	0.0134	2350	0.054
4.	30	0.167	0.0196	2470	0.118
5.	30	0.091	0.019	2580	0.21

In the method of sudden cooling above described there is certainly the possibility that ammonia was formed not by direct synthesis but secondarily by the reduction of nitric oxide by the excess of hydrogen, it being well known that mixtures of oxygen and nitrogen at the temperature of the oxy-hydrogen flame give rise to nitric oxide. This secondary formation of ammonia is not, however, probable since, firstly, if such mere reduc-

tion of nitric oxide to ammonia took place at high temperatures, and if the ammonia equilibrium did not have an increased value at such high temperature, the system would pass under conditions in which equilibrium is established practically simultaneously, from an equilibrium state into non-equilibrium, this being contrary to experience, and, secondly, if the reduction took place at low temperatures (below 350° C.), so that the ammonia formed was in equilibrium at the time of its formation, the amount of ammonia formed would be increased by decreasing the rate of cooling, nitric oxide being capable of being recovered practically undecomposed even by less rapid cooling than the slower method used in the above experiments. Such an increase in the amount of ammonia with decreased rate of cooling is in direct opposition to experimental evidence, and in any case the reduction of nitric oxide to ammonia by free hydrogen in the absence of a catalyst does not take place at the temperature (below 350° C.) at which the ammonia observed would be formed in equilibrium, especially in view of the extremely short time for which such temperature persists.

The results obtained would thus appear to form some ground for the view that the ammonia equilibrium at high temperatures increases more rapidly than can be deduced by assuming at such high temperatures a uniform change in the specific heat of ammonia, based on a relation of the simple form  $a+bT+cT^2$  obtained by measurements at low temperatures; moreover the equilibrium capable of being calculated by such low temperature formulae may possibly be affected also by dissociation effects. I am at present examining the formation of ammonia by very rapid cooling of the high tension arc flame burning in nitrogen and hydrogen, the results of which investigation will, if war conditions permit, be published in the near future.

#### *Nitride of iron.*

Nitrides of easily reducible metals possess a special interest technically, since a direct synthesis of such a nitride would afford the possibility of a process for the manufacture of ammonia by alternately treating, firstly, the metallic oxide with a reducing gas containing nitrogen, such as producer gas, and, secondly, treating the nitride thus formed with steam, with formation of ammonia and regeneration of oxide. Examples have already been given of the use of iron for the catalytic activation of nitrogen at moderate temperatures, for instance in the formation of ammonia and of cyanides. It was therefore considered interesting to investigate the possibility of finding conditions for the direct synthesis of iron nitride from iron and nitrogen, the iron acting both as catalyst and as nitride-forming element.

Iron nitride may, as is well known, be readily formed by leading a current of gaseous ammonia over reduced iron at an elevated temperature, preferably at about 460° C. On heating the grey powder thus obtained, either in a vacuum or in an inert gas, decomposition into iron and nitrogen begins at about 500° and is rapid at 600° C.

It should, however, be possible on theoretical grounds to reverse the dissociation represented by the equation  $Fe_3N_2 = 4Fe + N_2$  and thus to effect a direct synthesis of iron nitride, by treating finely divided iron—in the presence of which nitrogen is known to become activated—with nitrogen at a higher pressure than the dissociation pressure of iron nitride and at a temperature just above its normal decomposition point.

The furnace employed for this work has proved of very general utility for experiments at elevated pressures not only with nitrogen but also with other gases, and is therefore illustrated here



(see Fig. 2). It consists of a cylindrical steel vessel of the shape shown, closed by a flanged recessed cover provided with a lead jointing ring fitting into the recess. The electric furnace contained in the steel bomb was fastened rigidly to two short copper rods passing through the cover and insulated from this by the arrangement shown, these rods functioning both as furnace supports and as leads for the heating current. The lower part of the inside tube of the electric furnace contained a short piece of silica tube closed at one end and holding a platinum/platinum-rhodium thermocouple. The ends of this couple were taken round the outside of the furnace and through the cover by way of insulated stuffing-boxes rendered gas-tight by vulcanite fibre. The electric furnace contained, in addition to the pyrometer, the charge of finely divided iron, preceded and followed by rolls of fine copper gauze in order to prevent any possibility of oxidation.

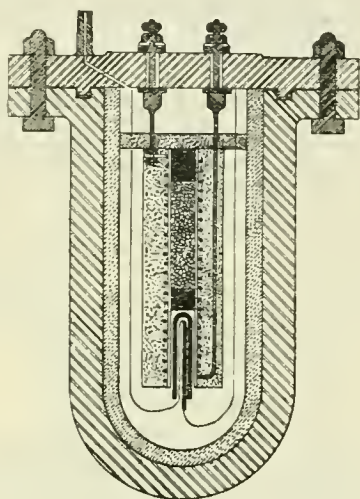


FIG. 2.

Gaseous nitrogen at a high pressure was introduced through the gas inlet, the furnace being immersed in water during each experiment. With the apparatus described I carried out a series of experiments at 100 atmospheres pressure and at various temperatures between 500° and 700° C. In no case was any trace of nitride obtained, showing either that the dissociation pressure of iron nitride lies above 100 atmospheres for the temperature region studied, or that iron itself is not a sufficiently active catalyst for the promotion of the reaction.

In consequence of this result, experiments were begun to measure, if possible, the dissociation pressure of iron nitride just above its normal decomposition point, or at any rate to determine whether this dissociation pressure is greater than that at which a commercial synthesis would be possible, this limit being probably about 200 atmospheres.

The method of investigation employed consisted in preparing a stock of iron nitride by the action of ammonia on iron and studying the pressure which is capable of being developed by the decomposition of this nitride in a closed system of small dimensions. For each experiment about 20 grms. of freshly prepared iron nitride, previously heated for one hour in a vacuum at 200° C., to expel any ammonia, was introduced into a steel tube, 1 cm. in internal diameter and 140 cm. long, provided with

two steel rods each 54 cm. long, loosely fitting into the tube. These steel rods served to fill up dead space on each side of the charge of nitride and also to ram the charge into a compact cake situated in the centre of the tube. This steel tube was closed at one end by a pressure gauge and at the other by a needle valve through which nitrogen could be introduced in order to raise the initial pressure to that required. The apparatus was remarkably gas-tight in that, in spite of its small dimensions, a pressure of, for instance, 200 atmospheres was usually capable of being retained overnight without sinking more than from one to two atmospheres.

In order to raise the nitride to the desired uniform temperature, a piece of 3-in. steel shafting about 15 inches long and bored longitudinally in such a way that the steel tube just slid into it, was slipped over the decomposition tube and the whole heated in a furnace, the temperature being measured by a pyrometer passed into a second hole in the steel shafting.

In order to displace air, nitrogen under pressure was alternately admitted to the system and blown away, this being repeated several times. The nitrogen was adjusted to the initial pressure required for the particular experiment, whereupon the needle valve was closed and heating begun, the temperature being gradually raised to 600° C. and maintained at this value for several hours, any abnormal increase in pressure other than the uniform rise due to expansion being noted. Finally the system was once more allowed to cool and the pressure was once more read.

The following temperature/pressure curves (Fig. 3) are typical of the results obtained, and

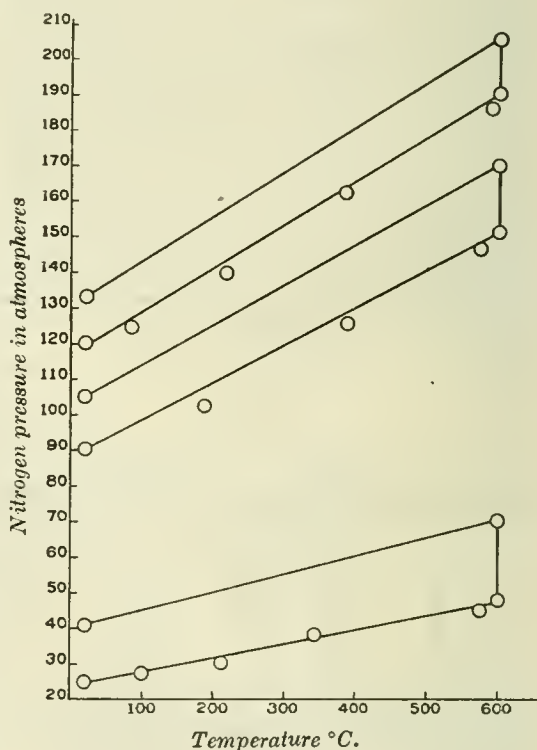


FIG. 3.

demonstrate the theoretical impossibility of synthesising iron nitride from iron and nitrogen, at any rate under the temperature and pressure conditions studied.

## DISCUSSION.

The CHAIRMAN asked what explanation could be given of the part played by iron as a catalyst in bringing about the combination of nitrogen and hydrogen. Did the active modification of Strutt play any part in any processes in which nitrogen combined with other elements?

Dr. D. F. TWISS, referring to Dr. Maxted's classification of processes for the combination of nitrogen and hydrogen into those dependent on the influence of a catalyst and those involving a primary activation of the nitrogen, enquired whether the remarkable power of leguminous plants to convert atmospheric nitrogen into compounds could be definitely allocated to either of these groups. He also asked whether the formation of ozone when a glowing Nernst filament was immersed in liquid oxygen was quite on a par with the production of ammonia from nitrogen and hydrogen as effected in Dr. Maxted's investigation because, although the formation of ozone in the former experiment was doubtless in the main a thermal one, it was likely that the ultra-violet portion of the radiation from the filament might play an appreciable part. Regarding the combustion of a mixture of hydrogen and oxygen in the presence of nitrogen, Dr. Maxted had apparently disregarded the possibility of formation of ammonium salts; as the combination of hydrogen and oxygen was only partial at the temperature of the oxy-hydrogen flame, and as at the same temperature oxygen and nitrogen could react yielding a little nitric oxide, it was possible that the rapidly cooled gaseous mixture would contain a little nitric oxide and oxygen or nitrogen dioxide so that the final aqueous solution might contain not only ammonia but ammonium salts such as the nitrate. Indeed, under the conditions of experiment, the possibility of the formation of some nitric acid by oxidation of part of the ammonia itself during the period of cooling did not seem to be altogether excluded.

Mr. W. H. PATTERSON enquired whether the author had tried methods of cooling other than by means of water. He pointed out that the cushion of steam which would surround the flame would probably act as a heat insulator. He suggested that cooling by means of a cold metal surface would be quicker and therefore more effective. Having spent 18 months in Haber's laboratory in Germany he had seen the early work carried out by him on the direct synthesis of ammonia from hydrogen and nitrogen. Early results of this work had been most unpromising; it was only when it had been realised that pressure played such a part in the equilibrium that progress had been made.

Dr. S. R. CARTER enquired whether any evidence had been found of the presence of nitric oxide in the cooling water. It seemed to him that the conditions were ideal for its formation. What was the proportion of nitric oxide to the total amount of ammonia in the cooling water?

Mr. MANLEY suggested cooling by causing the flame to impinge upon a block of ice. Steam would not be formed.

Mr. E. C. ROSSITER pointed out that it was possible easily to get a pure iron, over 99%. Some years ago when experimenting he obtained specimens containing approximately only 0.2% of oxygen, but he had not succeeded in getting iron entirely free from oxygen. That fact might have considerable influence on the decomposition of the iron nitride.

Dr. H. W. BROWNSDON recalled the fact that Ramsay had used lithium for the absorption of nitrogen. He asked what other nitrides besides that of iron had been examined with a view to possible practical application.

Mr. F. H. ALCOCK observed that Rayleigh and

Ramsay had used magnesium for the absorption of nitrogen when they were preparing argon.

Dr. MAXTED, in reply, said that Dr. Morrell had raised an interesting point as to the function of a catalyst. We needed to know more, however, of what a catalyst really was. It was noteworthy that only those metals that formed, for instance, unstable hydrides were capable of activating hydrogen. Similarly in regard to the formation of nitrides, only those metals which formed unstable nitrides were able to activate nitrogen. Thus, iron only acted as a catalyst at temperatures higher than the decomposition point of its nitride. It was generally found that a catalyst must not form a stable compound with the substance to be handed on. He was unable to answer Dr. Twiss' question as to catalytic power of plants. As to the thermal formation of ozone, the yields of the latter obtainable by using liquid oxygen for the sudden cooling, were almost equal to those capable of being calculated thermodynamically from the ozone-oxygen equilibrium. He thought not only that the formation of ammonium salts together with ammonia in the cooling water was possible, but that there was good reason to think that that was the case. Small traces even of soluble gases were usually capable of passing through several wash bottles without absorption, whereas in these experiments absorption either by water or by dilute acid gave practically identical results. He had cooled the gases by sweeping into water-cooled capillaries, but at present he could not publish the results. Cooling by ice was not much more intense than with rapidly cooled water because even on ice the cooling surface consisted of a film of water covered by a film of steam caused by contact with the flame. A large number of metals formed nitrides directly; but he was interested specially in the formation of nitrides of easily reducible metals. The object was to find a nitride that could be formed directly by the action of producer gas on a metallic oxide at a moderate temperature. It was possible to get a good yield of nitride directly from manganese and chromium but the temperature of reduction of the oxides was too high for the purpose desired.

## Glasgow Section.

*Meeting held at Glasgow on Saturday, January 26th, 1918.*

MR. JAMES MACLEOD IN THE CHAIR.

## NOTES ON CARBOLIC ACID: APPLICATION OF PHENOL TESTS TO WORKS PRACTICE.

BY GEORGE H. SHARPLES.

## PART I.

Some years ago it was noticed that there was always a serious variation between the quantities of phenol recovered from various oils and the theoretical valuation. These losses were important inasmuch as the quantity of recoverable phenol entered into the valuation of the oil when purchasing, and there was in consequence serious danger of commercial loss in working the oils.

Up to this time the valuation of tar acids had been carried out chiefly by Lowe's method and expressed in terms of 60's carbolie. As there seemed to be some doubt as to the precise constitution of 60's carbolie, this method was displaced in favour of the Weiss method for the determination of the phenol direct (first method, this Journal, 1913, 692).

The discrepancies continued, however, so it was decided to compare results obtained by the two methods in the same sample of tar acids, and



at the same time to examine the principles underlying each method. It was decided to adhere strictly to works conditions and use only such specimens as are produced daily in the works in the ordinary course of washing out the tar acids.

A series of samples was examined by both methods. In addition to the usual procedure in Lowe's method, a thermometer was placed in the retort with the bulb at the neck to note the range of the 625 grains collected and the specific gravity was taken. Each fraction obtained in the Weiss method was examined separately before mixing and where possible the quantity of phenol in the second fraction was determined. The sum of the quantities was taken and compared with the result obtained by mixing together the two fractions. The results (see Table I.) are stated with respect to the original samples.

This afterwards proved very useful in showing the relation of the specific gravity and the crystallising points to the amount of phenol in the presence of predominating quantities of mixed cresols, and the variation is no doubt largely due to *o*-cresol. In Weiss' method it will be known that the distillation is carried up to 170° C. At this point the flask is changed and the phenol separated from the water is added to the first fraction of the test. This small amount of phenol, as before stated, and confirmed by drying it carefully with concentrated sulphuric acid (cooled), contains considerable quantities of water, and as much as 2% of water might thus be added to the fractions under examination.

The effect of small quantities of water on the crystallising point and specific gravity was noted and the corresponding quantity of phenol determined.

TABLE I.

Raschig's test.						Weiss test 1. Fraction up to 190° C.					Weiss Test 2.								
											190°—197° C.				Fractions 1 and 2.				
% Water	Crys. pt.	Spec. grav.	% Phenol.	From	To	Vol.	Spec grav.	Crys. pt.	% Phenol	Vol.	Spec grav.	Crys. pt.	% Phenol	Spec grav.	Crys. pt.	% Phenol	Sum of cols 10 & 14.		
1	15	16.7	1.055	4a.	4b.	°C.	°C.					below 32°	—	1.058	17.25	33.5	34.4		
2	15	15.6	1.054	44.0	27.93	183	189	151	1.058	19.7	25.15	35	—	32°	—	1.056	28.93		
3	12	5.6	1.055	44.02	30	184	189	151	1.061	20.6	30.52	46	—	32°	—	1.058	32.88		
4	13.5	16.1	1.055	45.5	30	186	193	132	1.060	21.5	30	40	1.048	32°	4.67	1.057	34.67		
5	13.5	11.1	1.056	34.0	34.2	178	190	154	1.062	21	30.8	42	1.050	33°	4.9	1.058	35.7		
6	13	12.8	1.056	38.4	34.2	182	183	190	1.058	13	31.6	23	—	below 38°	—	1.056	33.13		
7	11	16.7	1.058	46.8	36.7	182	187	167	1.059	18.5	29.67	59	1.050	37°	6.88	1.057	37.55		
8	13	15.6	1.055	44.0	30.0	181	189	157	1.061	18.5	31.4	43	1.048	32°	5.00	1.059	36.4		
9	11	16.1	1.059	45.5	37.32	181	199	185	1.061	19	37	30	1.046	32°	3.38	1.059	40.38		
1.	2.	3.	4a	4b	5.	6.	7.	8.	9.	10.	11.	12.	13.	14.	15.	16.	18.		

Method of obtaining results:—

Column 4b: Calculated from Weiss' tables on the volume of crude acid, 5% being added for the first 10 c.c. distilled after driving off the water in accordance with Lunge's statement that this portion is 50% hydrated acid. (Coal Tar and Ammonia, p. 608, Vol. II., 4th Ed.).

Columns 5 and 6 range in which the 62.5% distils over (thermometer in vapour).

Columns 10, 14 and 17: Calculated upon original volume of crude acid.

In column 4a is inserted the % phenol obtained from a graph representing the value determined by a German firm according to

Lowe's method: this graph is made from several years' observations.

The oily matter was eliminated before the tests.

In Weiss' method it will be noted that in some instances it was not possible to determine the phenol in the second fraction alone, but phenol is adjudged to be present in small quantities as in every case but one the result of the combined fractions is greater than No. 1. fraction alone. The sum of the two fractions, however, where both could be determined, was greater than the single result on the combined fraction. In Lowe's method, column 4a represents the percentage of phenol from a graph based on observations of several years' use of this method by a German firm. The results compared with those obtained by Weiss' method, are widely divergent, being invariably higher.

Weiss' tables (Graph I.) were applied to the fraction of 625 grains in Lowe's method to determine if any relation between these two methods could be established, and 5% was added in accordance with Lunge's statement that the first 100 grains consists of 50% hydrated phenol. This result is only relative, as the range of the temperature, even in a retort, distilling direct, shows that considerable quantities of phenol must remain in the residues. This is confirmed by the difference between columns 4a and 4b. The results, however, are again widely divergent, and conclusions drawn therefrom as to the value of the tar acids must vary accordingly.

As Weiss' tables offered too much latitude in their use in determining the percentage of phenol from the crystallising point and the specific gravity, the graphs were rearranged as shown in Graph II.

% Water.	Sp. gr.	Cryst. pt.	% Phenol.
0.	1.0570	15.5	47.4
1.	1.0565	11.2	47.4
2.	1.0558	8.5	48.0
3.	1.0540	4.5	Out of range of table.

Weiss has modified his method since (this J., 1917, p. 863).

Table II. shows the effect on Lowe's tests caused by varying the amount of *m*- and *p*-cresol, and the water. There appears to be no rational conformity of results.

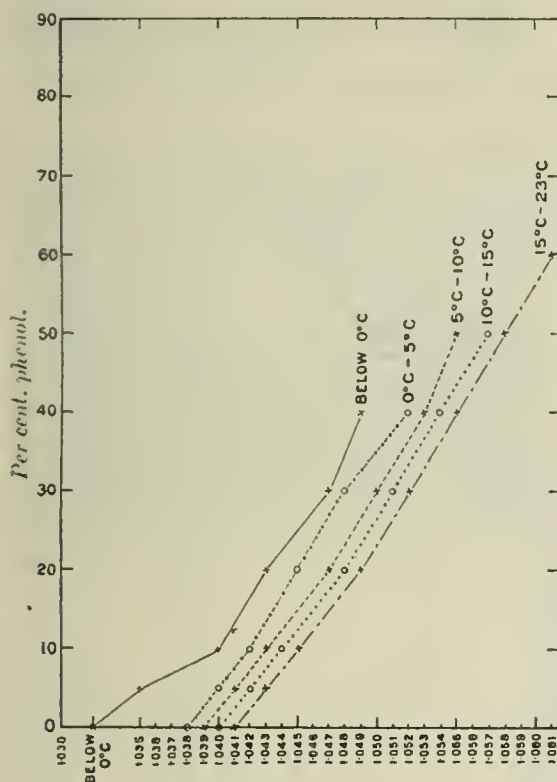
Lowe's tests.

TABLE II.

Phenol.	Mixed cresylic acid.	<i>m-p</i> -cresol.	H <sub>2</sub> O.	Cryst. pt.	Solubility in 12% NaOH.
%	%	%	%	° C.	
60	40	—	—	25	1 in 5.4 pts.
80	—	40	—	20	1 in 6 "
60	30	—	10	20.4	1 in 4.5 "
60	—	30	10	20.7	1 in 5 "
40	30	30	—	4	1 in 5 "
30	30	30	10	below 3	1 in 3.6 "
43	—	44	13	11.1	1 in 6.3 "
44	—	46	10	10.9	1 in 5.8 "
44.5	—	48.5	7	7	1 in 5 "
45	—	55	—	11.5	1 in 5 "
45	—	43	12	9.4	1 in 6.5 "
45	40	—	15	11.2	1 in 6 "

## PART II.

Lately (in January, 1916) it was found necessary in order to prevent loss of phenol in residues to adopt some ready means of determining small quantities of the former in crude cresylic acid. Lowe's test was not applicable, and our previous experience led us to seek other methods than Weiss'. It was necessary to adopt a quick method so that the plant was not held up whilst awaiting results. The Government shorter method was satisfactory when phenol was absent, but the difficulty was manifest when small quantities of phenol were present. The method described by Fox and Barker (this J., Aug. 15, 1917, p. 842) gave concordant and trustworthy results. The data given in that paper were accepted and the method applied directly to works' conditions and works' samples.



Sp gr. at 60°F.

GRAPH I.

It was found necessary to obtain pure samples of carbolic acid and distil cresylic acid until no phenol was present; *o*-cresol was then added to replace any lost in the extraction of phenol and to make such a mixture that more than 50% would distil over up to 195°C. In practice with cresylic acid containing low contents of phenol I have never found a mixture which would fulfil these conditions without the addition of *o*-cresol, so that 35 to 40% of *o*-cresol was always added at the commencement, dispensing with the preliminary distillation. Graph III. has been constructed with such a mixture, whilst Graph IV. was made without the addition of *o*-cresol.

Tests made with known mixtures on this method gave the following results:—

	Exp. 1.	Exp. 2.
Phenol in original mixture .....	19.4	15.0
Phenol estimated .....	19.2	15.9
<i>o</i> -Cresol present .....	64.6	57.0

The amount of *o*-cresol present in Experiment 1 approximates more closely to the constitution of the cresol used in Graph III.

The necessity for a graph made with a pre-determined mixture of cresols was amply demonstrated in the experiments made with other methods and other mixtures. In several instances negative results were obtained, that is, the quantity of phenol found was actually less than the phenol added. This was due entirely to the use of a graph made with the aid of a mixture of cresols not corresponding to the mixture under examination.

The method used by Masse and Leroux (this J., 1916, 1148), was examined in the course of the investigation but was soon abandoned for the above reason, coupled with the large quantity required for each test and the number of crystallisations required.

In this method the *o*-cresol is distilled off in the first portions, so that the latter portions contain only small quantities of phenol and are relatively weak in *o*-cresol. It is to these portions that the phenol must be added and the result is obtained from a graph which, in virtue of the presence of *o*-cresol, gives lower results from a corresponding crystallising point, hence a lowered value.

Again, the quantity of material taken affects the results, e.g., using a mixture of 70% of phenol and 30% of cresylic acid, 1501 grains gave a phenol content of 96% of that taken and 254 grains 90%. With smaller proportions of phenol the discrepancy was more marked.

In a mixture of 40% phenol and 60% *o*-cresol, over 100% of the added phenol was indicated by this method. In all the cases cited, Graph IV., which was made without the additional *o*-cresol, was used, giving higher results in the latter case, as the *o*-cresol was interpreted as phenol. In the former cases the opposite results were indicated. The Government method avoids this pitfall by determining the phenol always in a similar mixture.

The original Weiss method shows the same tendency to variation. The results are affected by the variation in the mixture of cresols used, a mixture with 30% *m*- and *p*-cresol alone, bringing the apparent percentage of phenol down to 70% and 60% *o*-cresol alone increasing the phenol to 125%.

In the application of these methods to practical work it is plainly seen that Masse and Leroux's method only furnishes a result which is a measure of the quantity of crystallisable phenol obtainable under ordinary working conditions, but the Government method gives a true index of the total phenol and bears no relation to the quantity obtainable. This was tested in the following experiment.

## PART III.

Dark cresylic acid was distilled and concentrated in the laboratory so that the phenol could be extracted under conditions approximating to the ordinary working conditions. It was impossible, however, to reproduce those conditions exactly because within the works fresh material is constantly added and the concentration may be said to proceed in two directions: (1) → to phenol, (2) to cresol ←, the intermediate fraction containing proportions of phenol receiving additions in bulk which enable the distillation to proceed more freely.



In the laboratory, however, these fractions are constantly diminishing unless a large series of primary distillations are undertaken. The cresylic acid under examination was estimated to contain 4.09% of phenol.

#### Distillation of ordinary cresylic acid.

2000 c.c. in copper still with 15-disc Young's column yielded:—

Water.	Oil.	
255 c.c.—12.25%	50 c.c.—2.5%	Separated from the cresylic acid.

Fractions collected.		
I. to 190° C. ....	20 c.c.	} Cryst. pt. 35° F.
II. to 197° C. ....	234 c.c.	
III. to 200° C. ....	460 c.c.	

Total to 200° C. ....	714 c.c.
IV. above 200° C. ....	981 c.c.

A. From I. and II.

		Yield.
254 c.c. ....	to 197° C. ....	156 c.c. to D.
	197°—200° C. ....	71 c.c. to C.
	above 200° C. ....	27 c.c.

C. From IV.

	981 c.c.	Yield.
From A to 200° C.	71 c.c.	197° C. .... 13 c.c.
From B to 200° C.	90 c.c.	200° C. .... 280 c.c.
Total ....	1142 c.c.	above 200° C. .... 850 c.c.

D. All fractions from A, B, and C.

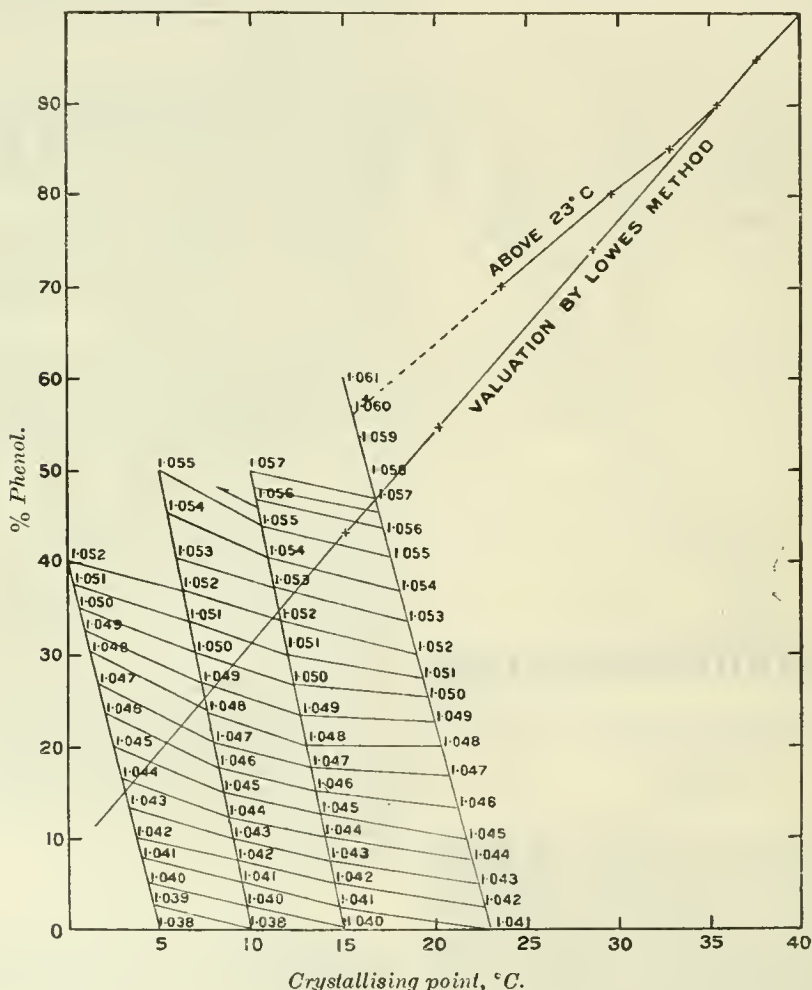
	Distilling to 197° C.	Yield.
From A.	156 c.c.	142 c.c. with cryst. pt. 39.9° F.
From B.	157 c.c.	116 c.c. to 200° C
From C.	13 c.c.	
Total ....	326 c.c.	

E. Coupled with C.

F. Redistilled crystallisable portion of D=142 c.c.

	Yield.
38 c.c. ....	cryst. pt. 60° F.
84 c.c. ....	cryst. pt. 41.2° F.
Residue on to H = 70 c.c.	

G. Recrystallised second portion of F. after concentration, cryst. pt. 55.8° F.



Crystallising point, °C.

GRAPH II.

B. From III.

		Yield.
460 c.c. ....	to 197° C. ....	157 c.c. to D.
	198.5° ....	39 c.c.
	200° C. ....	90 c.c. to C.
	above 200° C. ....	174 c.c.

H. consisted of—

	Yield.
B.	39 c.c.
C.	280 c.c.
D.	116 c.c.
F.	70 c.c.
Total	505 c.c.

K. Concentrated 231 c.c. from H to 197° C.; no crystallisable fraction obtained here.

L. G concentration gave 14 c.c.—cryst. pt. 60° F.

Total crystallisable recovered.

From F... 38 c.c.

From G... 14 c.c.

52 c.c. containing 1.17% phenol calculated to original sample.

on the original tar acids, but if these be fractionated to 190° C. a crystallising point can be obtained by Lowe's test. This test was adopted because it was the quickest method and did not hold up the washers whilst awaiting results. It will be noticed on referring to Table III. that the crystallising point was raised in the weak carbolate used.

TABLE III.  
*Washing of light creosote.*

	Distillate to 190° C.				Raschig test on distillate to 190° C.; fraction 62.8%.	
	Tar acids, %	Phenol, %	Sp. gr. at 15.6° C.	Cryst. pt.	Sp. gr. at 15.6° C.	Cryst. pt. °C.
Light creosote .....	17	6.6	1.046	below 0° C.	—	—
First carbolate wash before using .....	18.5	32.3	1.052	"	1.051	3.4
First carbolate wash after using .....	20.0	43.5	1.055	"	1.0565	12.4
Second carbolate wash before using .....	20.0	35.3	1.052	"	1.053	6.8
Second carbolate wash after using .....	22	32	1.052	"	1.053	0.5
First soda wash .....	16.5	17	1.052	"	1.052	0

It is noteworthy that in section K, although a considerable quantity distilled below 194.5° C., viz., 50 c.c., no crystallisable phenol was obtained. This fraction proved to consist largely of *o*-cresol.

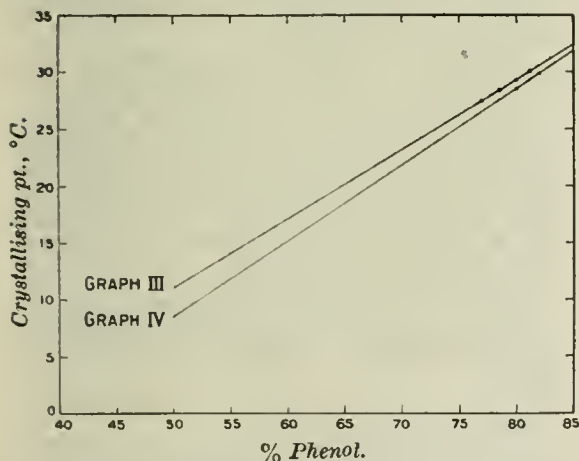
The bromine test as described by Fox and Barker was applied to the non-crystallisable fractions and showed the presence of phenol in slight quantities.

It has been found in making 60's carbolic by fractional washing that considerable losses occur, much phenol being left in the cresylic acid from the heavier oils, but if the whole of the tar acids are extracted without resorting to fractional washing and afterwards distilled much greater recovery of phenol can be effected.

That phenol is present in oil as heavy as creosote is well known, but by ordinary fractional washing it cannot wholly be recovered as the process would become tedious and involved, requiring

Phenol is here indicated in recoverable quantities in light creosote.

With reference to the bromine test used to ascertain the presence or absence of phenol in the residual fractions as indicated by Fox and Barker, trials were made to ascertain the limiting quantities of phenol which would be indicated. At first the indications appeared inconclusive, but with practice one was able to determine freely the presence or absence of phenol. Mixtures were made containing 10%, 5%, 2.5%, 1.25%, 1.0%, 0.66%, 0.5%, 0.2%, 0.1%, and 0.0% phenol. From these it was ascertained that 1.0% could be ascertained with certainty; below that point the results appeared doubtful.



GRAPHS. III. and IV.

Graph III. Mixed cresol and 35% *o*-cresol.

Graph IV. Mixed cresol only.

Graph III. Note.—Above 66% phenol the quantities of mixed cresol and *o*-cresol were reduced proportionately to allow for increased addition of phenol.

many batches of graded weak carbolates to carry forward the phenol to the strong carbolate. The following tests show the presence of phenol, although Lowe's method gives no crystallising point

## Liverpool Section.

Meeting held at the University, on Friday, February 15th, 1918.

MR. JOHN GRAY IN THE CHAIR.

## THE MANUFACTURE OF TINPLATE.

BY T. LEWIS BAILEY, PH.D., F.I.C.

Tinplate consists of sheet iron, or more properly mild steel, which has been coated with a thin film of metallic tin, the main object of this being to protect the steel from oxidation (rusting) in a moist atmosphere.

One of the chief uses to which tinplate is put is for casing petroleum and some few years ago one-third of our export of tinplate went for this purpose; other extensive applications are for canning beef, fish, vegetables, fruit, etc., the manufacture of containing vessels for confectionery tobacco, and so on.

The production in this country runs into very high figures and entails the use of over a million tons of steel yearly and something like 16,000 tons of tin.

The tinning of iron sheets on any scale dates from the early part of the 17th century and



the early home of the industry appears to have been Saxony and Bohemia. In the latter part of the same century tinplate was made to some extent in England, though the industry did not flourish here until 1720, when works were established at Pontypool in Monmouthshire by Major John Hanbury; other works soon followed in the same county and the industry extended westward throughout the whole of South Wales. It is a curious fact that to-day this trade is almost confined to South Wales and Monmouthshire. The number of works in 1750 was four, in 1850 it had increased to 34, and 25 years later there were 75 works, since which date the actual number of works has not increased; present-day works, however, are on a much more extended scale, development in more recent times having been largely in the direction of increased output.

An excellently illustrated paper on modern tinplate manufacture was read by R. Beaumont Thomas in 1906 at a meeting of the Institution of Mechanical Engineers at Cardiff\*, and a Home Office Report was published in 1912†, but textbook literature deals almost entirely with the older, now historical, methods, and a general summary may be found useful.

The earliest methods of making tinplate were described by Reaumur in 1725. Iron bars, about one inch square, were hammered out into thin sheets, which were cut to the requisite sizes by hand shears, then scrubbed with sandstone and pickled in water, rendered acid by means of fermenting barley meal. The pickled plates after being cleaned with sand and well washed were coated with tin. The pickling process occupied a couple of days and was carried out in vats, placed in vaults in which large fires were kept burning; periodically the workmen engaged on the operation entered the vaults in order to turn the sheets in the liquor to ensure even pickling.

The tinning of the sheets was carried out as follows: They were first immersed in molten palm oil, whence they were transferred vertically to a bath of molten tin; a thorough brushing of the sheets with a hempen brush followed this, and then came immersion in a second bath of tin, the molten tin in this case being covered with a layer of tallow. Excess of tin was removed by immersion in a bath of molten grease, after which the tinned sheets were cleaned and polished by hand with sawdust and moss. An excellent article was produced, with a heavier coating than is usual at the present time, when cost and competition are factors of such importance.

Present-day tinplate processes are of a totally different order.

The steel works deliver rolled bars, appropriately low in carbon (about 0.1%), to the tinplate manufacturer: these bars are from 6 to 10 inches in width and from  $\frac{3}{8}$  to  $\frac{1}{2}$  inch thick. The operations may be summarised as follows: details of general knowledge only may be given, of course.

(1) The bars are cut by means of bar-shears into desired lengths, usually about 20 inches.

(2) The cut bars are heated in the mill-furnaces and are rolled hot through two sets of rolls (roughing and finishing rolls). Sheet rolling was first introduced in 1728. According to present practice there are usually five separate heatings and rollings necessary to produce the requisite gauge of sheet.

After two heats the thickness of the sheets produced is under one-tenth of an inch; to ensure satisfactory rolling the sheets are doubled; doubling follows also the third heating and the fourth heating, so that after the fifth heating and rolling each sheet consists of eight layers.

By the employment of such methods a considerable output of thin sheet is attained in a short time. As an example of the results of rolling, a  $\frac{1}{2}$ -inch bar will be transformed into a sheet of eight layers, each layer having a thickness of little over a hundredth of an inch.

Another method of rolling (so-called matching practice) common in America, consists in putting two pieces together after a first rolling; first doubling thus gives four layers and second doubling four layers.

Occasionally in matching practice three plates are used, in which case a first doubling gives six layers and the next twelve.

(3) The rolled "eights" are sheared by means of a plate shearing machine into three portions, which are trimmed, and thus from an original bar 20 in.  $\times$  8 in.  $\times$   $\frac{1}{2}$  in. there will be produced, for example, 24 sheets, each 14 in.  $\times$  20 in.

(4) Owing to the repeated heating, doubling, and rolling there is considerable adherence of the individual sheets and the operation known as "opening" is necessary; this is effected by starting separation at one corner and then tearing the sheets apart.

(5) So-called black pickling now follows, the sheets being immersed in dilute sulphuric acid, heated by steam. The object is to remove from the surface of the sheets the oxide scale, which has formed during repeated heating. In this operation the sheets are packed loosely into a carrier, which depends from one arm of the pickling machine. This is rotated to a point above the pickling vat, then lowered so that the sheets enter the acid, and an up-and-down motion is continued during the period of immersion. The pickling occupies a matter of a few minutes only and, when complete, raising of the carrier and further rotation takes the carrier with its load to a washing tank plentifully supplied with running water. At the same time a second arm of the machine has brought a fresh batch of sheets to the acid bath.

For pickling purposes hydrochloric acid took the place of barley meal in 1760, and this in turn began to give place to sulphuric acid in 1806. Sulphuric acid is now universally employed in the tinplate trade proper. Waste "pickle" still contains free sulphuric acid; after treatment with iron scrap, ferrous sulphate (copperas) is recovered by crystallisation.

Proposals have recently been made in the United States to pickle electrolytically\*, the sheets forming the cathode in an acid solution.

(6) Annealing of the sheets is now necessary (black annealing). For this purpose the sheets are stacked horizontally on cast-steel or wrought-iron stands and covered with boxes of like material, sand luting being provided between cover and base. The annealing furnaces are either coal fired or gas fired and the operation occupies from 10 to 12 hours. The product is now what is generally known as "blackplate."

(7) Now comes a further rolling (cold rolling); the sheets are rapidly passed separately through three or sometimes four successive trains of rolls, whereby they attain considerable smoothness and polish.

(8) A second annealing (white annealing) follows, occupying about seven hours.

(9) A second pickling in sulphuric acid is necessary, but this is of short duration and after thorough washing in water the sheets are ready for to receive their coating of tin.

(10) Tinning. Hand labour is dispensed with as far as possible in all the operations hitherto described, and this applies also to the tinning operations; the old arduous methods have been superseded by the introduction gradually since 1866 of mechanical devices. The sheets are fed

\* Proc. Inst. Mech. Eng., 1906, 499.

† Report on the Conditions of Employment in the Manufacture of Tinplates, with special reference to the Process of Tinning; by Edgar L. Collis and J. Hilditch. [Cd. 6394.]

\* Thompson and Mahlman, this J., 1917, 1097.

by hand into the cast-iron tinning pot and are removed by hand ready for cleaning (though a device has even been introduced for transferring them mechanically from the tinning pot to the cleaning machines).

The tinning pots are of two varieties, a double horizontal type and a vertical type. In either case the sheets separately enter the tinning pot through a layer of zinc chloride solution, floating on the surface of the molten tin. The pots usually contain 3 or 4 tons of tin, kept molten by a coal fire under the pot. A succession of pairs of revolving rolls and guides at various positions guide the sheets on their way through the tin, downwards and then upwards until they emerge from the tin and pass vertically through a column of molten palm oil. During their passage through this latter a pair of rolls removes excess tin.

(11) The tinned sheets are now passed on to a cleaning machine, which carries them twice through a mass of bran or of "pink meal" (consisting largely of gypsum, mixed sometimes with sawdust) and this machine delivers them to a battery of dusting rolls covered with sheepskin, each alternate pair of rolls running at different speed to ensure polishing action on the surface of the now finished sheets.

The development of the tinplate trade has been essentially along mechanical lines but throughout the operations described there are problems which would well repay chemical investigation. At the end of the process, moreover, there exists a problem which is peculiarly chemical: it is that of the treatment of the dross, which collects on the surface of the tin in the tinning pot, a material technically known as tin scruff. This is periodically skimmed off and put on one side for treatment. It is a very complex mixture, containing 50% or even more, of metallic tin, mixed with chlorides of tin and zinc, iron and sulphur compounds and palm oil, together with oily decomposition products of palm oil. It is quite impossible satisfactorily to sample the material for sale to tin smelters and consequently it is heated in reverberatory furnaces at the tinplate works with production of a certain amount of metallic tin and a considerable oxidised residue containing tin, iron, and zinc, but now in a condition to admit of sampling for sale purposes. This operation, which should be a liquation process purely, is frequently carried out at abnormally high temperatures and causes the evolution of very dense fumes, containing considerable quantities of oxides and chlorides of tin and zinc, free hydrochloric acid, and products of destructive distillation of palm-oil. Such fumes, at one time evolved from short 20-ft. chimneys, were a source of trouble: they were very irritating and were, moreover, destructive of some types of vegetation in the neighbourhood of the works—the thick fumes travelled for considerable distances in the form of a thick white fog. Works dealing with the smelting or liquation of scruff were included as a class in the provisions of the Alkali, etc., Works Regulation Act, 1906, and investigations were carried out with a view to mitigating the nuisance due to the evolved fumes, and this resolved itself essentially into devising simple means for the reduction to satisfactory limits ( $\frac{1}{2}$  grain per cubic foot) of the hydrochloric acid contained in the gases passing from the chimneys.\* The problem of ascertaining the best possible method of treating tin scruff for the complete recovery of its valuable constituents could not receive attention, but it will be of interest to record some of the results obtained during the working out of the problem as it presented itself from an Alkali Act standpoint.

The method employed in "scruff-melting" usually consisted in charging a couple of hundred-weight or more of scruff on to the hearth of the heated furnace, allowing it to remain for a quarter of an hour until thoroughly heated to softening point, then stirring it, allowing it once more to remain dormant for a few minutes, then rabbling well for short spells during the remainder of the operation, until metallic tin practically ceased to run from the furnace. As it was usual to mix together all skimmings, those containing much zinc chloride and those containing little, it is evident that there were great possibilities of loss occurring mechanically and chemically during the furnacing operations: in most cases no dust-depositing flues were in use and the chimneys were very short; where flues of any length did exist they were frequently of small cross-section and led to a high boiler-chimney, not dampered off at the chimney end of the flue—a furnace damper only, naturally, had little effect on the deposition of fume in such flues.

The first modification introduced was to keep "flux skimmings" (*i.e.*, those especially rich in zinc chloride) separate from the scruff proper and to wash these thoroughly in running water heated by steam. This precaution had the effect of considerably reducing the amount of fume evolved and at the same time lower temperatures could be used for liquation and better yields obtained.

From many estimations made at different works, it was shown that, with flux skimmings present in the charge, the chlorides in the chimney gases were equivalent to 2 grains of hydrochloric acid per cubic foot, whereas when flux skimmings were kept out the chlorides in the chimney gases were reduced to less than 1 grain per cubic foot, in terms of hydrochloric acid: when flux skimmings were present in the charge 2½ grains of  $\text{SnO}_2$  per cubic foot was present in the chimney gases, but if flux skimmings were kept separate, the escape of  $\text{SnO}_2$  was less than  $\frac{1}{2}$  grain per cubic foot.

The second step was the introduction of depositing flues for the purpose of retaining as much oxide of tin as possible, and at the end of the flues was built a simple type of wash-tower, in which hydrochloric acid was to be absorbed by water. All forms of tower packing proved impracticable: even pigeon-hole brickwork quickly became obstructed with thick greasy deposits that could not be removed by water. Towers with sloping shelves proved satisfactory from an absorption point of view, but breakages of the shelves were frequent, and ultimately unpacked brick towers were adopted, and to these water was supplied in the form of a shower from perforated plates, placed on the top of the towers. Two towers, each 9 feet high and 4 feet square, working in series, proved reasonably satisfactory for the purpose of removing hydrochloric acid from the gases. As a result of the various alterations adopted in the process the free hydrochloric acid was gradually reduced, after several years of patient work, from an average of 0.8 grain per cubic foot of chimney gases to 0.086, a figure well below the limit prescribed as the maximum permissible. The wash-towers have, moreover, had a further beneficial effect, in that they reduced the total acidity of the chimney gases (in terms of  $\text{SO}_4$ ) from 1.55 grain per cubic foot to 0.48 grain. The extent to which it was possible to reduce the loss of oxide of tin depended chiefly on the depositing flues that were built: where reasonably large flues were put in, with a control damper at the chimney end of the flue, good recovery of flue dust rich in  $\text{SnO}_2$  resulted, and this proved a valuable product, of course.

In 1915, at the desire of the Chief Inspector, special investigations were carried out on one plant, where a depositing flue and wash-towers had been installed. It was desired to ascertain the efficiency

\* Annual Reports on Alkali, etc., Works, 1908 to 1913, the portions relating to Districts VI. and VII.



of both flue and wash-towers. Full details of the investigations, which extended over several days, are given in the Alkali Report for that year.\* The depositing flue in the case in question was 125 feet in length and 2 feet square in section; the speed of the gases ascertained was from 4 to 5 feet per second (cold), furnace temperatures during the operation were 800° to 900° C., the mean temperature of the gases entering the flue was 440° C., ranging between 650° C. and 350° C., and this became reduced to 240° C. by the time the wash-tower was reached. The wash-tower contained two outer compartments, each 9 feet high and 4 feet square, and one intermediate compartment 9 feet high and 4 feet by 9 inches section, each compartment was provided with a water shower, the gases ascended in the two larger compartments and descended in the smaller intermediate compartment; the wash-tower cooled the gases from 240° C. to 105° C. Under such conditions 82% of the hydrochloric acid was removed and 42% of the oxide of tin leaving the furnace was condensed. The amount of tin passing away entirely from the system still constituted 10% of the total tin charged into the furnace. The furnacing operation includes long spells of rabbling of the charge and at such times there is admission of much air, so that an oxidising atmosphere prevails, by no means an ideal condition in a process of this kind. A furnace temperature of 900° C. must be considered high (considerably higher temperatures have been met with) and it was clearly indicated in the course of the investigations that with lower working temperatures there is improvement in the results, but the existing system itself might well be improved by the adoption of a larger condensing area, better regulation of draught, and by contact with water even in the earlier part of the system.

A method of scruff treatment that proved particularly suitable for small works consisted in washing the whole of the tin scruff for some days in water kept at somewhere near boiling point by means of steam, dissolved zinc chloride, etc., being continuously removed by a small continuous supply of water to the washing tank. The washed scruff lends itself to furnace treatment at a very low temperature, yields are good, and there is practically no evolution of either hydrochloric acid or tin compounds; depositing flues and wash-towers thus become unnecessary.

But of course there is in none of these methods any attempt at recovery of by-products with the exception of oxide of tin. What one would like to

see investigated is a self-contained method of complete scruff treatment on chemical lines, but, until some sort of chemical control obtains in the tinplate works, there is little likelihood of this and other problems being taken sufficiently seriously.

During the past few years three patents have been taken out in connection with the treatment of tin scruff. In 1909, H. J. Bailey\* patented a combined smelting and lixiviating process and in the same year James Stephens† patented a separation and washing method, in which recovery of dissolved metallic compounds was aimed at. In 1912, S. B. Bowen‡ brought out an entirely different type of process: a preliminary heating of the scruff in a closed vessel by waste heat from the scruff furnace proper brought about liquation of a proportion of the metallic tin and at the same time chloride of tin and oily matter distilled and were condensed in small cooling towers; the liquated residue was then washed in water and furnace for further recovery of tin.

But progress has not been made on these lines; with lack of chemical control it can scarcely be looked for.

I desire to thank the Chief Inspector of Alkali, etc., Works for permission to give details regarding the work done in connection with scruff-furnace gases.

#### DISCUSSION.

Mr. JOHN GRAY hoped that chemists would find their way into this important industry in which there was obviously scope for them. Why was palm-oil used in preference to any other oil?

Dr. HORTON asked if there were any difficulties due to lack of uniformity in pickling and if so, what steps were taken to deal with them?

Mr. CARRUTHERS THOMSON wished to know the average percentage of tin in tinplate of modern manufacture. About 30 years ago he believed it had been of the order of 2½%.

Dr. BAILEY in reply said that tallow used to be used in the tin-pots but palm-oil was now used because it was eminently suited to the process and, moreover, was easily obtainable. In pickling, uniformity was ensured by insisting on a reliable acid (arsenic-free and of definite strength). At the present time tinplate contained about 1% of tin on the average.

\*Annual Report on Alkali, etc., Works, 1909, p. 108. Eng. Pat. 17,474 of 1909; this J., 1910, 1663.

†Annual Report on Alkali, etc., Works, 1909, p. 108. Eng. Pat. 17,345 of 1909; this J., 1910, 497.

‡Annual Report on Alkali, etc., Works, 1912, p. 108. Eng. Pat. 7945 of 1912; this J., 1913, 493.

\* Annual Report on Alkali, etc. Works, 1915, pp. 48 to 61; E. Linder and T. L. Bailey; this J., 1916, 1012.

## Canadian Section.

Meeting held at Toronto, Ontario, on Friday,  
December 21st, 1917.

MR. S. B. CHADSEY IN THE CHAIR.

# THE ACIDIMETRY OF COLOURED SOLUTIONS: AN APPLICATION OF THE POCKET SPECTROSCOPE.

BY ALFRED TINGLE, PH.D.

(Preliminary Note.)

It became necessary at this laboratory to make acidimetric titrations of deeply coloured alkaloidal solutions, and it was found that the colouring matter could not be removed without vitiating the quantitative nature of the results. Ordinary methods of meeting the difficulty having failed, it has been found that the spectroscope offers a solution.

The absorption spectra of indicators are, of course, markedly different in acid and in alkaline solution, and the presence of an independent spectrum, arising from the natural colouring matter, does not prevent the observation of the first one, unless indeed it should happen that in some individual cases the two spectra overlap.

In practice the author has found it very nearly, if not quite, as accurate to determine the end point of an acidimetric titration spectroscopically as with the naked eye, and the spectroscopic method is certainly available in many cases where otherwise no end point could be seen. Obviously it can be used at night as easily as in day time.

## The spectroscopic method.

Two similar vessels are provided, one of which holds the solution to be titrated, the other an equal volume of distilled water. To the latter is added one drop of the standard alkali to be used, and then an accurately-measured quantity of the indicator. The latter is added, a little at a time, till the characteristic absorption band shows a sharp enough edge. The quantity of indicator is noted and the position of the edge of the band is also noted. This position marks the end point.

The same volume of indicator solution is next added to the liquid to be titrated, after which titration is carried out in the ordinary way but for the fact that the spectroscope is used in place of the naked eye to watch the change of colour. In most cases this will be found to result from the shifting of an absorption band rather than the substitution of one band for another. The shifting may be gradual, but when the band watched has reached the position registered for it in the "blank experiment," the end point has been reached. In every case, it need hardly be said, the same thickness of liquid must be examined, but the shape of the vessel is of no importance. The author has often used a conical flask and obtained as good results as with any other container.

The spectroscope used in this work was a direct-vision Beek-Thorp diffraction instrument. It is one of the cheapest on the market and extremely convenient. A large spectroscope would be very unsuitable for such a purpose as the present one, but any handy instrument could be used. No

attempt has been made to express numerically the positions of the bands of the different indicators worked with. These vary with the dilution, thickness of layer examined, and perhaps other factors, and each worker must find those conditions best suited to his own peculiar eyesight.

In the following experiments the volume of liquid worked with was in each case about 60 or 70 c.c., and the thickness of layer examined about 45 mm. The solutions were contained in flat-sided tincture bottles. It will be noted that the volume of indicator used is much larger than would be suitable were observations being made with the naked eye.

*Experimental results.*—Every indicator hitherto worked with has been found to have its own peculiarities, which must be carefully studied before it can be used in connection with the spectroscope. Some of these are still being looked into and at present the author can only give details of methyl orange and cochineal.

A reliability test was made by measuring out definite quantities of decinormal sulphuric acid, colouring these with carefully neutralised extracts of black tea or stick liquorice, so that the end point would not be visible to the unaided eye, and then titrating by the method outlined above against decinormal soda.

Exp. No.	Vol. of N/10 H <sub>2</sub> SO <sub>4</sub> by measurement. c.c.	Vol. of N/10 H <sub>2</sub> SO <sub>4</sub> found by titration. c.c.	Indicator used.
1. ....	15.0	15.21	} cochineal 2.5 c.c.
2. ....	26.0	25.98	
3. ....	20.0	19.96	
4. ....	21.0	21.01	} methyl orange 1 c.c.
5. ....	17.5	17.49	
6. ....	14.0	14.11	
7. ....	18.5	19.16	

The failure in the last case quoted appears to have been due to a temporary defect in the source of light. This trouble was not suspected till the readings had been taken and the solution thrown away, so that re-titration was impossible. As a further factor in considering the degree of accuracy, it should be noted that while performing these titrations the author did not know what result he might expect, the acid having been measured by Mr. Babington, Chief Analyst to the Customs, the amounts not being disclosed till the titrations were completed. For this and much other help in other directions the author's best thanks are due to Mr. Babington. The cochineal solution used was a saturated extract made with 50% alcohol. The methyl orange solution was 0.1% of colouring matter in water.

Lately Mr. Babington and the author\* commented on the different titre shown by solutions according to whether cochineal or methyl orange is used as indicator. The spectroscopic method appears to bring methyl orange back into line with cochineal and lacmoid. Figures cannot be quoted at present, but it is apparent that the spectra of neutral methyl orange and alkaline methyl orange are the same. From this it follows that the usual practice with this indicator of titrating to an intermediate tint is mistaken. Alkali should be added till the fullest change to yellow has taken place. Cochineal, on the other hand, changes colour as it becomes more alkaline.

The author hopes shortly to publish a more complete account of this work in connection with other indicators.

Customs Laboratory, Ottawa.

\* Chemical Industry in Canada during the War, X., 32.



## Glasgow Section.

*Meeting held at Glasgow on Tuesday, 18th December, 1917.*

MR. JAS. MACLEOD IN THE CHAIR.

### THE PRESERVATION OF BUILDING STONE.

BY CECIL H. DESCH, D.SC., PH.D.

(Abstract.)

The method of protection of building stone by means of soluble silicofluorides, first employed by Kessler\*, is free from all the objectionable features possessed by the other methods which have been proposed or adopted for the purpose. The alkali salts are not used, on account of their sparing solubility, and also of the fact that, like the alkali silicates, they leave soluble carbonates in the pores of the stone as by-products of the reaction. On the other hand, the silicofluorides of magnesium, zinc, and aluminium, which are stable salts dissolving in water to form slightly acid solutions, yield only insoluble products with calcium carbonate. Carbon dioxide is freely evolved when the acid solution is applied to the surface of the stone, and the pores are closed by the formation of silica and insoluble fluorides. When the magnesium salt is used, the reaction is as follows:—

$\text{MgSiF}_6 + 2\text{CaCO}_3 = \text{SiO}_2 + \text{MgF}_2 + 2\text{CaF}_2 + 2\text{CO}_2$ , the protective layer being thus composed of silica and magnesium and calcium fluorides. I have found this method to be thoroughly effective in preserving even highly porous limestones against disintegration, and in reducing the porosity of those exposed to weather, as in the case of walls with a south-westerly aspect, through which the rain was able to penetrate freely before treatment.

A simple test of the porosity of a limestone may be made by using a flat sawn surface, and cementing on to this a short length of brass tube, 2 or 3 cm. in diameter, by means of a ring of molten paraffin wax. A measured quantity of water, say 5 c.c., is introduced into the tube by means of a pipette, and the time taken by the water to disappear completely is noted. A fresh area of the surface must of course be taken for each test.

Should the original limestone be of very open texture, it is advisable to prepare the surface by brushing with a cream made of dust of the same stone and water, allowing this to fill the pores before applying the silicofluoride solution. This assists in the formation of a compact layer, and does not alter the appearance of the stone.

Magnesium and zinc silicofluorides are crystalline substances, which dissolve without difficulty in water. The aluminium salt, which has certain advantages when used to treat highly porous limestones, does not crystallise well, and is usually replaced by the double salt of aluminium and zinc. All of these compounds are colourless, and as they yield only colourless products, the appearance of the stone is unaltered by the treatment. Copper and other coloured silicofluorides have been employed in the production of coloured surfaces on stone, but of the use of these materials I have no experience. The hardness of the limestone is increased considerably by the treatment, as is easily observed when the surface is scratched with a knife.

The silicofluorides, or "fluates," as they are called in commerce, have been manufactured at Clermont-Ferrand, where extensive deposits of fluorspar occur, and it does not appear that the industry has been established elsewhere. They

are transported in the solid state, and only dissolved when actually required for use. The method of protecting stone by this treatment is largely employed in France, but is rarely referred to in discussions on the subject in this country, which is my reason for communicating this short note to the Society.

## London Section.

*Meeting held at Burlington House on Monday, March 25th, 1918.*

DR. CHARLES A. KEANE IN THE CHAIR.

### SOME COTTON SEED PRODUCTS IN THEIR RELATION TO PRESENT-DAY NEEDS.

BY ED. C. DE SEGUNDO, ASSOC. M. INST. C.E.,  
M.I. MECH. E., M.I. E.E.

(Abstract.)

For many decades past, economists have drawn attention to the grave danger of our increasing dependence upon foreign countries for the necessities of life and for some of the raw materials indispensable to the welfare of our great staple industries. These economic problems have reached an acute stage as a direct consequence of the world-war, and we have good reason now to deplore the fact that we have to draw some 80% of our wheat supplies from overseas, and that only 20% of the cotton required for our spinning industry is obtained from regions within the British Empire.

The fact that Lancashire is dependent upon the United States crop for 80% of its cotton is serious enough, but the menace to the prosperity of our spinning industry is accentuated by the fact that within comparatively few years, the proportion of the American crop spun in American mills has risen from 36% to over 54%.

There can be no question as to the vital necessity of taking steps forthwith to increase the cultivation of cotton within the Empire; and as the production of cotton seed is an inevitable concomitant of the growing of cotton, it is of interest to draw attention to certain products obtainable from cotton seed—and particularly those of the woolly variety—which would serve:—

1. To provide a grade of edible oil admittedly superior to that obtained by the method now practised in Great Britain in crushing cotton seed, and admittedly unrivalled for the manufacture of margarine and artificial lard;\*

2. To provide a flour, containing over five times the protein and fat content of wheat flour, the employment of which would enable us to reduce our wheat imports; and

3. By turning to account the short cotton fibres remaining upon the shell of the woolly cotton seeds, to provide a supply of raw material for the manufacture of explosives, paper, etc.

Probably upwards of 95% of the cotton grown in the world is of the "Upland" or "woolly" variety, frequently referred to as "rain-grown" because it is cultivated under ordinary climatic conditions, *i.e.*, without recourse to irrigation. This seed is covered with an under-growth of "fuzz" or short cotton fibre in addition to the long staple cotton.

Under certain conditions, naturally obtaining or artificially produced, a variety of cotton is grown, which, upon being ginned, leaves the seed "bald" or "black." Practically all the cotton is of long staple and removable in the ginning process. The quantity of such cotton grown

\* L. Kessler, Comptes rendus, 1883, 96, 1317. Faure and Kessler, Eng. Pat. 4954 of 1883; this J., 1884, 414.



is very small compared to the world's crop of some 21,000,000 bales. The greater portion of such seed comes from Egypt. A very small quantity is grown in the United States.

*American and British systems of crushing cotton seed.*

(a) *British practice.* According to the British practice, the cotton seed—whether it be of the bald or of the woolly variety—is crushed whole just as it is received. The oil is consequently impregnated with the tarry compounds and deep colouring matter contained in the shell, and in spite of recent advances in the processes of refining and deodorisation it is difficult to remove these impurities; indeed some authorities state that they can never wholly be got rid of.

The residue after expression of the oil, consists of the kernel-remains, the shell, the "fuzz," and a certain amount of dirt and foreign matter, forming a cake containing about 4½% to 5% of oil and used only as a food for adult cattle. The presence of the "fuzz" detracts, moreover, not only from the appearance, but from the food value of the cake.

In pre-war times, Egyptian seed fetched from £2 to £3 more per ton than the woolly varieties, and the cake made from Egyptian seed sold at 20s. to 30s. more per ton than that made by the British method from woolly seed.

(b) *American practice.* The system adopted practically throughout the United States is that known as the "decorticating system." The cotton seed is run through a "huller" the function of which is to split open the shell of the seed and free the kernel. The mass of broken shell ("hulls") and kernels ("meats") issues from the "huller" and falls upon a shaking sieve, through the meshes of which the meats can pass easily. The hulls matt together by reason of the fuzz upon them, pass over the shaking sieve, and are carried away to a suitable store.

Since the fuzz absorbs oil very readily from the naked kernels in the hulling machine during decortication, it is customary in American practice first to remove a portion of the short cotton fibre in a linting machine. If too much of the fibre be removed, however, pieces of the hull will be more liable to fall through the shaking sieve together with the meats. In first-class practice (in pre-war times) an efficiency of separation of the meats from the hulls of 98% and even of 99% has been claimed with up-to-date decorticating machinery.

The residue of the meats after expression of the oil is called "decorticated cotton seed cake" and is sold as such or is ground to a meal, when it is known as "cotton seed meal." From this meal the cotton seed flour for human consumption is prepared.

During the early years of the development of the industry in the United States, the "hulls" were looked upon as a waste product and were frequently used as a fuel but more often burnt to dispose of them. Subsequently it was found that they could be used as a roughener in the manufacture of feed for cattle—indeed, large quantities of hulls have been fed direct to cattle.

Of recent years, means have been devised for the mechanical separation of the hulls into their component parts of cotton fibre and shell-bran, the former being employed for the manufacture of explosives, paper, artificial silk, cellulose acetate, and other cellulose derivatives, and the latter as a basis for the production of various forms of mixed feeds for cattle.

When the process of decortication is efficiently carried out and a practically complete segregation of the meats (kernels) achieved, the oil expressed is very pure and easily refined, the loss on refining being from 5% to 9%. On the British system, this loss may be taken as from 11% to 15%, and,

in some varieties of Indian seed, the loss is stated to be from 16% to 20% and even higher.

Twenty years ago the decorticated cake made in the United States contained as much as 10% of oil. In good (pre-war) American practice it was between 6% and 7%. Since the war broke out, American oil millers have succeeded in further reducing the percentage and to-day some mills turn out cake containing little more than 5%.

In first-class British practice, the percentage of oil in undecorticated cake is from 4.5% to 5%. The percentage of decorticated cake made (calculated upon the weight of the seed as received by the mill) is from 40% to 45%, depending upon the degree to which the seed is linted before decortication. The weight of undecorticated cake, on the British system, is 85% or more.

From these figures it will be seen that as the ratio of the weight of undecorticated cake to decorticated cake is greater than the ratio of the percentage of oil left in the decorticated cake to that left in the undecorticated cake, the percentage of oil expressible, on the weight of seed, by decorticating must be higher than that obtainable by crushing the seed whole (British system)—subject to a small correction for oil left in hulls. The following figures will serve to illustrate this point:—The average production of cake and hulls in the United States expressed as a percentage of the weight of the seed crushed, was 43.7% cake and 33% hulls for the years 1910-11, 1911-12, 1912-13 (calculated from the returns of the United States Census Bureau). Taking the percentage of oil left in decorticated cake as 6%, that in the hulls as 0.7% (average for seven years), that in undecorticated cake as 4.75%, and taking the total oil content in average Upland American seed as 18%, the oil expressed from 100 tons of such seed efficiently decorticated would be 15.15 tons. If these hundred tons had been crushed on the British system, it may be calculated that the amount of oil expressed would have been 13.9 tons, or 1.25% less than with efficient decortication.

The presence of shell in the undecorticated cake imparts astringent and other properties to the cake which are not without certain advantages, particularly in the feeding of milch cows at certain times of the year, but this circumstance can, at best, only be urged in extenuation of the British system, not in praise of it. On the improved American system, the defibrated hulls are recovered as a separate product and can, together with other suitable ingredients, be compounded with the kernel-remains of cotton or other seed to produce a balanced ration. The "compounding" of the kernel-remains with the shell and other substances entails of course some additional expense, but apart from the better price such "balanced feed" should command, the cost of grinding and mixing should be more than off-set by the profit on the short cotton fibres recovered from the decorticated hulls of woolly seed, not to speak of the increased value as a roughener.

*Edible oil for the manufacture of margarine.*

Cotton seed oil obtained by the American method, i.e., expressed from the kernels alone, is univalued for the production of the highest qualities of margarine and the oil is easily refined with a smaller loss and at a lower cost than that expressed by crushing the seed whole as on the British system.

*Cotton seed flour as food.*

The credit for the introduction of cotton seed flour as an article of food for human consumption is due entirely to the late Colonel J. W. Allison, one of the pioneers of the cotton seed oil industry in the United States. He first directed attention to the possibilities of decorticated cotton seed meal as a food for human beings in 1876. He stated



recently that cotton seed flour is twenty-five times as nutritious as potatoes, five times as nutritious as corn (maize) meal, and four times as nutritious as oatmeal.

Choice cotton seed meal must contain (according to the rules adopted by the Memphis Merchants' Exchange) not less than 49% of combined protein and fat. The average analysis (before the war) is given as 45% crude protein and 6% fat. Owing to the rough and ready manner in which the cake is handled in the mill, impurities and foreign matter are liable to be introduced, and the meal ground therefrom must therefore undergo certain purifying processes in its conversion into cotton seed flour for human food.

The serious attention of the United States Government is being given to the utilisation of cotton seed flour as a means of mitigating, in some measure, the deficiency in the supply of wheat flour and also as affording, in some measure, a substitute for meat.

The composition of cotton seed flour will naturally depend upon that of the meal from which it is produced. Some samples contain as much as 66.64% of crude protein and fat (of which 10% is said to be fat). G. S. Fraps gives the composition as 51.19% crude protein, 11.40% fat, 22.22% nitrogen-free extract (carbohydrates), 3.05% crude fibre, 7.21% water, and 6.0% ash. It is stated that the nitrogen-free extract of cotton seed meal is composed most largely of raffinose and that it contains no starch.

Messrs. Cross and Bevan give the following analysis of imported cotton seed flour:—Albuminoids (N  $\times$  6.25) 50.40%, oil and fat 8.77%, digestive carbohydrates 22.79%, crude fibre 4.50%, moisture 8.32%, ash (mineral matter) 5.16%. They report that the sample may be described as a starch-free seed-flour.

It may be said that cotton seed flour contains no starch, about 54% crude protein, and from 7 to 10% of fat, while wheat flour contains a high percentage of starch, from 10 to 12% crude protein, and from 1½ to 2% fat. The coefficient of digestibility of the protein in cotton seed flour is given as about 88% and that of the protein in wheat as about 94%. On the other hand, the digestibility of the fat in the cotton seed flour is about 93% as compared with 90% for the fat contained in wheat flour.

The protein and fat content of cotton seed flour is about 5 times that of wheat flour. It cannot therefore be substituted entirely for wheat flour in making bread (except perhaps under medical advice in cases where a starch-free diet is necessary), but it is eminently adapted for use as a diluent for wheat flour up to one-fourth or one-third of the wheat flour. Trial bakings made in London with various mixtures of cotton seed flour, potato flour, and wheat flour indicated that the best all-round proportions were about 25% of cotton seed flour, about 69% of wheat flour, and about 6% of potato flour (percentages calculated on solids only). Taking the moisture in these loaves at 25%, the crude protein content would be about 16.9% by weight, which is about twice that in an all-wheat loaf, and, indeed, approximates to that of high quality lean meat.

A great number of experiments on feeding cotton seed meal to animals have been made and it is well known that cattle thrive upon a properly balanced cotton seed meal ration. Experiments on the dietetic value of cotton seed flour for human beings have not been carried out for long enough periods of time to enable final conclusions to be drawn, but so far as such experiments have gone, there would seem to be no objection whatever to cotton seed flour as a food for human beings, provided it be intelligently administered.

According to an American authority (Dr. de Loach) it would be safe to supply 20% of the

protein we daily consume (or should consume) from cotton seed flour. A mixture of 5% cotton seed flour, 10% potato flour, and 85% wheat flour (percentages calculated on the weight of solids only) would yield about 9% protein on the total weight of the bread, including water.

To produce 350,000 tons of cotton seed flour (which is 5% of our annual consumption of 7,000,000 tons of wheat flour) would require the decortication of about 1,200,000 tons of cotton seed. Bearing in mind that the kernel occupies about 50% of the volume of the whole seed, our British cotton seed oil mills, which now crush upwards of 500,000 tons of cotton seed per annum, would, by suitably modifying their press boxes, be able to deal with 1,200,000 tons per annum (if the decortication system were employed) without interfering with the treatment of the other oil-bearing seeds now crushed.

With the production of 350,000 tons of cotton seed flour from Upland seed by the decortication system, there would be produced about 170,000 tons of refined cotton seed oil suitable for the best grades of margarine, about 2,500,000 bales of cotton for spinning, about 60,000 bales of high grade linters, and about 250,000 bales of hull fibre and seed lint. The Anglo-Egyptian Sudan would be suitable for the growth of the necessary cotton seed for these products, but it must be borne in mind that the cost of putting the 2,500,000 acres that would be necessary under cotton alone would involve large sums of money, and that the labour required could only be supplied by a population many times greater than that of the Anglo-Egyptian Sudan to-day.

The admixture of 5% cotton seed flour and 10% potato flour with the wheat flour employed in making our bread would reduce our wheat imports by about 1,050,000 tons, or by nearly 20%.

#### *Utilisation of the short cotton seed fibres on the woolly varieties of cotton seed.*

Until recently, these short fibres were entirely neglected, but they are now successfully separated from the hulls by a mechanical process. The short fibres (or hull fibres) have already been employed in very large quantities by the Du Pont de Nemours Powder Company in America, for the manufacture of explosives, and before the outbreak of war, increasing quantities were being annually employed by papermakers in Great Britain and in France in substitution for rags and textile wastes for the manufacture of the higher grade qualities of paper.

Quite recently another machine has been introduced which, it is claimed, will detach the shorter fibres from woolly seed that has already been lightly linted and deliver the fibre in so clean a state that the yield of cellulose obtained therefrom approximates to that of raw long cotton.

It may be asked why the American system has not been adopted in Great Britain. One answer to this question is given in the recently published Bulletin of the Imperial Institute (No. 3 of Vol. XV., July—Sept., 1917) in the following words:—“The reason of the backward condition of the oil and seed crushing industry (in Great Britain), taken as a whole, is mainly that it began in the days when oil-cake for feeding cattle was the chief desideratum and the oil a relatively unimportant material, and this has largely determined the kind of seeds crushed.”

It is certainly the fact that, speaking generally, British crushers have devoted their attention in the past chiefly to oil seeds giving a comparatively low yield of oil and a high yield of cake. One obstacle to the adoption of the American system by British seed crushers is to be sought in the difficulties attending any attempt to introduce products differing in appearance or character from



those for which the demand has been long established. On the other hand it appears that the technical questions involved in modifying the oil mill equipment do not present any serious difficulties, nor would the capital outlay on such modifications constitute a matter of governing importance.

In recent years, and in particular since the outbreak of war, the whole situation has been greatly changed. The demand for oils for the manufacture of margarine and other edible fats which sprang up some ten years or so ago on the Continent, was not at first reflected in this country. The German seed crushing industry, being of comparatively recent origin, commenced with the advantage of up-to-date equipment. Special attention was paid to copra, palm kernels, ground nuts, and other seeds having a high oil content, and in the result, German seed crushers "have not only monopolised to a large extent the crushing of rich oil seeds but have also made inroads into the crushing of cotton seed which, so far as Europe is concerned, had been until the last few years (before the war) practically a British monopoly."

The extent of these inroads may be gathered from the fact that, in 1905, 94.4% of the exported Egyptian crop came to British mills and in 1913 the proportion had sunk to 51.6%, the remainder going practically entirely to Germany.

At first sight, it would seem that the era of working cotton seed is drawing to a close. This is, however, not necessarily the case. Let it be conceded that, in the future, oil will be the primary and cake the secondary consideration, and that the richer oil-bearing seeds will come more and more into demand, the fact remains that we must have cotton.

The greater the preferential demand for the richer oil seeds, the more will cotton seed tend to become a drug in the market—at least in the export market. Nevertheless, the intrinsic value of the high grade oil and of the protein and fat content of the decorticated cake, will remain unaltered, and the rise into favour of the richer oil seeds should therefore operate to stimulate activity in the crushing of cotton seed in the countries of origin. The pure oil will always hold its market and the cotton seed cake or meal, or the cotton seed flour obtainable from the pure kernels, should always command a market at a price based upon its high protein and fat content. In view of the fact that the recovery of the short cotton fibre, before and after decortication of the seed, is now a commercially established industry, there is no longer any reason why the crusher should hesitate to leave enough "fuzz" on the woolly seeds to ensure high efficiency in decortication and thus to produce the highest grade of oil and cake.

One mill in this country, at least, has under serious consideration the erection of a decortivating plant with a view to the adoption of this system on a large scale upon the conclusion of the war, when supplies of woolly seed may be expected to become available in even larger quantities than hitherto.

For reasons arising partly out of the increase in the home consumption of cotton in the United States, it is probable that after the conclusion of the war, Great Britain will have to provide a means of crushing very much larger quantities of the woolly varieties of cotton seed than have hitherto been dealt with. Thus, the question of the relative merits of the British and the American methods is likely to become an increasingly important one.

The decortivating system, plus the plant for defibrating the seed (before decortication) and the hulls (after decortication) involves a larger capital outlay per ton of seed treated than the British system. On the other hand, the profit realisable from the treatment of woolly cotton

seeds by efficient decortication in combination with judicious linting and defibration of the seed, and defibration of the decorticated hulls, is appreciably higher than that realisable for the products arising from crushing the seed whole, after deduction of the milling costs involved in each system.

In dealing with woolly seed, there can be little question as to the method of milling which should be adopted.

It is clear that, did a decortivating system exist whereby the meats of bald seed could be segregated efficiently from the hull (or shell of the seed) such seed could be turned to more profitable account in that way than by crushing it whole.

Logically, the grading and selection of the seed for planting, the growing and picking of the cotton, the ginning of the seed-cotton, the baling and pressing of the fibre, the linting and defibrating of the seed, the decortication of the seed, the production of oil and cake, the defibration of the decorticated hulls, and the utilisation of the shell-bran should, as far as practicable, be carried out under the control of one organisation in the country in which cotton is grown. Only those practically concerned with cotton cultivation realise what an immense difference it would make if proper attention were paid to the selection of seed and if the production of seed and the milling thereof were in the same hands. Incalculable advantage would also be gained by the country itself, as only such proportion of the products would be exported as were not consumed in the country, the manurial value of the cake fed to cattle alone being an important item from the agricultural standpoint. Having regard to the enormous areas throughout the Empire where climatic conditions favour the cultivation of cotton, there should be room for many such organisations, and opportunities would be afforded for the remunerative employment of practically unlimited capital.

#### DISCUSSION.

DR. BERNARD DYER doubted whether the author was right in attributing the dark colour of the oil obtained from the English crushing method to the presence of the husk of the seed. In a cotton seed there were a number of crimson spots which indicated colouring matter. In the laboratory dark red oil was obtained from decorticated seed. He did not know that it would altogether be to the advantage of the farmer if Egyptian seed were decorticated before it was crushed. The very presence of the husks gave a special value to the cake; it gave an astringent property which was very valuable to cattle feeding on grass at the full height of growth, and the farmer valued it very much for that reason. When the woolly Bombay seed was here crushed whole, it was anticipated that it would be injuriously indigestible, but experience had shown that cattle did as well on it as on Egyptian seed cake, allowing for the slightly less value. It was more than 40 years ago that he first became familiar with decorticated cake when he was working under the late Dr. Voelcker. This cake contained sometimes as much as 50% of albuminoids; if the seed was squeezed sufficiently the oil might be 10%, but was usually from 12 to 15%. That most valuable cake, however, had for a number of years ceased to appear in England, but instead there were enormous quantities of American so-called decorticated cotton seed meal. It was an extraordinary thing that really decorticated cake did not come here if the old process was still worked. The meal now sent usually contained something like from 33 to 37% of albuminoids, and though it was excellent food he thought it should be called "partially decorticated" cotton seed meal. He had been assured that the large proportion of hulls in this meal was not due to the re-admixture of



hulls with the decorticated cake but was due entirely to the fact that owing to the better process of de-linting they were no longer able to decorticate the seed properly. He had supposed that the fraction which was called the "bran" was mixed with the seed, but he had been assured that that was not the case. Could the author tell them what really was the case? Was the process carried out as he had described or was it only occasionally carried out for special purposes in its original form?

MR. ERWIN W. THOMPSON, after outlining the methods of decortication and of the extraction of the oil from cotton seed in use in the United States, said that progress in the art of milling, looking towards a greater yield of oil, as well as changes in the relative market values of the products, had brought about many changes in the character of the products. At first efforts were made to produce pure cake entirely freed from the hulls. In sifting the cut seeds with this end in view, much of the kernel was inevitably carried away with the hulls. As manufacturing profits began to diminish, it became necessary to conserve these wasted kernels, but in sifting for this latter result much of the hulls would inevitably be run in with the kernels. Thus the resulting cake would contain less nitrogen than formerly. Just before the war, average merchantable American cake would contain about 40% crude protein and 7% fat, compared with 50% and 12% in the "pure" cake as at first made. Since the beginning of the war, cake had degenerated still further, say to 30% and 5%, thus approaching the composition of the Egyptian cake made in England. This condition had been indirectly brought about by the war demand for cellulose and raw materials from which to make it. A certain amount of fuzz or short lint left on the hull was a *sine qua non* for good decortication by the American system. Now, in the effort to produce the greatest amount of fibre to meet the war demand, the seeds were being more closely defibrated before cutting, and hence the smooth hulls passed through the sieve along with the kernels. Meanwhile the quality of the oil pressed from these hully kernels was also depreciated, but the demand for fats of all kinds kept the price high and the buyer uncritical. After the war there would probably be a reaction in the United States toward cleaner decortication; but if the demand for fibre continued, as it probably would, the mills would have to meet the situation in one of two ways, *v. z.* :—

(1) By the invention of some new way of decortication for the separation of denuded shells from kernels, or (2) the division of the production of fibre into two operations, first the removal of only a portion from the seed before cutting (leaving enough fibre to ensure perfect decortication according to the old method) and then removing the rest from the hulls after they had been separated from the kernels. If the demand for cottonseed flour should rapidly develop for ameliorating the wheat shortage, the return to better decortication would probably begin very soon in the United States, because good flour could not well be made from seed worked whole. Cottonseed flour could be made from the woolly seed which constituted about half of the British crush of cottonseed, by installing standard American machinery and methods of decortication, and by altering the boxes in their hydraulic presses, but for the bald Egyptian seed some new method would have to be invented to separate the kernel from the shell or hull. No such method had heretofore been installed because the demand for decorticated cakes in England had not been important. In fact some British millers went so far as to say that the British cattle feeders actually preferred undecorticated cake. That, however, was an artificial and unscientific con-

dition and did not exist to any extent outside of the British Isles. The demand for undecorticated cake had grown up, not because of its excellence, but because of the large home supply. New conditions which were now developing in England in the supply of oil cakes would eventually develop a new demand. Pressing palm kernels was one of the industries which England had taken away from Germany, which formerly had a practical monopoly of it. Copra, soya beans, and peanuts were also being pressed in England in greater quantities than heretofore. Cakes made from all these new materials must be sold and fed just as they had always been to such great advantage in Germany. Undecorticated cakes made in Germany were not fed there but exported to England. Soya and peanut cake had about the same composition as decorticated cottonseed cake, so that a demand once established for any one of these cakes would be easily satisfied with either of the others. But whether Egyptian seed were ever decorticated in England or not, it was quite probable that woolly seed would be, particularly if there were to be an augmented supply of these products from Uganda, Nigeria, and the Sudan.

MR. E. T. BREWIS asked what was the percentage of oil in the kernels, as distinct from the total oil contained in the seed, and also what was the minimum amount of oil that could commercially be left in the cake.

Capt. C. J. GOODWIN asked for more information as to the manufacture of artificial silk. How did the raw material compare as regards cost and yields with the materials usually employed, and what process had been found most suitable for its manufacture from the cotton seed fibres? With regard to the amount of oil left in the cake referred to by Mr. Brewis, this was from 5 to 7% in the American system and from 4½ to 5% in the case of the British system. Was it intentional that there was more oil left in the American cake or was it due to bad crushing?

DR. R. SELIGMAN asked if the figures given for the loss in refining were strictly comparable, *i. e.*, whether they were records for the same year and for the same season of the year; also to what extent the refining was taken in each case, whether to the "soap oil standard" or to the "finely deodorised edible oil standard."

MR. P. J. NEATE asked as to the possibility of installing decortication plant where the seed was grown, dealing with it on the spot and then importing the kernels instead of the seed. He also endorsed the possibility of winnowing the seed instead of sifting it.

MR. E. C. DESEGUNDO, replying to the discussion, said that the reason that the decorticated cake was now different from formerly was not because the decortication system, *qua* system, had been interfered with, but because so much of the short fibre was now being taken off the seed, especially since the war started. The shell was thus so denuded of fibre that the pieces could not lap together and a portion of the shell fell through with the seed and became crushed with the cake. He granted that the shell gave an astringent quality to the cake but that fact could only be urged in extenuation of the British system and not in praise of it. The seeds could very well be decorticated and then some of the shells mixed with the cake in any proportion desired. As to the kind of oil and the standard of refining, the figures were fairly comparable so far as he was aware. Twenty years ago decorticated cake contained from 10 to 11% of oil. The American oil miller prided himself in those days on the efficiency of decortication and often got as much as 98% of kernels. His statement that cottonseed flour had five times the value of wheat was based on the fact that it contained from 60 to 66% of crude protein against 12% in wheat. He agreed that



there was no starch in cotton seed and that there was in wheat, but in the comparison in the paper that had been taken into account as the calculation was made in calories. The object of the paper was to indicate that if the 600,000 tons of cotton seed that was crushed in this country to-day were decorticated, there would at least be some additional flour. The percentage of oil in cotton seed varied from crop to crop, and it was only possible to work on rough averages. He had known it vary from 18 or 19% to from 30 to 35%. In the naked kernels it had been as high as 38%. The minimum that could be left in the cake was about 5%; he did not think it could be got lower. That made a very hard cake. He had been told that on the British system, using Bombay cake, it had been brought down to 31%. With regard to artificial silk, within the last two years there had been great conflict of opinion among experts as to whether better results were obtained with wood cellulose or cotton cellulose, but it had recently been definitely decided that cotton cellulose gave the best results as regards lasting qualities and water-proofing. He did not think it would be practicable to import the kernels instead of the seed as the kernels would soon decompose on the voyage.

### Newcastle Section.

*Meeting held at Bolbee Hall on Wednesday, March 20th, 1918.*

MR. H. PELLE IN THE CHAIR.

#### THE DELETERIOUS EFFECT OF USING SALINE OIL COKE IN THE MANUFACTURE OF CARBON ELECTRODES.

BY H. SCRAGG.

The object of this note is to show the adverse influence of the presence of salt in the manufacture of amorphous carbon electrodes where high density and purity are the essential features aimed at. The saline oil coke is chiefly derived from Russia, presumably from around the Black Sea, and contains from 6 to 7% of sodium chloride. Scottish oil coke, however, is free from such impurity and gives rise to no trouble. The following is an analysis of saline oil coke:—Moisture, 2.89%; volatile matter, 4.65%; insoluble ash, 0.80%; sodium chloride, 6.40%; and carbon (by diff.), 85.26%. It is obvious from the analysis that the sodium chloride can very readily be washed out by suitable means with hot water, and it is possible that, during calcination, a loss of sodium chloride would occur. That, however, is based on the supposition that the temperature attained in a furnace would be as high as usual, but it has been found that the presence of sodium chloride seems to prevent such a high temperature being attained as with saline-free oil coke, when the temperature would be as high as from 1400° to 1500° C., the lowest being from 1100° to 1200° C. The highest temperature recorded when calcining saline oil coke was 1100° C., whilst, in parts, it was as low as 880° C. That circumstance has an important bearing on the question, as the volatile sodium chloride would not be expelled but would merely pass from one portion of the furnace to another, finally condensing on the cooler coke at the top or dropping back into the furnace when the lower portion is withdrawn. Instead of a loss of sodium chloride, a process of concentration would go on until the furnace was finally drawn. Moreover, the density of the oil coke is not all that could be desired. In practice, it has been found that the sodium chloride increased from 6% in the original to 8%

in the calcined coke and, at the same time, the insoluble ash rose from 0.80% to 1.2%. The latter was very probably due to the action of fused chloride upon the furnace lining. It would, indeed, be very bad policy to attempt to make electrodes from the calcined material and, apart from washing out the soluble chloride, the only alternative would be to dilute the coke with other oil cokes free from salt, thereby obtaining a mixture containing, say, 2% NaCl. There, again, on mixing the ground coke containing from 1 to 2% NaCl with suitable binding material and pressing the whole into block form and baking, a further set-back is experienced. The density of a finished electrode should be at least 2.00, that is if it is to be at all durable; it would seem that, in the case of electrodes containing salt, the density falls with increasing percentage of that impurity. Thus, with oil coke containing 1.51% of NaCl, the density was 1.95; with 1.63%, 1.92; and with 1.78%, 1.89; whilst another block, baked at the same time but free from salt, had a density of 2.00. The presence of sodium chloride only would tend to make the density more than normal, supposing, of course, that the remainder of the block attained a density of 2.00, so that it is quite evident that the low density must be accounted for in other ways, e.g., low temperature of calcining with saline oil coke, improper baking temperature, or the sodium chloride in the block preventing the escape of volatile matter. In one case an electrode free from chlorides, when baked alongside of some containing salt, attained a density of 2.00. Many of the electrodes when finished are found to be blistered and to have whitish marks on the face; these blisters point to the volatile matter being unable to escape.

#### DISCUSSION.

The CHAIRMAN said he did not know whether the presence of salt in an electrode would have a bad effect in a steel furnace. It was difficult to say to what the bad effect was really due.

Dr. J. T. DUNN thought that it would have been possible to wash out the salt.

The CHAIRMAN explained that all the best oil coke at present was being used in aluminium manufacture. The oil coke used in the manufacture of electrodes was mixed with other ingredients, such as gas carbon and anthracite, the result of which was to give a better product than was procurable from any one sort of carbon.

Mr. F. CAMPBELL remarked that, with reference to steel furnaces, he thought the only trouble would be that salt would damage the bricks and make them "run." It would not interfere with the steel; in any case it would be volatilised before it got into the steel. One of the troubles in making aluminium was that, if the furnace got too hot, there was much more sodium formed.

Mr. W. JONES said that some Scottish coke he had examined recently contained from 3 to 4% of sodium sulphate. Very often it contained as much as 5%.

The CHAIRMAN said that the best cokes made in Scotland were free from salt, but some of the other Scottish cokes contained it.

### Communication.

#### ON THE QUALITY OF GLYCERIN FROM WHALE OIL.

PART I.—AN EXAMINATION OF THE IMPURITIES IN GLYCERIN FROM WHALE OIL WITH REFERENCE TO THE SUITABILITY OF SUCH GLYCERIN FOR THE PRODUCTION OF DYNAMITE.

BY A. H. SALWAY, D.S.C., PH.D.

It is well known that, in recent times, the supply of oils and fats has been so regulated that



the best vegetable oils are reserved for the manufacture of butter substitutes, whilst non-edible oils, including fish oils, are utilised for the manufacture of glycerin and in other industries. In consequence of this system of control, whale oil is now being used to a much larger extent than heretofore in the production of glycerin for explosives. From time to time, however, doubts have been expressed regarding the suitability of whale oil for this purpose, chiefly on account of the possibility of impurities from the oil passing into the glycerin and rendering the subsequent nitration process dangerous. It seemed, therefore, desirable, as suggested by the Ministry of Munitions, that an investigation of the subject should be made with the object of determining the amount and the nature of the impurities in glycerin from whale oil, and also for the purpose of deciding whether such glycerin can be safely employed for the manufacture of dynamite. In accordance with this suggestion, the investigation recorded in this paper was undertaken.

#### 1. The examination of whale oil glycerin for the presence of trimethyleneglycol.

Since whale oil of poor quality is liable to undergo fermentative changes with the possible production of trimethyleneglycol, it was decided to examine a number of crude glycerins from whale oil for the presence of this compound. Accordingly a number of whale oils of different quality (No. 0 to No. 4 quality) were procured. These oils were severally hydrolysed in 100-lb. batches, some by the autoclave method, others by the Twitchell process, and others by alkaline saponification. The aqueous glycerins so obtained were purified by the methods usually adopted in the respective processes of hydrolysis, and then concentrated to a strength of about 80% glycerin. For the estimation of trimethyleneglycol in the crude glycerins, the latter were distilled under diminished pressure, and the specific gravity and apparent glycerol content (acetic method) of the distillate determined. From these figures a sufficiently accurate estimation of the trimethyleneglycol content can be made, assuming that the only substances present are trimethyleneglycol, glycerol, and water. Thus, for example, suppose  $x$  is the true glycerol content and  $y$  the amount of trimethyleneglycol, then:—

$$\text{Sp. gr. of mixture} = 1 + 0.00263x + 0.00053y$$

$$\text{App. glycerol content} = x + 0.81y.$$

From these two equations the unknown factors,  $x$  and  $y$ , can be calculated. This indirect method of analysis fails when less than 1% of trimethyleneglycol is present. In this case, it is necessary to fractionate the glycerin and submit the fraction boiling at 110°–280° C. to analysis as described above. As an illustration of the method the following example is given: A known amount (2500 grms.) of crude glycerin from a No. 2 whale oil was fractionally distilled at 80 mm. pressure, the distillation being continued until the temperature had reached and been maintained for some time at the boiling point of glycerol. The distillate (250 grms.) presumably contained all the trimethyleneglycol which was originally present in the crude glycerin; it was again fractionated under diminished pressure and the following fractions collected:—

a.	Distilling up to 80° C. at 85 mm. ....	75 grms.
b.	"   from 80° to 215° C. at 85 mm. ....	12 "
c.	"   from 215° to 220° C. at 85 mm. ....	50 "

The trimethyleneglycol, if present, would be contained in the fractions *a* and *b*; these were therefore distilled at the ordinary pressure, using a small fractionating column. The fraction 110°–280° C., amounting to 8 grms., was analysed, with the following result: Sp. gr. 1.0324; apparent glycerol 15.6%. Using the figures in the equations quoted above, it is found that the 110°–280°

fraction contains trimethyleneglycol 5.9%, glycerol 10.8%, water 83.3%. This amount of trimethyleneglycol corresponds with 0.02% on the original crude glycerin.

This method of examination was applied to a number of crude glycerins from whale oils of different quality, the results obtained being as follows:—

Whale oil.	Source of the glycerin.	Glycerol content.	Trimethyl- eneglycol.
No. 0	Saponification process	85.0	Nil
0	Autoclave	78.0	"
*2	Twitchell	93.3	0.02
*2	Autoclave	80.0	0.05
3	Saponification	77.5	0.07

\* These were different specimens of No. 2 whale oil.

It is evident from these figures that the crude glycerins contain only a very small proportion of trimethyleneglycol, and as this amount would be still further reduced by distillation, it may be concluded that the effect of trimethyleneglycol as an impurity in dynamite glycerin from whale oil of quality No. 0 to quality No. 3 is negligible.

It yet remained to ascertain what amount of trimethyleneglycol is contained in glycerin from the worst quality whale oil. For this purpose, several No. 4 whale oils were obtained and the worst of them selected for examination. This particular whale oil contained 56% of free fatty acid as well as an excessive nitrogen content, and evidently fermentative changes had taken place, which should be favourable to the production of trimethyleneglycol. A quantity of this oil was therefore hydrolysed by the autoclave process and the glycerin water evaporated to a syrup. For the distillation 1500 grms. was taken, distillation in this case being effected by means of superheated steam under diminished pressure and the vapours condensed in a series of air condensers as in large-scale practice. After about 30% of the mixture had distilled, the distillates in the various receivers were analysed with the following results:—

	Amount.	Sp. gr.	App. glycerol.	Trimethyl- eneglycol (calculated).
1st Receiver	grms. 160	1.1913	81.0	14
2nd "	60	1.0462	24.4	11.0
3rd "	80	1.0011	—	—

From these figures it is evident that fractions Nos. 1 and 2 contain an amount of trimethyleneglycol corresponding to 1.9% of the original crude glycerin.

It may be concluded from these results that crude glycerin from whale oil of very poor quality contains comparatively large proportions of trimethyleneglycol, and is not suitable for the production of dynamite glycerin.

#### 2. Examination of whale oil glycerin for nitrogenous substances.

Whale oil of poor quality frequently contains a considerable proportion of nitrogenous matter, and it is therefore to be expected that crude glycerin from such oils would also be contaminated with nitrogen compounds. Since the presence of these impurities might conceivably lead to complications during the manufacture of dynamite, it was desirable that the matter should be further investigated. Accordingly a number of crude glycerins were prepared from whale oils of varied quality and the nitrogen content of each determined. The nitrogen determination was effected by heating 1–2 grms. of the material with sul-

phuric acid (Kjeldahl), then distilling with alkali and estimating the ammonia in the distillate by means of Nessler's reagent. In the cases where considerable ammonia was present, the ammonia was absorbed in *N*/10 sulphuric acid and the excess acid titrated. Blank experiments were also carried out to correct the results for traces of nitrogen in the reagents. The results of these experiments are given in the following table:—

Source of glycerin.	Total nitrogen.	Glycerol.
	%	%
From No. 0 whale oil—		
By the autoclave process .....	0.025	85.0
"saponific. " .....	0.032	70.0
"Twitcheil " .....	0.076	88.0
From No. 2 whale oil—		
By the autoclave process .....	0.035	80.0
"saponific. " .....	0.012	80.0
"Twitcheil " .....	0.092	88.0
From No. 3 whale oil—		
By the autoclave process .....	0.026	87.0
"saponific. " .....	0.032	77.5
"Twitcheil " .....	0.021	81.0
From No. 4 whale oil—		
By the autoclave process .....	1.10	74.0
"Twitcheil " .....	2.03	82.0

The crude glycerins examined in the above manner were all prepared on a fairly large scale (100 lb.). For the purpose of comparison another series of crude glycerins were prepared on the laboratory scale under carefully controlled conditions. In this case the nitrogen content of both the original oils and the crude glycerins was determined with the following results:—

Source of glycerin.	Nitrogen in the original oil.	Nitrogen in the crude glycerin.
	%	%
No. 0 whale oil, Twitcheil process	0.0014	0.007
No. 3 " " "	0.0042	0.010
No. 4 " " "	0.35	1.35
Cotton seed oil, " "	—	0.004
Palm kernel oil, " "	0	0

It may be concluded from these results that the percentage of nitrogen in the crude glycerin from the best quality whale oil is about the same as that in glycerin from good vegetable oils. It is only when the whale oil is of very poor quality that the amount of nitrogenous impurity in the crude glycerin is at all considerable. It is also evident that a considerable proportion of the nitrogen originally in the whale oil is ultimately found in the crude glycerin. If all the nitrogen of the oil passes into the crude glycerin the percentage of nitrogen in the latter will be approximately ten times that of the former.

### 3. The fate of the nitrogenous matter in impure glycerin during distillation.

As already shown, crude glycerin from poor quality whale oil contains an excessive proportion of nitrogenous impurities. It was, therefore, important to ascertain the effect of distillation on the nitrogenous matter and whether any appreciable quantity of nitrogen passes into the distillate. For this purpose two crude glycerins were examined, viz., a crude glycerin containing a comparatively large proportion (1.1%) of nitrogen, derived from No. 4 whale oil, and a crude glycerin containing 0.032% N, obtained from a No. 3 whale oil. The distillations were effected in a current of superheated steam (300° C.) and at a pressure of 85 mm., the crude glycerin being heated throughout in an oil bath at 230°–210° C. The condensers consisted of three air-cooled vessels in series, followed by a final water-cooled con-

denser; this system of distillation being a close imitation of large-scale practice. The results obtained were as follows:—

	Amount.	Nitrogen content.	Glycerol content.
	grms.	%	%
Crude glycerin from No. 3 whale oil .....	475	0.032	77.5
Distillate in 1st receiver	180	0.015	95.9
Distillate in 2nd receiver	65	0.07	29.0
Residue undistilled ....	200	—	—

The distillate in the first receiver gave a slight opalescence with phosphotungstic acid in the presence of sulphuric acid, whilst that in the second receiver gave a slight precipitate with this reagent.

	Amount.	Nitrogen content.	Glycerol content.
	grms.	%	%
Crude glycerin from No. 4 whale oil .....	1500	1.1	74.0
Distillate in 1st receiver	160	0.53	81.0
Distillate in 2nd receiver	60	0.95	21.4
Residue undistilled ....	920	—	—

Each of the distillates gave a strong precipitate with phosphotungstic acid in the presence of 5% sulphuric acid.

From these results it is clear that some of the nitrogenous matter of crude whale oil glycerins is volatile and that a considerable proportion of it collects in the first receiver containing the best distillate. Thus in the case of glycerin from No. 4 whale oil, the first portions of the distilled glycerin (dynamite glycerin) contained as much as 0.5% of nitrogen in the form of an organic base. It can be concluded, therefore, that crude glycerin containing an excessive proportion of nitrogen (above 0.1%) is unsuitable for the manufacture of dynamite glycerin unless further purification is undertaken.

### The chemical nature of the nitrogenous substances in whale oil glycerins.

Having ascertained that whale oil glycerin contains, under certain circumstances, nitrogenous bases, it became a matter of some interest to consider whether any light could be thrown on the chemical character of these substances. There is no doubt, in the first place, that the basic substances in crude glycerin are derived from the proteins originally present in the oil. During the hydrolysis of the oil, this protein would be converted into amino-acids, the greater portion of which would pass into the crude glycerin. On distilling the glycerin the amino-acids, although not directly volatile, would yield by decomposition volatile bases. We should, therefore, expect to find, in the distillates from such glycerins, the decomposition products of amino-acids. In the case of vegetable oils the amount of protein or albuminous matter is usually very small and the crude glycerin from the oils is practically free from nitrogen. On the other hand, in the case of fish oils, the proteins are usually present in much greater amount, and are partly converted during hydrolysis and distillation into aromatic bases, which have about the same degree of volatility as glycerin and are, therefore, not easily separated from the latter by distillation. Moreover, Gautier and Etard\* have isolated from putrid mackerel a base boiling at 210° C., which they considered to be identical with dihydrocollidine, whilst Conine†

\*Comptes rend., 1881, 97, 263.

†Comptes rend., 1888, 106, 1604.



obtained from cuttle fish a similar pyridine base, and it is therefore probable that poor quality whale oil—and glycerin from the same—would contain pyridine bases.

Attempts were made to obtain some experimental support for the above suggestion that the bases present in distilled glycerin from whale oil of poor quality consist of pyridine derivatives. Two methods of isolation were investigated, viz., direct extraction from the glycerin by means of ether, and secondly precipitation of the base in the form of its phosphotungstate and the regeneration of the base from the precipitate (Barger, "Simpler Natural Bases," p. 118). By both methods of extraction the base was obtained as a dark brown oily substance possessing an odour suggestive of stale tobacco. Efforts were made to isolate from it definite crystalline derivatives with gold chloride and platinic chloride, but the aurochlorides and the platinochlorides produced were indefinite and amorphous. The odour of these bases certainly suggests that they contain pyridine derivatives, but the amount of substance available for investigation was too small to admit of definite identification of these substances.

In addition to the presence of pyridine bases in glycerin from whale oil of poor quality, it is also quite possible that acid amides may be present. Thus the fermentative changes occurring in whale oil would produce, amongst other substances, ammonia and volatile fatty acids (acetic acid, etc.), and these would appear in the crude glycerin as ammonium salts. In the subsequent distillation the ammonium salts would be converted into amides (acetamide, b.pt. 220° C.), which are volatile and would contaminate the distillate.

To test the correctness of this suggestion, a distillation experiment was carried out with glycerin mixture known to contain ammonium acetate. The crude glycerin mixture was made up as follows:—82% double distilled glycerin (free from nitrogen), 8% water, 2½% sodium chloride, 2½% ammonium acetate, and 5% sodium acetate. The resulting distillate (92% glycerin) contained no free ammonia, but when heated with alkalis gave a decided ammoniacal odour. It seems, therefore, safe to conclude from this experiment that part of the nitrogen in distilled glycerin from whale oils of poor quality is in the form of an amide.

#### 4. The possible effect of trimethyleneglycol and nitrogenous substances on the nitration of glycerin.

It has been shown in the previous part of the investigation that distilled glycerin from whale oils is liable to contain as impurities trimethyleneglycol and nitrogenous bases. The question now arises whether these substances would have any effect on the course of the nitration when using such glycerin for the manufacture of nitroglycerin. Considering the matter theoretically, it is very doubtful whether small quantities of trimethyleneglycol would have any deleterious influence on the nitration process. On the other hand, small quantities of nitrogenous bases and amides would possibly induce decomposition of the nitrated mixture with dangerous results, especially as it is known that acid amides are decomposed by cold nitric acid, with liberation of nitrous oxide. Obviously the best method of ascertaining the effect of impurities in the course of nitration would be to carry out actual nitration experiments. Such experiments, however, we have not undertaken, but we have been able to show that, when the nitrogenous bases are isolated from impure whale oil glycerin and then treated with a cold nitrating mixture of sulphuric acid and nitric acid, decomposition with gradual evolution of gaseous matter takes place. It may, therefore, be reasonably concluded that it would be dangerous to nitrate

glycerin containing a moderate amount of such impurities.

#### Conclusions.

1. The best varieties of whale oil (No. 0) yield glycerin equal in quality to that from vegetable oils and may therefore be safely used for the manufacture of dynamite glycerin.

2. Medium quality whale oils (Nos. 1—3) yield distilled glycerin containing minute quantities of trimethyleneglycol and nitrogenous matter. The amount of impurity is, however, too small to affect the glycerin deleteriously.

3. Very inferior whale oils (No. 4 quality) are unsuitable for the production of dynamite glycerin, and should be rejected. Such oils may be recognised by their high nitrogen content and high percentage of free fatty acid and by the fact that distilled glycerin prepared from them gives a precipitate with phosphotungstic acid in the presence of 5% sulphuric acid. In general, if a sample of glycerin responds to the latter test, it should be rejected.

## ON THE QUALITY OF GLYCERIN FROM WHALE OIL.

### PART II.—AN INVESTIGATION OF THE FACTORS INFLUENCING THE QUALITY OF CRUDE GLYCERIN WHEN MANUFACTURED BY THE TWITCHELL PROCESS.

BY L. V. COCKS AND A. H. SALWAY.

In the first part of this investigation the quality of crude glycerin from whale oil was estimated by the proportion of trimethyleneglycol and nitrogenous substances found in the material. In practice, however, the percentage of ash and organic residue is usually taken as a measure of the quality of the glycerin. This method of analysis was applied to the crude glycerins described in the foregoing paper, but it was soon found that, although the organic residue is influenced to some extent by the quality of the whale oil, other factors depending on the process of hydrolysis tend to obscure the results and prevent definite conclusions being drawn. It was therefore desirable that an investigation of the factors influencing organic residue in glycerin obtained by various methods of hydrolysis should be undertaken, and to this end a commencement has been made with the Twitchell process as described in the present paper.

The quality of crude glycerin obtained by the Twitchell process may be influenced by the quality of the Twitchell reagent and also by the quality of the oil employed. The influence of each of these factors on the quality of the glycerin was investigated separately. In all cases, unless otherwise specified, the glycerin was prepared by hydrolysing the oil with 1% of Twitchell reagent in the presence of 2% of sulphuric acid, the mixture being steamed for 8 hours, which usually caused about 70% hydrolysis. To prevent excessive condensation of steam during hydrolysis, the mixture was gently heated from without. The glycerin waters separating at the conclusion of the operation were carefully neutralised with milk of lime, then filtered and concentrated under diminished pressure until the glycerin was about 50% strength. At this stage the liquor was again filtered to remove calcium sulphate and finally further concentrated to 88% glycerin.

#### 1. The influence of the Twitchell reagent.

Some of the organic residue in crude glycerin obtained by the Twitchell process is undoubtedly derived from the Twitchell reagent, and it was therefore of importance to determine what contribution the latter makes to the total organic residue. In most of the experiments recorded

in this investigation the reagent employed was a so-called double saponifier of commercial origin, but in a few experiments another type of saponifier, acid in character and almost entirely soluble in water, was employed. The effect of each of these saponifiers on the organic residue was determined by the following method: A known weight of the Twitchell reagent was mixed with dilute sulphuric acid and the mixture heated with open steam for 8 hours. The liquid was then neutralised with lime, filtered, and the organic residue in the filtrate determined in the same way as in glycerin analysis. The results were as follows:—

Materials used.	Organic residue calc. on original saponifier.
	%
Double saponifier— 1 pt. saponifier .....	5.2
1 pt. sulphuric acid .....	
20 pts. water .....	
Soluble saponifier— 1 pt. saponifier .....	12.5
0.1 pt. sulphuric acid .....	
20 pts. water .....	

From these results a simple calculation will show how much organic matter the Twitchell reagent will introduce into the crude glycerin during hydrolysis. Suppose 1% of reagent is employed and the hydrolysis is carried to 70% free fatty acid, as in the majority of the experiments herein recorded, then the amount of reagent per 100 parts of crude glycerin (88%) is 12.7 parts, of which 5.2% in the one case and 12.5% in the other case passes into the glycerin as organic residue. Hence the amount of organic residue in crude glycerin due to the double saponifier is

might be expected to be inferior to the glycerin from vegetable oils. In order to ascertain the difference between glycerins from a vegetable oil and from whale oil of poor quality, the following experiment was undertaken. Whale oil of No. 3 quality, containing 13.8% of free fatty acid and 0.021% of nitrogen, was hydrolysed with Twitchell reagent in the manner described in the introduction to this paper. The glycerin obtained (88% strength) was compared with the glycerin prepared in a similar manner from refined palm kernel oil, when results were obtained as follows:—

	Extent of hydrolysis.	Organic residue.	Calc. organic residue due to Twitchell reagent.	Difference.
		%	%	%
No. 3 whale oil ....	78% in 8 hrs.	1.23	0.73	0.50
Palm kernel oil ....	74% in 8 hrs.	0.70	0.62	0.08

It is thus evident that there is more organic residue in the crude glycerin from No. 3 whale oil than in the glycerin from palm kernel oil. Presumably the difference is due to the nitrogenous matter in the whale oil. To get a clearer idea of the effect of nitrogenous substances it would be of interest to examine a series of oils of varying nitrogen content, but as most whale oils contain only a comparatively small amount of nitrogen it was decided to test the effect of adding nitrogenous matter to palm kernel oil and then preparing and examining the glycerin from the impure oil. This was done with the following results:—

	Extent of hydrolysis.	Glycerol.	Organic residue.	Calc. organic residue due to Twitchell reagent.	Difference.
		%	%	%	%
Pure palm kernel oil .....	72% in 6½ hrs.	88	0.45	0.64	—
Do. with 0.1% casein (N=0.016%) .....	68% in 6 hrs.	88	0.97	0.68	0.29
Do. with 0.5% casein (N=0.083%) .....	66% in 6 hrs.	88	3.70	0.70	3.00
Do. with 1.0% casein (N=0.166%) .....	51.5% in 7 hrs.	88	9.26	0.90	8.36

0.66% and that due to the soluble acid saponifier is 1.59%.

In order to see whether these figures would be corroborated in practice, several oils were hydrolysed with the two Twitchell reagents, and the crude glycerins (88%) analysed with the following results:—

	Extent of hydrolysis.	Organic residue found.	Organic residue due to Twitchell reagent.
		%	%
1% Double saponifier. { Palm kernel oil .....	74% in 8 hrs.	0.70	0.62
1% Double saponifier. { Refined whale oil .....	78.2% in 8 hrs.	0.59	0.58
1% Soluble acid sapon. { Palm kernel oil .....	63% in 11 hrs.	1.90	1.77
1% Soluble acid sapon. { Refined whale oil .....	68.7% in 8 hrs.	2.40	1.62

It is clear from these figures that good agreement results, except in the last case, and it may be concluded that when hydrolysing a good oil, as for example palm kernel oil, under the aforementioned conditions, practically all the organic residue in the crude glycerin comes from the Twitchell reagent.

## 2. Influence of the quality of the oil.

*Effect of nitrogenous matter.*—It is generally considered that the presence of nitrogenous substances in an oil will seriously affect the quality of the crude glycerin, and, as whale oil is particularly liable to be contaminated with such impurities, the crude glycerin from the latter

method prescribed above, then the following generalisation may be made:—

0.02% N in an oil gives 0.5% organic residue to the crude glycerin.
0.035% N " " " 1.0% " " " "
0.06% N " " " 2.0% " " " "
0.085% N " " " 3.0% " " " "
0.10% N " " " 4.0% " " " "

It was next of interest to see how far these figures agree with the experimental results obtained when hydrolysing whale oil of known nitrogen content. For this purpose, whale oils of No. 1 and No. 3 quality were hydrolysed under the specified conditions with the following results:—



	Free fatty acid.	Nitrogen.	Extent of hydrolysis.	Organic residue.		Found.
				Due to Twitchell reagent.	Due to nitrogen.	
No. 1 whale oil .....	7.4	0.002	78.2% in 8 hrs.	0.62	0.05	0.59
Unrefined No. 3 whale oil .....	13.8	0.021	78% in 8 hrs.	0.73	0.50	1.23
Refined No. 3 whale oil .....	14.0	0.001	—	0.72	0.02	0.68

It is thus clear that the organic residue determined experimentally in the above crude glycerins is approximately the sum of the organic residue contributed by the Twitchell reagent, and the organic residue calculated from the nitrogen content of the oil on the basis of the figures already quoted.

Incidentally the figures show also that the crude glycerin from properly refined whale oil is equal in quality to the glycerin from vegetable oils.

*Effect of oxidation.*—In connection with these experiments it seemed desirable to ascertain whether an oxidised oil would yield a glycerin with higher organic residue than glycerin from an unoxidised oil. For this purpose a No. 3 whale oil (free fatty acid 15.1%) was divided into two portions. One portion was heated to 100° C. and a current of air passed through it at this temperature for 6½ hours, after which it was hydrolysed in the usual way. The unoxidised portion was hydrolysed in the same way for comparison. In the first case the organic residue of the crude glycerin (88%) was 1.09%, whilst the unoxidised oil gave a glycerin with 1.03% of organic residue. The treatment with air, therefore, had no appreciable effect on the glycerin quality, and it may therefore be concluded that whale oil may be exposed to atmospheric oxidation without influencing the quality of the glycerin.

#### Summary and conclusions.

The various factors influencing organic residue in crude glycerin obtained by the Twitchell process have been investigated. It has been shown that the chief factors are:—

1. The quality of the Twitchell reagent.
2. The nitrogenous matter in the oil.

A method is described for determining the proportion of organic residue due to the Twitchell reagent, and figures are given enabling the organic residue due to nitrogenous matter to be calculated from the nitrogen content of the oil. Under good conditions of working the organic residue should be the sum of these two figures.

The authors, in conclusion, desire to acknowledge their indebtedness to Messrs. Lever Bros., in whose research laboratories the investigation was undertaken, and to the Ministry of Munitions, for permission to publish those parts of the research which are of general interest.

## Discussions on Papers.

### UTILISATION OF NITRE CAKE.

Mr. J. N. STEPHENSON, of Montreal, writes as follows:—

In the discussion on nitre cake in the December 15th (1917) issue of the Journal (pp. 1216 A—B), no mention was made of its use in the pulp and paper industry. For some time experiments have been made and some application has resulted in connection with the use of nitre cake to replace part of the alum used in the engine sizing of paper. Substitution to the extent of some 35% of the alum previously used has been claimed to have been effected. Another use would apply to the manufacture of sulphate or "kraft" pulp. In this

process sodium sulphate or salt cake is the material generally employed and during the incineration of the evaporated waste liquors this is reduced to sodium sulphide, other sodium salts being converted to sodium carbonate which is later causticised with lime. The use of nitre cake entails some difficulties which have been satisfactorily overcome by some pulp mills. It will be seen that acid sodium sulphate will furnish only half the sodium sulphide which can be derived from salt cake. It will be further observed that the action of acid sulphate will convert sodium carbonate to sodium sulphate with the liberation of carbon dioxide, which it is not desirable to have in the furnace. The acid will also convert the sodium sulphide into hydrogen sulphide. This action has the further disadvantage of destroying the sodium sulphide, the presence of which is desired in the process. If the action of the nitre cake is allowed to proceed where the gaseous products may freely enter a room in which workmen are employed there is danger of suffocation by the carbon dioxide and of poisoning by the hydrogen sulphide unless the acid character of the nitre cake is first neutralised. Of the two dangers, that from the hydrogen sulphide is undoubtedly the more serious. In fact, several deaths have resulted from improper use of this material. With proper handling, however, nitre cake is entirely satisfactory in the pulp mill.

### RECOVERY OF POTASH FROM BLAST FURNACE GASES.

Mr. E. H. LEWIS, of the Glasgow Iron and Steel Co., sends the following particulars of the amount of potash charged into their blast furnace during the month of October, 1917, for comparison with the figures given by Professor Berry (this J., 1918, 23 T):—

Charged into furnaces.	% K <sub>2</sub> O.	Lb. of K <sub>2</sub> O per ton of pig iron produced.
Iron ores .....	0.13	5.16
Coal and coke .....	0.05	2.15
Limestone .....	0.101	0.81
		8.12
Recovered.		
Slag .....	0.146	1.63
Pitch .....	1.20	2.67
Tube cleanings .....	2.23	0.07
Dirt from washers .....	1.20	0.07
Heavy dust from tubes .....	0.20	1.58
Waste liquor .....	2.49	
	grains per gall.	
Less in running off slag and unaccounted for .....		6.02
		2.10
		8.12

## Canadian Section.

*Meeting held at Montreal on Friday, January 25th, 1918.*

MR. T. H. WARDLEWORTH IN THE CHAIR.

### THE WASTE SULPHITE LIQUOR PROBLEM.

BY BJARNE JOHNSEN.

The pulp obtained by heating wood at high temperature and pressure with a solution of calcium and magnesium bisulphites represents only about 50% of the dry weight of the wood, while the other 50% of the wood substance is contained in the waste liquor, which is not at present utilised in Canada. The utilisation of this waste has always been one of the great problems of the sulphite pulp industry, especially in countries which do not possess large forests, as well as in countries where legislation has prohibited the discharge of waste sulphite liquor into rivers which are too small to afford the dilution of the liquor, which is considered necessary to prevent injury to fish life.

It is not necessary to mention all the processes which have been proposed for the utilisation of waste sulphite liquor during the last 40 or 50 years: it will suffice to say that they involve uses of a most varied nature, such as the manufacture of tanning materials, cattle food, dyestuffs, preservative for eggs, ethyl alcohol, etc. The object of this paper is to report on certain modern processes, which in some places have gained industrial importance and which might be introduced with advantage into Canada.

According to Klason the chief constituents of European spruce wood average as follows:—Cellulose 50%, lignin 30%, carbohydrates 16%, protein 0.7%, resins and fats 3.3%. As most of the analytical work on waste sulphite liquor has been carried out in Europe the figures given below are based upon the composition of the liquor obtained from the cooking of this material. In Canada various species of spruce, fir, and hemlock are used in the manufacture of sulphite pulp. These woods vary considerably in their chemical composition, the cellulose content of white spruce, for example, being about 4 or 5% higher than that of balsam fir or hemlock and the lignin content also varying within 4 or 5% in the different species. The same is true of the carbohydrates and of tannin, the amount of the latter being very low in the spruces and in balsam fir. The composition of the wood also varies in the same tree with the dimensions and the conditions of growth. As to the nature of the substances mentioned above our knowledge is still very limited. Klason considers the lignins to consist of condensation products of coniferyl alcohols and therefore to contain an aromatic nucleus. He also assumes the presence of three ethylene groups and an active carbonyl group which are able to combine with the sulphurous acid of the cooking liquor. On the basis of these assumptions we may consider the main reaction between the lignin complex and the cooking liquor as consisting of an addition of  $\text{SO}_2$  to the lignin complex forming sulphonie acids, the barium salt of which has been isolated from the waste liquor by Klason, who established for this compound the empirical formula  $\text{C}_{10}\text{H}_{14}\text{O}_5\cdot\text{S}\cdot\text{Ba}$ , with a molecular weight of at least 6000, which indicates the formula for lignin to be  $(\text{C}_{10}\text{H}_{12}\text{O}_4)_n$ . The lignin compound is further characterised by a number of reactions showing for instance the presence of methoxyl and acetyl groups. A further action of the liquor in the cooking process

is the hydrolysis of the polysaccharides of pentoses and hexoses which are thereby converted into soluble sugars. The cooking process must be regarded as consisting of a great number of both complicated and simple reactions, which with our present limited knowledge of the constitution of wood we are unable to follow in detail. This fact, as well as the variation in the wood and in the method of cooking, makes it impossible to form any definite opinion with regard to the quantitative composition of the waste liquor, but the composition of the wood substance on the one hand and the purity of the wood pulp obtained on the other hand, as far as we possess methods for determining these factors, give us an idea of the substances which may be expected in the waste liquor.

Waste sulphite liquor has a specific gravity of about 1.05, gives an acid reaction, has a strong reducing power, and contains about 12% of organic and 1 to 1.5% of inorganic substances. The latter originate mainly from the cooking acid but partly from the inorganic substances of the wood, which represent about 0.25 to 0.5% of the dry weight of the wood. Sulphurous acid is present to some extent in the free state and partly so loosely combined with organic substances that it may be removed by heating. Most of the sulphurous acid is, however, present in the form of sulphonie acids. The liquor also contains small quantities of sulphuric, acetic, formic, and oxalic acids as well as traces of citric acid, small quantities of vanillin, furfural, and succinic and protocatechuic acids. A precipitate obtained by addition of animal glue has been used as a reaction for tannin compounds, but the presence of such compounds, at least in any appreciable quantity, is questioned by the tanning industry.

The following summary of the constituents of the waste liquor has been calculated by Klason on the basis of a short ton of pulp:—

	Liquor I.	Liquor II.
	lb.	lb.
Lignin .....	1287	1200
Carbohydrates .....	622	650
Proteins .....	31	30
Resins and fats .....	146	60
Sulphurous acid combined with lignin .....	470	400
Lime .....	204	180
	2760	2520

The theoretical yield of by-products is often calculated upon a recovery of up to 1800 gallons of liquor per ton of pulp, which represents very nearly the total liquor in the digester. As a matter of fact, only about 740 gallons will drain off from the blow pits per ton of pulp, while probably a maximum of 800 gallons could be obtained if the draining were continued for a longer period without the use of any special equipment.

The liquor might now be utilised in one of different ways, either by recovering the total solids as a whole or by isolating one or more of the most valuable constituents. The former method, involving more or less complete evaporation, has been employed at places where a discharge of the waste liquor into the rivers is prohibited. The concentrated liquor is a tar-like product with binding properties, and has therefore been proposed as a binder for powdered materials and for roads. As a result of extensive experiments the U.S. Department of Agriculture classifies concentrated sulphite liquor of specific gravity 1.13 as a temporary or semi-permanent road binder and dust preventive. One half gallon per square yard was found to be effective for a period of six weeks. The cost,



including labour and material, was estimated at \$0.0355 per square yard. Favourable results have also been secured at Queen's University using concentrated liquor as a road-binder. The binding and water-resisting qualities may be improved by emulsifying the liquor with a mineral oil.

It is often desired to remove the lime from the liquor, and this can be effected by the addition of sulphuric acid or a soluble sulphate with subsequent filtration. This is no doubt especially desirable when the concentrated liquor is to be used as a binder for powdered fuel, blast furnace dust, fine pyrites, or foundry core sand. In all these ways the waste sulphite liquor has been used successfully. The hygroscopicity and solubility in water of the binder from waste liquor have been pointed out as drawbacks in its use as a binder for fuel. Several methods have been proposed for the elimination of this disadvantage, such as carbonisation of the binding material at a high temperature or the addition of chromium salts and finally aluminium sulphate. The briquettes may also be immersed in a mixture obtained by heating crude petroleum oil with bitumen or phenols to the boiling point, with strong aeration, until a mixture is obtained which solidifies on cooling.

The product known as "Zellpech" or "Cell pitch" is waste liquor neutralised and filtered through coke and afterwards concentrated to 35° B. or to a solid substance of 10% moisture content. One ton of coal is required to produce a ton of "Cell pitch," the heat value of which is 5700 B.Th.U.

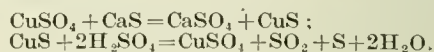
An adhesive can also be obtained if the waste liquor is concentrated after the addition of zinc carbonate or oxide, or if the liquor is concentrated *in vacuo* with an excess of magnesium oxide with magnesium chloride added in a proportion equivalent to the excess of magnesium oxide, in order to form the oxychloride. Usually, however, the adhesives are obtained by precipitation with glue or albuminous substances.

A tanning material can be obtained by concentrating the liquor to about 30° B., but it is necessary to remove certain impurities which are detrimental to leather, such as lime, iron, and the free and loosely combined sulphurous acid. The sulphurous acid can be removed by evaporation, with or without the addition of sulphuric acid, or may be precipitated as calcium sulphite by the addition of the required quantity of lime. The organically combined lime is usually removed by precipitation with sulphuric acid or a soluble sulphate. Oxalic acid is often recommended for the removal of the last trace of lime, or lactic acid is used since the resulting calcium lactate has no injurious effect upon the leather. There are a great number of more complicated methods for obtaining a tanning material from waste sulphite liquor, such as the addition of the sesquioxide of aluminium or chromium, the precipitation of an organic cyanogen compound from the liquor after a previous heating with potassium or sodium cyanide, etc.

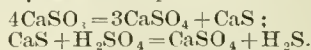
The tanning value of these extracts, known on the market as "sulphite extract," "spruce extract," "Norego," etc., has been the subject of lively discussion. It has been claimed by tanners that these extracts have no tanning value because they do not contain tannin, or at least only very small quantities. This is true, and the reactions obtained from sulphite liquor with glue and with iron solutions, which have been assumed to be reactions of tannin, must be due to the sulphonic acids of the lignin. However, experiments have shown that hide absorbs as much as 23—25% of substances from a "sulphite extract," being transformed into leather which has in some cases surpassed both hemlock and oak leathers in quality. It is not recommended to use these "sulphite

extracts" alone, as the best results are obtained in admixture with other extracts, for example quebracho extract, since the "sulphite extract" is a good solvent for the difficultly soluble portions of the quebracho extract, namely, the phlobaphenes. It is also possible by employing "sulphite extract" in mixture with other extracts to obtain a proper proportion between the tannin and non-tannin substances, and to accelerate the tanning process. The low cost of "sulphite extract" ( $\frac{3}{4}$ —1 cent per pound) results in a considerable saving. The opposition of the tanning industry to this extract may be due to the poor quality of the first products put on the market, but it is generally admitted by the tanners that for certain leathers "sulphite extract" may be used with great advantage and, as a matter of fact, tanning extracts from waste liquor are at the present time imported to Canada and used quite extensively in Canadian tanneries.

It was mentioned above that the "cell pitch" obtained by evaporation of the waste liquor had a heat value of 5700 B.Th.U. but even with modern evaporators a recovery of this product for use as a fuel would hardly be economical. Strehlenert's process for manufacturing fuel from waste liquor is based upon the precipitation of the lignin compounds at high temperature and pressure. The decomposition is effected by the sulphuric acid which is formed from the sulphurous acid present in the liquor. According to the first patents the formation of sulphuric acid is effected by oxidation at a comparatively low temperature by the introduction of air or oxygen into the autoclave. The reaction  $4\text{CaSO}_3 = 3\text{CaSO}_4 + \text{CaS}$  takes place subsequently. As copper autoclaves are used copper oxide and finally copper sulphate are formed and the following reactions take place:



The copper therefore causes the direct formation of fresh quantities of sulphur dioxide. It is found, however, that the presence of even small quantities of iron, zinc, manganese, or other hydrogen-forming metals decreases the yield of precipitate considerably. By introducing the air at a higher temperature the oxidation and decomposition take place simultaneously and so rapidly that the action of the metal is eliminated. The formation of  $\text{SO}_3$  at lower temperatures is avoided by introduction of  $\text{SO}_2$  from the sulphite digesters. When the sulphuric acid is formed at high temperatures the reaction may be expressed by the equation:  $3\text{SO}_2 = 2\text{SO}_3 + \text{S}$ , and theoretically no air is required. But the presence of air is of advantage, as it causes the organic compound to be precipitated in a coarser form. The new autoclaves are lined with lead to prevent the reaction between the  $\text{SO}_2$  and the autoclave material. The reactions may then be expressed as follows:—



In this process only 75% of the total solids is precipitated, and Strehlenert concludes that this portion consists of compounds of sulphonic acids, the remaining 25% consisting of sulphonic acid compounds. In his calculations Strehlenert assumes a liquor recovery per ton of pulp of 1200 gallons, or 1800 gallons when diffusers are used. He also assumes the most favourable conditions with regard to yield and to the concentration of the liquor (namely, 17% solids). The operating cost per short ton of fuel in a Scandinavian plant is \$2.88, according to Strehlenert's calculations, including depreciation, labour, and repairs, assuming a recovery of 1200 gallons of waste liquor per ton of pulp in a plant having a capacity of about 20,000 tons of pulp per year. But this fuel is of an inferior quality, containing about 18%



ash and about 50% of water. Considering these two points the cost would be equivalent to \$1.75 per short ton of good coal of 14,400 B.Th.U.

Assuming the concentration of the waste liquor to be 11%, however, and considering the higher cost of labour, etc., in North America, the manufacturing cost of recovered fuel will be equivalent to \$6.80 per ton of good coal of 14,400 B.Th.U. on the basis of a 33,000-ton pulp mill and a recovery of 800 gallons of liquor (328 lb. of fuel) per ton of pulp. On the basis of 1200 gallons of liquor (492 lb. of fuel) per ton of pulp, the manufacturing cost of recovered fuel would be equivalent to \$6.35 per short ton of coal of 14,400 B.Th.U.

This cost could probably be somewhat reduced by the recovery of by-products such as acetone, acetic acid, formic acid, and methyl alcohol, but in the present form this process would hardly be of practical interest to the Canadian industry. Under the conditions, however, which at present prevail in Scandinavia the process is of considerable value. At least one plant is in operation in Norway, and orders have been placed for several plants of the same type in Sweden, Finland, and Russia.

When certain by-products are recovered by destructive distillation the fuel process appears to better advantage. It has been found that distillation of the precipitate gives 56% of charcoal, 18.7% of crude acid, 2.1% of tar, and 23.2% of non-condensable gases and that there are present in the crude acid, on an ash- and water-free basis, 1.0—1.1% of methyl alcohol, 0.12% of acetone, 0.45% of acetic acid, and 0.014% of formic acid. The economy of destructive distillation of the residue has not yet been sufficiently investigated.

A process which has been more seriously considered in America is the manufacture of ethyl alcohol from waste sulphite liquor. The amount of fermentable sugars in the liquor varies considerably since their formation from the lower hexosans of the wood as well as their destruction in the cooking process depends largely upon the strength of the acid and the temperature during the last part of the process. Usually the liquor contains between 1.5 and 2% of fermentable sugars, but in some cases this value may be as high as 2.4%. Klason found a waste sulphite liquor to contain mannose 0.526%, galactose 0.271%, dextrose 1.65%. Of these sugars galactose ferments more slowly than the others, and the highest yields of alcohol obtained, namely, 1.4% by volume of the liquor, corresponds approximately to the mannose and dextrose content. On an average only 1% of alcohol is obtained on a commercial scale, although Landmark claims an average of 1.1% for his process, and the Marchand process is said to give 1.25% of alcohol.

The principle of the various processes of manufacturing alcohol from waste liquor is the same. The free sulphurous acid, which is strongly toxic to yeast, is partly removed by evaporation and partly by neutralisation to a point of acidity favourable for fermentation. The difference in the various processes lies mainly in the method of neutralisation. It is not advisable to neutralise with lime alone, as a large excess is required, causing the destruction of a large proportion of sugars and an undesirable turbidity of the liquor. In the Swedish plants which are operating under Ekström's and Wallin's patents, a smaller quantity of lime with the final addition of calcium carbonate effects the neutralisation. In Marchand's process barium carbonate is used, while in Landmark's only calcium carbonate is employed. The waste liquor does not contain nitrogen and phosphates in the quantity required for normal fermentation, and if yeast extracts which do not

contain the necessary amount of these substances are used they must be added to the liquor in a suitable form. In Ekström's process a "tempered yeast" is added which is prepared to meet the special condition of the sulphite liquor. This is not necessary in Landmark's process; in this case milk or whey is heated with acid, the precipitate of "lignocasein" (which can be used in paper sizing) is filtered off, and the filtrate added to the liquor, which can then be fermented directly with ordinary brewers' yeast. The cost of the milk is said to be covered by the value of the "lignocasein."

Hagglund has calculated the cost of manufacturing 100% alcohol based upon a recovery of 800 gallons of liquor per ton of pulp. His calculations are based on 30,000, 20,000, and 10,000-ton mills in Scandinavia. However, if we assume for Canadian conditions that the costs of plant, labour, salaries, and repairs are 50% higher than in Scandinavia, and if we further allow 15% depreciation and interest, the total cost of one Imp. gallon of 100% alcohol will be \$0.220, \$0.251, and \$0.321 on the basis of a 30,000, 20,000, and 10,000-ton pulp mill respectively.

The manufacturing cost estimated by Landmark for his process is not directly comparable with the figures given above because he bases his calculations on a recovery of 1400 gallons of liquor per ton of pulp and upon a plant connected with a 15,000-ton mill. It is, however, possible to bring these calculations to a certain accordance with those used in the discussion of the previous process whereby the cost of production, including depreciation and interest and cost of handling, would be very close to \$0.19 per Imp. gallon of 100% alcohol, which is appreciably higher than the cost estimated by Landmark himself. With regard to the cost of production of alcohol, reference may be made to an article by Breckler (see J. Ind. Eng. Chem., 1917, 9, 612; this J., 1917, 902) giving the average cost of alcohol from grain for the past five years as high as 17.5 cents per proof U.S. gallon or about 24 cents per Imp. gallon, corresponding to \$0.48 per Imp. gallon of 100% alcohol, which is considerably higher than the cost of sulphite spirit. The crude sulphite spirit contains about 3% of methyl alcohol. The two alcohols can be separated, but this is not necessary as the spirit can be used in the crude form for industrial purposes, as motor fuel, for illumination and cooking, replacing gasoline and petroleum. In its eighth annual report, the Commission of Conservation points out the importance of establishing an alcohol industry as Canada is now dependent upon foreign sources for her supply of petroleum and petroleum products.

The production of sulphite spirit has already reached commercial importance in other countries. In Norway two plants are in operation, while four plants in Sweden are producing one million gallons per year and three more plants are being erected. In Germany 13 plants are being built or are probably now in operation and in the United States two, and in Switzerland one plant is operated. Considered from the standpoint of utilisation of waste liquor this process does not solve the problem completely, as the fermentable sugars represent only 15—20% of the total solids, but the remaining liquor may be used in one of the other processes, for instance, for the recovery of a binding material, or fuel, or for destructive distillation of the dry residue obtained by evaporation.

Waste sulphite liquor has also been suggested for use as a fertiliser, but in this case its value is only indirect as it contains the substances required for a good fertiliser in very small amounts. In mixture with other substances it may be of some value for poor soils because of its content of organic substances which form a good medium for the growth of nitrogen-forming bacteria.



As the amount of solid substance in the waste liquor is very low (only 11–12%), the economy of most of the processes suggested for its utilisation depends largely upon the cost of concentration. A much higher efficiency is claimed for a new type of evaporator which is now built in Sweden and by which the heat of the vapours from the liquor is utilised much better than in the usual multiple effect evaporators. Oman has recently announced a quite different method of concentration, namely, by freezing. The liquor can be concentrated to one-fourth of its original volume much cheaper than by evaporation. No details regarding this new process are yet available, and it would probably only be feasible in countries with cheap water power and high coal prices.

The Canadian sulphite pulp industry consumed in 1916 about 728,000 cords of wood. This means that approximately 1000 tons of wood substance was carried away daily as waste with the liquor, and it shows that it would mean a tremendous saving for Canada if existing processes could be adopted or new methods developed by which this waste could be eliminated or at least considerably reduced in an economical way.

In conclusion, I have to thank Mr. O. F. Bryant, of the Forest Products Laboratories, for the valuable assistance given me in the preparation of this paper.

## THE DETERMINATION OF CELLULOSE IN WOOD.

BY DR. BJARNE JOHNSEN AND R. W. HOVEY, B.S.C.

The value of having a correct method for the estimation of cellulose in wood has been recognised for many years and has resulted in a large number of methods being proposed. It would be beyond the scope of this paper to review all these methods, but those who desire to make a detailed study of them are referred to the original works of Renker\* and of König and Huhn† and to the references which will be found in the textbooks on cellulose published by Schwalbe‡ and by Cross and Bevan§. Generally it may be said of the methods of estimating cellulose that they, like the commercial processes for manufacture of wood pulp, are based upon the high resistance of the true cotton cellulose towards chemical reactions such as hydrolysis and oxidation and the comparatively low resistance of the non-cellulose substances. The disadvantage of these methods, however, is that they either do not effect a complete removal of the non-cellulose matters, or if they do, that the strong reagents employed also destroy part of the cellulose itself. Some of the methods which give a comparatively pure product are too complicated and require too much time.

There is, however, one method which has created more interest than the others, namely, Cross and Bevan's chlorination method. This method has been extensively used in scientific investigations and generally adopted by the industry. Its advantages are that the chief reaction, the chlorination of the lignin, has been comparatively well studied and is in itself a colour reaction which indicates when the purification is completed and finally that the method is carried out in a comparatively short time. The chlorination method has in recent years been considered to give the most satisfactory results, but the method has been carried out in so many different ways in the various investigations that the values obtained are hardly comparable. In an analytical study of the various Canadian wood species used in the paper industry

it was considered of significance to be able to determine the cellulose content with the greatest possible exactness. However, in adopting the chlorine method in this work it was intended by taking advantage of recent modifications and attempting further improvements to establish a standard method which could be recommended for future investigations.

The action of chlorine gas upon moistened vegetable fibres was originally studied by Cross and Bevan\* in their investigation on jute fibre. They proved that with jute, which they consider as the typical form of "lignocellulose," the reaction with chlorine gas represents a chlorination of the lignin groups with the formation of a lignin chloride of the empirical formula  $C_{19}H_{15}Cl_2O_9$ , a yellow product which is soluble in absolute alcohol, in alkali, and in sodium sulphite. When sodium sulphite is added to the chlorinated product a red colour is at once produced, which can be considered as the most typical lignin reaction and therefore represents an excellent test for the degree of purification during the operation of the method, whereby unnecessary prolongation of the chlorination can easily be avoided. With jute the chlorination is apparently a simple replacement of hydrogen by chlorine without any secondary reactions, as it was stated by Cross and Bevan that hydrogen chloride was formed in the process to the same extent as chlorine was fixed to the lignin complex. With wood, considerably more chlorine is used for the formation of hydrogen chloride than is used in the formation of lignin chloride. This fact was observed by Cross and Bevan and it has been generally believed that this was caused by an oxidation of the cellulose. But Heuser and Siebert†, who studied this reaction with spruce wood more closely, and who confirmed Cross and Bevan's results, were able to prove that the lignin could be completely removed by the chlorination process without any oxidation of cellulose, and that therefore the large excess of hydrogen chloride must be due to reactions with chlorine, probably oxidation of non-cellulose substances. If, on the other hand, chlorine gas is allowed to act upon the cellulose residue for any length of time after the lignin has been removed, the cellulose is gradually transformed into oxycellulose, and this reaction is accompanied by a slight loss in weight of the substance when subsequently heated with sodium sulphite. Accordingly it is important to reduce the time of chlorination to a minimum. The chlorine gas reacts in the first place with the surface of the fibre, forming a layer of lignin chloride which very soon prevents the gas from acting on the interior of the fibre, and unless the lignin chloride is first dissolved a prolonged exposure to the gas has no favourable effect. The removal of the total lignin can accordingly only be effected by intermittent treatments with chlorine and sodium sulphite. This was shown distinctly by Siebert and Walter‡, who stated that the lignin could be totally removed from finely divided spruce wood in four chlorinations with a total time of exposure to the gas of one hour, if the periods of each chlorination were limited to 20, 15, 15, and 10 minutes, the chloride being dissolved in sodium sulphite after each period. This was found by the present authors to be true with all the Canadian species which have been analysed up to date. This is naturally only possible when the material used is a fine and uniform sawdust, which should be of such a fineness as to pass an 80-mesh sieve but not a 100-mesh sieve. The sawdust can easily be obtained by rasping the wood with a suitable wood rasp. The apparatus used for the purpose in this laboratory consists of two wood rasps fastened

\* "Ueber Bestimmungsmethoden der Cellulose," Berlin, 1910.  
† "Bestimmung der Cellulose in Holzarten und Gespinnstfasern," Berlin, 1912.

‡ "Die Chemie der Cellulose," Berlin, 1911, p. 613.

§ "Cellulose," London, 1916, p. 94.

\* "Cellulose," 1916, p. 101.

† Z. angew. Chem., 1913, 26, 801.

‡ Papier-Fabr., 1913, 11, 1179.

to a support to which a water motor gives a reciprocating horizontal movement of about 6 inches on full stroke. The wood is held in a jacket and pressed down on the wood rasps by means of lead weights. The sawdust drops into a box below.

Since Schorger\* found that the final cellulose residue still contained fragments of wood that were only partially reduced, and therefore gave a strong methoxy reaction, it is not surprising that his values for cellulose were extremely high and that great variations in the results were experienced. This could no doubt have been avoided by using a more uniform material. Referring to Heuser and Sieber's investigation, mentioned above, the authors are unable to agree with Schorger when he says that "in obtaining the cellulose absolutely free from lignin the error involved by destruction of the cellulose is out of all proportion to that caused by the presence of small quantities of lignin."

Variations in the results obtained by the chlorination method may be partly due to the mechanical handling of the material during the process of purification. According to the original method the fibrous material was transferred from chlorination flask to crucible and *vice versa* several times, which involved a possibility of fibre losses even with very careful handling. Schorger and Renker used this method of operation. Dean and Tower† were the first to propose that the fibres be left in the same container during the entire operation, and the arrangement suggested by Sieber and Walter, which is based upon this principle, is a great step towards the perfection of the method. As in recent investigations use has not been made of this improvement, which is in our opinion extremely valuable, the preparation of the Gooch crucible as suggested by Sieber and Walter is described here. A perforated porcelain plate, A (see fig.), which fits well to the bottom of the Gooch crucible, is sewn with cotton thread between two pieces of fine bleached calico cloth, B, and the projecting parts cut off close to the edge of the plate. After the plate has been purified by boiling with alcohol and water it is fastened in

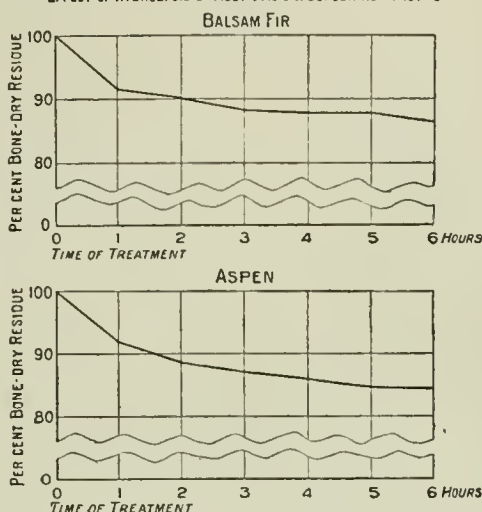


the bottom of the crucible with a fine platinum wire, C. It is often necessary, owing to uneven plates, to place a little absorbent cotton between the plate and the crucible bottom. The crucible is then dried at 105° C. to constant weight and weighed in a weighing bottle. By the use of this crucible, in which the fibrous material remains during the entire process, very satisfactory results are obtained as regards duplicate work, but the values are very high in comparison with the yields obtained in the commercial cooking processes and also in comparison with other methods.

As all the lignin is removed in the chlorination process this would indicate the presence of other non-cellulose substances in the residue. In fact König and Huhn have strongly criticised this method because the residue, by distillation with hydrochloric acid, yields large amounts of furfural, which can only be removed by hydrolysis. According to König's method and a method proposed by Tollens and Dmochowski,‡ these furfural-yielding impurities can be removed by hydrolysis with inorganic acids and with alkalis which, though effecting a nearly complete removal of these substances, attack the cellulose itself seriously,

making these methods unsuitable for exact analysis. All inorganic acids, even if very dilute, will destroy the cellulose more or less, but this is not true of certain organic acids. It was found that acetic acid in admixture with glycerin, at a high temperature, is able to hydrolyse the lower carbohydrates and a large proportion of the furfural-yielding substances without attacking the cellulose. The mixture was made by mixing glacial acetic acid with glycerin of sp. gr. 1.26 in the proportion of their molecular weights (60 : 92). This mixture can be heated at 135°–140° C. in an open flask, making it possible to carry out the hydrolysis at this high temperature without complicated apparatus. The glycerin causes the fibre substance to swell, whereby the action of the chlorine gas is greatly facilitated. The accompanying curves show that the loss in weight of the wood by treatment with this mixture is at first very rapid, but that after 3–4 hours a maximum

EFFECT OF HYDROLYSIS BY ACETIC ACID &amp; GLYCERINE AT 137° C



is reached. The mixture also dissolves the resin to a great extent, but a treatment with alcohol previous to the hydrolysis is considered to be favourable in order to secure a total removal of the resin and also possibly to effect a dehydration of hydrated cellulose.

The following is the method adopted for the determination of cellulose. Two samples of about one gram each of air-dry sawdust, passing an 80-mesh but not a 100-mesh sieve, are weighed exactly, transferred to small flasks, and heated on a water bath for ½ hour with alcohol, filtered into the crucibles, and washed with hot alcohol. The samples are then transferred from the crucible to 150 c.c. flasks and covered with about 75 c.c. of the mixture of glycerin and acetic acid and heated in an oil bath at 135° C. for four hours, using long glass tubes as air condensers. The material is then collected in the crucibles, washed well with hot water, and the crucibles after cooling placed in the chlorination apparatus in which they are connected with the source of chlorine in such a manner as to prevent escape of the gas into the laboratory. The gas is now passed through the crucibles for 20 minutes, after which the free chlorine is removed from the material by washing once with a cold, dilute solution of sulphurous acid in water. The crucibles are then placed in small beakers which are filled not quite to the top of the crucibles with a 3% solution of sodium sulphite and heated in a water bath for ¾ hour. The material in each crucible is washed with hot water, using a filtering flask, and after cooling, the chlorination process is repeated in the same

\* J. Ind. Eng. Chem., 1917, 9, 556, 564.

† J. Amer. Chem. Soc., 1907, 29, 1119.

‡ J. Landwirtschaft, 1910, Reprint.



way three times, the periods of chlorination being first 15, then 15, and finally 10 minutes. After the last treatment with sodium sulphite the fibres, after being thoroughly washed, are dried at 105° C. for four hours or to constant weight and weighed in closed weighing bottles. The residues are calculated in percentage of bone-dry wood, the moisture of the original sawdust having been determined in a separate sample by drying about two grams of the material for four hours at 105° C.

Table I gives a comparison of the cellulose content of a few Canadian wood species as determined by this modified method and by the original chlorination method (without acid hydrolysis).

TABLE I.—Cellulose content of some Canadian woods.

Species.	Diam., ins.	Annual rings.	Cellulose %.	
			Original method.	Modified method.
Aspen ( <i>Populus tremuloides</i> )	11½	38	{ 60.95 60.95	{ 57.35 57.15
Black spruce ( <i>Picea mariana</i> )	9½	74	{ 54.55 54.60	{ 50.78 50.50
Red spruce ( <i>Picea rubens</i> )	10½	60	{ 54.95 55.10	{ 52.95 52.95
White spruce ( <i>Picea canadensis</i> )	10½	83	{ 60.66 59.95	{ 56.55 56.40
Jack pine ( <i>Pinus banksiana</i> )	8½	61	{ 53.08 53.20	{ 49.35 49.13
Balsam fir ( <i>Abies balsamea</i> )	6½	54	{ 54.45 54.40	{ 51.50 51.70
Hemlock ( <i>Tsuga canadensis</i> )	12½	119	{ 48.68 48.72	

The figures show that the cellulose values obtained by the modified method are from 2 to 4% lower than those obtained by the original chlorination method. The hydrolysis has consequently removed certain substances which are not removed by the latter method. As the chief non-cellulose substances in the wood, other than resins, are known as lignin and furfural-yielding substances, the yield of methyl groups by distillation with hydriodic acid and the yield of furfural by distillation with hydrochloric acid and precipitation with phloroglucinol were determined in the residues obtained by treating balsam fir and aspen according to the two methods.

TABLE II.—Comparison of the residues obtained by the two methods.

Residue from—	Method.	Furfural.	Methylfurfural.	Total furfural.	Methyl (CH <sub>3</sub> ).
Balsam fir	Original	% 3.87	% 1.56	% 5.43	% 0.1399
	Modified	2.95	1.43	4.38	0.1302
Aspen	Original	10.49	1.39	11.88	0.135
	Modified	8.73	1.43	10.16	0.127

The residues give no colour reaction for lignin and are completely soluble in concentrated sulphuric acid giving a colourless solution. A low yield of methyl is still obtained. This methyl reaction is characteristic for the bleached commercial wood pulps, which are generally recognised as the purest form of wood pulps. The reaction is of the same strength and must originate from the presence of very small quantities of an undefined highly resistant substance. The furfural values show a very noticeable difference in favour of the modified method and it is interesting to note the similarity of the residue to the high grade commercial pulps with regard to furfural yield. It was not possible to secure a sample of commercial bleached sulphite pulp from balsam fir alone, but a bleached sulphite pulp from spruce

in mixture with a small percentage of balsam fir gave 3.10% furfural and may be used for comparison, as the difference of the two species with regard to furfural yield is not very appreciable. A soda pulp from aspen wood gave 9.69% furfural. These values are very close to those found for the cellulose residues obtained by the modified method. It would, of course, not be correct to judge the value of a method for cellulose determinations from the similarity of the residue to the commercial pulps, but this similarity of products obtained with different materials by different methods indicates that there is a certain amount of a very resistant substance in the wood which may be considered as the ultimate product of the commercial processes of purification. As an identical substance is obtained in the modified method of cellulose determination, it may be considered very useful in the valuation of the various woods for the commercial paper pulp processes.

Interesting differences in the distribution of the cellulose throughout the tree can be detected. On a previous occasion\* the decrease in cellulose content from the base towards the top of the tree was reported. The cellulose content was also determined at the same height from the ground at various distances from the pith of the tree towards the bark. The following figures were obtained from a disc of balsam fir with 64 annual rings and a diameter of 10½ inches:—

TABLE III.—Variation in cellulose content in balsam fir from pith to bark.

Cellulose %.		
A.	B.	C.
51.08 51.20	53.25 53.28	54.27 54.15

A.—Includes sample from pith to 15th annual ring. B.—From 20th to 35th annual ring. C.—Sap wood (outer 15 rings).

From a disc of aspen 9½ inches in diameter and 80 annual rings the cellulose content of sapwood and heartwood was compared.

TABLE IV.—Cellulose content of aspen.

Sap.	Heart.
% 61.24 61.45	% 59.88 60.19

In both species the sapwood has a higher cellulose content than the heartwood, and from the former table it may be taken that there is an appreciable variation in the cellulose content throughout the heartwood, at least in trees grown under normal conditions. But the growth of a

\* Pulp and Paper Mag., 1917, 15, 333.

tree is very much dependent upon the position and upon the growth conditions determined by its surroundings. In fact, it is seldom that a tree of absolute normal growth is found.

A disc was also taken from a balsam fir tree with a great proportion of "rotholz" (a condition due to strain or climate during the period of formation). One part of the disc showed very rapid growth and another part slow and normal growth. The disc had an average diameter of 9 inches and contained 54 annual rings. The differences in cellulose content were so considerable in the various parts of this disc that for comparison the lignin content was determined by treating the fibres with 72% sulphuric acid in the cold for a period of three hours. The yield of methyl by distillation with hydriodic acid and the solubility in alcohol were also determined.

TABLE V.—Analysis of disc of balsam fir of abnormal growth.

	" Rotholz."	Rapid growth.	Slow growth.
Cellulose, %	39.42	50.35	52.85
Soluble in alcohol, %	1.38	2.05	2.85
Lignin, %	33.6	24.44	24.85
Methyl (CH <sub>3</sub> ), %	2.74	2.43	2.02

Even after many years of normal growth, trees may gradually lose their value both for structural purposes and as paper-making material as the result of attack of wood-destroying fungi. Vast areas of forest are often covered with trees so seriously damaged by fungous growth that they have not yet been used as a raw material in any industry, and their utilisation is a great problem to the forester. The fungus *Fomes igniarius* (false tinder fungus) is distributed practically all over the world. Starting from a wound in the tree it extends its path of destruction over the entire heartwood, rendering the wood entirely useless for structural purposes.

According to von Schrenk and Spaulding\* one of the principal effects of the fungus *Fomes igniarius* is the solution of the lignin elements of the cell wall. But an analysis of a typical sample of aspen attacked by this fungus did not confirm this statement. Samples from the decayed heartwood and from the sound part were analysed with the result shown in Table VI.

TABLE VI.—Analysis of aspen attacked by the fungus *Fomes igniarius*.

Material.	Diameter.	Cellulose.	Furfural.	Methylfurfural.	Total furfural.	Lignin.	Soluble in hot water.
	ins.	%	%	%	%	%	%
Sound wood	10½	58.80	10.52	1.64	12.16	7.86	4.0
Decayed wood	—	42.29	9.98	2.10	12.08	13.16	14.85

This table distinctly shows, contrary to the claim of von Schrenk and Spaulding, that the cellulose has been broken down, and it further shows that the furfural-yielding substances are also attacked. The copper numbers (1.99 for the sound part and 8.05 for the decayed wood), and the solubility in boiling water also show that the cellulose has been broken down into reducing substances, presumably sugars.

These few examples of the application of the modified method of cellulose determination may be sufficient to demonstrate its usefulness in the analysis of wood, especially in relation to the production of chemical wood pulp.

The results recorded below were obtained in an

investigation of the action of the various reagents employed in the method of cellulose determination upon the most important constituents of the wood. This work was undertaken in order to secure as much information as possible regarding the details of the method of purification and to obtain, if possible, new data regarding the general composition of wood.

In discussing the determination of cellulose, reference was made to the other quantitatively most important substances in the wood, namely, lignin, the substances yielding furfural, and the other carbohydrates of comparatively low molecular weight. The characteristic reactions of these substances have been used in determining their presence in the original wood as well as in the residues isolated from this. These reactions were also used in studying quantitatively the nature of the substances which are removed by the various reagents employed in separating the cellulose residue from aspen and balsam fir. Samples of these species were disintegrated by means of a rasp and the material which passed the 80-mesh sieve but not the 100-mesh sieve was used for analysis. The material was extracted by boiling first with ether for 6 hours and afterwards with alcohol for 6 hours under a reflux condenser and with continuous stirring in each case. The extracted wood was then boiled in water for 6 hours under a reflux condenser with constant agitation and the material afterwards washed with hot water and then air-dried.

1. Small samples of the original wood were treated in exactly the same manner and dried at 105° C. and the loss in weight determined. The extracted and air-dried wood served as material for the tests recorded below. The tests were as follows:—

2. One part of the extracted wood was heated, in a beaker placed in a boiling water bath, for 4 hours, with a 3% solution of sodium sulphite and the fibres afterwards collected on a filter, washed with hot water, and air-dried.

3. Another part was heated with the mixture of acetic acid and glycerin in a flask under a reflux condenser at 135° C., using an oil bath. The residue was then washed on a filter with hot water and air-dried.

4. Another sample was treated according to the original method of chlorination and the cellulose residue, after thorough washing with hot water, was air-dried.

5. One sample was treated according to the modified method of cellulose determination, the residue being thoroughly washed with hot water and afterwards air-dried.

The loss in weight by each treatment was determined in separate samples and calculated upon the bone-dry weight of original wood. With all the residues the following analyses were made:—The yield of furfural and methylfurfural was determined by distillation with hydrochloric acid and precipitation with phloroglucinol according to the Tollens and Kroeber\* method, and extraction of methylfurfural with alcohol according to Ellet and Tollens.† Lignin was determined by treating the fibre for three hours with cold 72% sulphuric

\* Diseases of deciduous Forest Trees, U.S. Dept. Agr. Bull. 149, 1909, p. 34.

\* J. f. Landwirtschaft, 1901, 48, 357.

† J. f. Landwirtschaft, 1905, 53, 13.



acid, the residue being considered as lignin according to Klason.\* Methyl groups were determined by distillation with hydriodic acid as proposed by Benedict and Bamberger.†

typical reaction. It must, however, be remembered that the furfural reaction is by no means a characteristic reaction for oxycellulose since, for instance, the typical oxycellulose

TABLE VII.—*Constituents of the original fibres and the residues from the various treatments.*

	Treatment.	Furfural.	Methylfurfural.	Total furfural.	Total pentosan.	Lignin.	Methyl (CH <sub>3</sub> ).
Balsam fir	Original wood	% 4.19	% 3.12	% 7.31	% 12.54	% 31.1	% 2.01
"	1	4.12	3.28	7.40	12.70	30.22	2.25
"	2	4.03	3.33	7.36	12.62	29.42	2.42
"	3	3.89	3.03	6.92	11.90	25.56	2.37
"	4	3.87	1.56	5.43	9.33	—	0.1399
"	5	2.95	1.43	4.38	7.55	—	0.1392
Aspen	Original wood	10.60	1.39	11.99	20.47	6.33	2.70
"	1	11.10	1.26	12.36	21.10	5.45	2.62
"	2	11.91	1.16	13.07	22.10	5.44	2.48
"	3	11.38	1.05	12.43	21.22	3.99	2.41
"	4	10.49	1.39	11.88	20.32	—	0.135
"	5	8.73	1.43	10.16	17.37	—	0.127

TABLE VIII.—*Percentage of various constituents removed by the several treatments (percentages of total amount of each constituent in original fibre).*

		Percent. removed, calculated on amount in original fibre.				
	Treatment.	Total loss in weight.	Furfural.	Methylfurfural.	Total furfural or pentosan.	Lignin.
Balsam fir	1	% 5.32	% 6.92	% 0.45	% 4.17	% 8.01
"	2	8.73	12.16	2.56	8.07	13.66
"	3	8.95	15.51	11.53	13.81	25.18
"	4	44.37	48.64	72.20	58.74	100.00*
"	5	48.94	64.08	76.60	69.32	100.00*
Aspen	1	5.36	0.85	14.25	2.25	18.48
"	2	13.94	3.30	28.06	6.17	26.08
"	3	12.54	6.04	26.70	9.26	44.86
"	4	39.14	39.70	39.14	33.68	100.00*
"	5	41.45	51.80	39.80	50.40	100.00*

\* The 100% as given in this table refers to the lignin test with 72% sulphuric acid, which gives no residue or colour reaction when used on the cellulose residue.

From the figures contained in these tables it is again seen that there are striking differences in the chemical composition of balsam fir and aspen or, in general, between the coniferous woods and the broad-leaved woods. The differences may be briefly summarised as follows:—The broad-leaved trees give a considerably higher yield of furfural than the coniferous woods, which latter contain much more substance yielding methylfurfural. This substance, or at least part of it, is apparently very resistant, as there always remains a considerable proportion of it in the residue. The commercial pulps likewise contain a large proportion of this substance, and it is interesting to note the high resistance of the substance yielding methylfurfural in the analysis of decayed aspen given in the previous part of this paper.

The action of the various reagents in the different treatments used is to remove the complex yielding furfural and methylfurfural; the quantities thus removed depend upon the hydrolysing effect of the reagent employed, the effect of the acetic acid treatment being most pronounced.

In the tables given above the yield of furfural is also calculated as pentosan, the formation of furfural being a typical reaction for pentosans. Other substances have also been shown to be responsible for the furfural yield.

Cross and Bevan, who have classified wood as lignocellulose, claim that the cellulose residues they obtain from wood are not true celluloses of the same type as cotton cellulose but that they are, in properties, true oxycelluloses, giving furfural as a

obtained by treatment with calcium hypochlorite solution does not give the reaction. Moreover these cellulose residues do not show the most typical oxycellulose reaction, namely, the high reducing power (copper number). It must also be considered that the substance which gives the furfural yield can be partly removed by a strong acid treatment, according to König\*, although not without a simultaneous destruction of the cellulose. Furfural is also obtained from "wood gum," a substance which can be extracted from the broad-leaved woods in large quantities (about 15%), and from coniferous woods only in very small quantities by treatment with cold alkali and precipitating with alcohol. The wood gum in some cases also gives a methoxy reaction. This wood gum cannot be considered a uniform product, but no doubt is a mixture of hydrolysed pentosan and dissolved lignin.

The furfural yield of the cellulose residue has also been traced to so-called furfuroids,† substances which are similar to the pentosans in giving furfural but differ from the pentosans in being fermentable. They are considered to be more resistant than the pentosans and are therefore contained in the isolated cellulose residues of the wood. There is also the possibility that the product is a mixture of various constituents of the wood, that is, chiefly products of hydrolysis of pentosans and hexosans, which would explain the characteristic reactions.

The values for lignin obtained by treatment

\* Svensk Papp. Tid., 1916, p. 129.

† Monats., 260, 11.

\* "Bestimmung der Zellulose in Holzarten und Gespinnstfasern," Berlin, 1912.

† Cross and Bevan, "Cellulose," p. 187.

with sulphuric acid, and for methyl (which also is considered to be a quantitative lignin reaction) show a very peculiar difference in the two classes of woods and also in the two methods. Aspen with the highest yield of methyl only gives a very low value for lignin with sulphuric acid. There is, therefore, absolutely no connection between these values and the apparent disagreement in the results must be explained by the presence of two very different types of lignin. That there are different types of lignin has been indicated by Klason,\* who isolated the barium salt of lignin-sulphonic acid from waste sulphite liquor. While only about one-half of the lignin could be recovered in this way the high yield of methyl from this compound, in comparison with the lignin content of the wood, points to the conclusion that there are at least two lignins in spruce wood, of which one is very low in methoxy groups. Klason was also able to remove from wood, by alternate extraction with alcohol and water, a small proportion of lignin of a comparatively low molecular weight, whereas the barium salt of lignin-sulphonic acid recovered from the waste liquor was found according to him to have a molecular weight above 6000. Langet† isolated from various woods two lignic acids of different composition which varied in their solubility in alcohol. Apparently there are considerable differences in the types of the lignin substances of aspen and of balsam fir which are especially characterised by their solubility in concentrated sulphuric acid. Klason's method of determining lignin by treatment with 72% sulphuric acid is recommended for spruce and can, as far as our experience goes with the Canadian woods, be used with advantage with the coniferous woods in general, but seems to fail entirely with the broad-leaved trees.

The tables given above also show that it has not been possible with any of the reagents to extract only one uniform substance from the wood. A greater or a smaller proportion of a substance may always be dissolved out, but always in admixture with the other non-cellulose constituents. This has been stated on many occasions and has been explained by a difference in resistance of the various modifications of each substance. In this way designations such as hemi-celluloses, pseudo-celluloses, hemi-lignins, etc., have been proposed. König and Rumpf‡ have classified the non-cellulose substance of the cell membranes according to their resistances towards hydrolysis as "proto-pentosans," "proto-hexosans" and "proto-lignins" which are removable with water at 2–4 atmospheres pressure; the "hemi" type of the same substances which are removed by 1–3% acids at 2–4 atmospheres; and the "ortho" types of the substances which can be removed only by strong acid or by dilute acid at high pressure. These examples will be sufficient to show how difficult it is at the present time to form any opinion as to the constitution of wood, but they indicate that besides analytical work a critical study of the literature on wood chemistry is very much needed in order to simplify the terminology of the chemistry of wood.

Wislicenus§ has studied the problem from the point of view of colloid chemistry. He was able to precipitate from the cambium sap of the plant 39% of its dry weight by adsorption on colloidal clay and put forward the theory that the lignin represents colloids separated out from the cambium sap by adsorption, one part of which is reversibly, and another irreversibly deposited on the cellulose. Klason|| favours this opinion

and from his own investigations draws the conclusion that 6% of the total lignin is reversibly combined with the carbohydrates of the wood, namely, the 2% of lignin which he was able to dissolve out merely by boiling with water and with alcohol alternately.

Wislicenus' theory according to which the lignin and the carbohydrates of the wood are combined on the basis of the force of the surface tension, and therefore in varying proportions, can no doubt be applied to the other substances of the wood, and will probably form the basis for future investigations in the chemistry of wood. The great similarity of the woods with regard to chemical composition and the fact that it is not possible to dissolve out any of the chief constituents without attacking the other substances, makes it probable that each of the substances cellulose, lignin, pentosans, and hexosans, are present in the wood, partly in a free form and partly more or less closely attached to the other substances, which explains the uniformity in the composition of each species. This equilibrium, however, must change as soon as one or more substances are changed in quantity or are totally absent. If we consider the high percentage of pentosans and the difference in the nature of the lower polysaccharides of the broad-leaved woods in comparison with the coniferous woods, it may be possible to explain the difference in behaviour of the lignin complex of the two classes of woods towards chemical reagents (hydrolysis) and also the apparently close attachment of the pentosans to the cellulose in the case of aspen.

This point of view does not, of course, exclude the probability that the lignin groups as well as the groups of pentosans and hexosans may consist of compounds of the same composition, but with varying molecular weights.

## Edinburgh Section.

*Meeting held at Edinburgh on Tuesday, December 11th, 1917.*

MR. D. B. DOTT IN THE CHAIR.

## THE PRESERVATION OF DECAYING STONE.

BY A. P. LAURIE, M.A., D.SC., AND CLERK RANKEN, D.SC.

(Abstract.)

This enquiry has been undertaken at the request of His Majesty's Office of Works with a view to dealing with the pressing problem of the preservation of decaying stonework in ancient buildings, and the following observations are the results of the preliminary experiments. It is therefore not merely a question of treating a stone which is at present free from decay, with some solution which will increase its durability. In the case of these ancient buildings the stone is already decayed to a certain extent and is in a crumbling condition. If this crumbling surface is cleaned off, the whole artistic value of the work is destroyed. It is therefore necessary to find something which will not only preserve the stone from further decay, but will reconstruct the existing crumbling stone.

Various substances have been put on the market with a view to stone preservation. These substances can be classified into certain groups. Some of them consist of materials of the nature of paraffin

\* "Beiträge zur Kenntniss der chem. Zusammensetzung des Fichtenholzes," Berlin, p. 12.

† Z. physiol. Chem., 1910, 14, 15, 217.

‡ Unters. Z. Nahr. Genussm., 1914, 28, 177.

§ Z. Chem. Ind. Kolloide, 1910, 6, 1–2.

|| Svensk Pap. Tid., 1916, 129.



wax dissolved in a volatile solvent, the idea being to fill the pores of the stone with a neutral substance which will prevent further admission of moisture and will tend to bind the particles together. Others consist of solutions of materials like linseed oil, which are gradually converted by the action of oxygen into jellies, which are meant to fill the particles of the stone.

There are others which are intended to act upon the calcium carbonate or sulphate, producing insoluble compounds which are intended to bind the stone together. And there are others in which two solutions are used, with a view to form a precipitate within the pores of the stone itself.

In the case of the simplest form of preservative—a solution of a solid which is to be left in the stone on evaporation, like paraffin wax—the difficulty is to ensure that the solid will be left within the interior of the stone, the probability being that in most cases—as a result of capillary action—it will be found in the outer layer. This also probably applies to solutions which are intended to decompose spontaneously, as they will be drawn out by evaporation before decomposition takes place.

Better results might be hoped for in the case of solutions which are intended to act on calcite or calcium sulphate already within the stone, and so produce an insoluble precipitate, though, on the other hand, they involve serious questions as to how far it is wise to attack an existing cementing material with a view of forming a new one.

In practice, it has been noticed that in many cases preservatives, while producing temporary effect, ultimately result in scaling.

The result of our experiments is to show that, even if a stone is sprayed several times with solutions of paraffin wax, it will not be rendered non-porous to moisture. It would be necessary to carry on the loading with paraffin wax to a point which would be quite impossible in practice, in order to obtain this result. But it has also been found that the paraffin wax has a marked effect on the process of evaporation which takes place afterwards. If the stone has been sprayed with paraffin wax before treatment with a potassium ferrocyanide solution, this spraying will only partially prevent the absorption of the solution, but tends to prevent on evaporation the crystallisation of the potassium ferrocyanide on the outside of the stone. The tendency, however, for such crystallisation is to take place principally just underneath the paraffin wax layer. This layer is very thin, the solution of paraffin wax being absorbed readily by the stone, but on evaporation depositing the paraffin wax at, or near, the surface. It is evident, therefore, that a stone so treated with paraffin wax will still continue to absorb moisture, while the resulting products of decomposition will tend to collect just under the surface layer, and by crystallising out in that position, will ultimately produce flaking of the stone.

It is probable, therefore, that all stone preservatives of a greasy nature will be peculiarly apt to produce flaking ultimately on the stone. This raises the interesting question how far the deposit of greasy smoke p. r. c. s. is an important agent in the decay of stone.

It is obvious then that the first problem is to ensure that the particular material used permanently penetrates to a considerable depth within the stone itself, the first penetration being of no value, owing to the subsequent evaporation.

#### *Phenomena involved in the use of two precipitating solutions.*

If a piece of stone about 1 in. in section was saturated with one solution and then poulticed on one side with another solution that would form a precipitate, we found in some cases that there

was not only a copious precipitate on the side of the stone that was poulticed, but a so a copious precipitate on the back of the stone, penetrating a little way into the interior; while in the centre of the stone, very little, if any, precipitation was visible.

#### *The cementing properties of precipitates.*

If the pores of the stone have been filled to a sufficient depth with an insoluble substance, are we justified in assuming that this substance will form a successful binding material, holding the particles of the stone together?

A large number of organic substances, such as linseed oil, paraffin wax, casein, resins, and gums, have the property of cementing particles together, and we also know from the study of stones themselves, that many substances also will act as a cement, even a crystalline body such as calcite forming a firm and strong cement between particles of quartz.

We are also familiar with the cementing properties of the jelloids produced during the setting of Portland cement, but in the case of stones, these inorganic cements have been produced very slowly and under special conditions, while in the case of Portland cement, the partial change into a gelatinous mass with the minimum quantity of water present is very different from the conditions of precipitation when a stone is treated in the usual way with two precipitating substances.

We are therefore not justified in assuming that even such substances as silica or alumina are going to act as a cementing material of any value when precipitated rapidly within the pores of the stone.

We decided in the first place to study what happens when a stone is treated with a simple solution whether of a salt in water or of a material like paraffin wax dissolved in a volatile solvent.

A large number of slabs, about 4 in. each way and about 1 in. thick, were cut from Cullaloe stone, a fairly porous stone, consisting almost entirely of silica, the analysis being as follows:—Silicious matter 99.17%,  $Al_2O_3$  0.11%,  $Fe_2O_3$  0.07%,  $MgCO_3$  0.12%, combined water, etc. 0.46%, moisture 0.03%. The binding material between the particles of quartz is apparently a silicious deposit. This stone was therefore selected as not being likely to introduce any chemical complications in the experiments.

A series of wooden frames were made, with a flange, similar to picture frames, into which the slabs of stone could be fitted, and pressed up against a rubber washer, by means of clamps. In this way one surface of the stone was exposed, and could be treated by spraying with various materials, while at the same time, the creeping of the solution round the edges of the stone was prevented by the rubber washer. In addition to spraying, we ultimately adopted, as a much more effective method, covering the surface with paper pulp soaked with the solution with which we wished to treat the stone, and covering the paper pulp again with lead foil, the whole being pressed gently against the stone, so as to remain in contact. Such solutions laid on in this way would remain moist for some time, and if necessary could be renewed from time to time.

We also had a small machine constructed, consisting of a cylinder and a screw piston, for compressing and forming small cylindrical bricks, in order to test the behaviour of various solutions when mixed with pure white sand, ground Portland stone, and ground Ketton stone, as representatives of pure silicious sandstone, calcareous sandstone, and purely calcareous stone. By mixing these powders with various solutions, precipitates, and preservatives, and pressing them into bricks, it was possible to test, under very advantageous

circumstances to the solutions used, how these solutions behaved, and to what extent they could be regarded as successful binding materials.

The first experiments were made to test the question as to how far, after treatment with the solution, capillary forces came into play, and removed the solution from the stone.

Some of the slabs of stone already described, after having been mounted in the wooden frames, were treated with the following salts in solution in water:—Potassium ferrocyanide, sodium phosphate, sodium sulphate, ammonium phosphate, copper sulphate, copper nitrate, lead nitrate, copper chloride, potassium chromate, potassium arsenite, potassium bichromate, cobalt chloride, ferric chloride, barium acetate, chromium chloride, soap. The reason for the selection of these salts was that in most instances they lend themselves to detection by means of strong colouring actions.

With the exception of ferric and chromium chloride, soap, and potassium arsenite, in every instance it was found that as the stone dried the salt in solution was practically removed almost entirely to the surface layer, although before the stone was allowed to dry it had been saturated with the solution, which had begun to crystallise on the back of the stone.

The tendency, however, to rush rapidly out from the stone is not equally strong in the case of all these salts, potassium ferrocyanide being the most marked example of the kind.

Three salts, ferric and chromic chloride and potassium arsenite, and also soap, were found to be exceptions to the general rule, these salts remaining to a very large extent within the interior of the stone.

In order to test organic substances, we selected solutions of shellac in alcohol, and of paraffin wax in xylol, and, as has already been stated, we not only found that several sprayings of paraffin wax were not sufficient to make a stone really watertight, but we also found that both these substances came to the surface of the stone during the process of drying. We also found that if the stone was first lightly sprayed with a solution of paraffin wax in xylol, and then treated with a salt solution, there was very much less tendency for the salt to crystallise on the outside of the stone, and that the distribution in the interior of the stone was better, but that there was a distinct tendency for an excess of the salt to be deposited just underneath the main paraffin wax layer, which lay from 1/16th to 1/8th of an inch below the surface of the stone. This result has already been referred to, as showing the danger of using greasy materials as stone preservatives, in that it tends to cause an accumulation of crystalline matter dissolved from the stone, just under the surface layer.

In order to check these experiments, we made a series of experiments with the bricks made of sand, with a view to finding out how the solution would be distributed under those conditions. In each case, of course, the solution was mixed evenly throughout the sand. The little brick was then allowed to dry, and examined; it was then, in many cases, immersed in water, to see what would happen under those conditions. Solutions of shellac, gelatin, paraffin wax, and albumin, were all tried, and in each case the result was formation on the outside of the brick—which was about 1 in. in diameter and about 2 in. long—of a hard layer about 1/16th of an inch thick, while if a hole was drilled inside the brick, the sand could be poured freely from the interior.

Some experiments, however, made with gelatin require to be discussed in more detail, as they illustrate a very interesting point in this connection. If a stone is treated with a liquid which solidifies without evaporation, the liquid should remain throughout the stone evenly distributed. That this is so was shown by experiments with bricks

made with solutions of gelatin in water. In the case of a 1% solution the hardening was entirely superficial, the sand pouring freely from the interior; this was still true, but to a lesser extent, for the 3% solution; but in the case of the 5% solution the hardening of the sand brick took place all through.

While, in the case of all these inorganic solutions which we have tested, with the exception of ferric and chromic chloride and potassium arsenite, the result has been to bring the solid to the surface, there are no doubt exceptions to this rule, as it is possible to select substances which, when dissolved in water, lower the surface tension instead of increasing it, such as a solution of soap; and, moreover, as the surface tension of a mixture is the sum of the surface tension of the separate constituents, it is quite possible that the tendency of one substance in solution to cause crystallisation on the outside of the stone might be counteracted by the condition of another substance. Investigation in this direction was carried out and the effects of sugar, glycerin, starch, and soap were tried on aqueous solutions of various salts.

(1) Glycerin on every occasion was found to favour retention. Even with saturated potassium ferrocyanide only a very small amount crystallised on the surface.

(2) Soap produced practically no effect.

(3) Starch caused retention to a small extent but, on the other hand, did not permit of the stone taking up the solution readily.

(4) Glucose caused retention better than starch, but not nearly so great as glycerin.

#### *Method of experiment.*

(A) Stones soaked with concentrated solutions of potassium ferrocyanide, copper chloride, or barium chloride and allowed to dry gave large crops of crystals on the surface after 2 or 3 days.

(B) Stones soaked with the same concentrated solutions to which a small amount of glycerin, etc., had been added and allowed to dry, were compared with (A). Also after standing drying for 3 weeks, the stones (B) were broken and the interior tested. The tests showed whether the stones did or did not retain; e.g., a Cullaloe sandstone slab treated with concentrated potassium ferrocyanide and a drop or two of glycerin was allowed to dry for 3 weeks, broken and the surface and the interior tested with drops of ferric chloride. The Prussian blue formed in the interior was as dense as that on the surface. Again, after drying for 9 months, the ferric chloride test still showed that the potassium ferrocyanide remained in the interior. It was also noticed that the stone had not got absolutely dry after 9 months, but still retained traces of moisture.

The effects of glucose and glycerin, nitrobenzene, aniline, methyl acetate, and ethyl benzoate on retention of alcoholic solutions were also tried. In all cases the addition of glycerin, etc., seemed to have no effect in causing the substances to remain inside the stone.

The result of these experiments is to show that while no reagent has been found to prevent the deposition in the outer layer of substances in organic solvents, the addition of a little glycerin to water solutions is sufficient to prevent this. We therefore recommend that in any practical tests of water-dissolved stone preservatives, the precaution of adding a little glycerin be taken.

In order to test how far various substances acted as cements, we made up a series of bricks mixed with various materials, directing special attention to the fluosilicates, as much attention has been directed to them lately as stone preservatives, more especially in Germany.

In the first place, we selected certain gelatinous precipitates which are generally supposed to act as cements, and having made up bricks mixed



with sand and gelatinous precipitates and given them three weeks to dry, we examined their condition and then immersed them in water and examined their condition again. We found that precipitates of alumina and silica completely failed to act as cementing materials in the case of sand. If mixed with a little lime, the bricks were a little harder, but at once got soft in water. Aluminium fluosilicate showed a distinct tendency to come to the surface of the brick, and, whether alone or mixed with lime, the resulting brick was at once softened in water. Zinc hydroxide and calcium hydroxide, alumina and barium hydroxide, stannic acid, and stannic acid and calcium hydroxide, etc., failed to produce bricks which would stand the test of water; while casein, and zinc hydroxide mixed with calcium hydroxide stood the water test successfully.

The results obtained will be found in the following table.

Sand.	Binding material.	Hardness after three weeks.	Hardness in water.
	Alumina .....	Crumbly.	Very soft.
	Silica hydrated.	Crumbly.	Very soft.
	Alumina + calcium hydroxide .....	} Not quite so crumbly.	Soft.
	Silica hydrated + calcium hydroxide....		Soft.
	Aluminium fluosilicate + calcium hydroxide	Harder.	Soft.
	Aluminium fluosilicate	Hard outside.	Very soft.
	Aluminium sulphate then barium hydroxide .....	Hard outside.	Very soft.
	Zinc hydroxide .....	Fairly hard.	Soft.
	Zinc hydroxide + calcium hydroxide....	Harder.	Hardish.
	Stannic acid .....	Powder.	—
	Stannic acid + calcium hydroxide .....	Crumbly.	Very soft.
	Casein .....	Hard.	Hardish.

We next made up a similar set of bricks with Ketton stone. In this case, zinc fluosilicate, magnesium fluosilicate, and what is known as "Doppel" fluosilicate, as supplied by Hans Haenschel, Berlin, failed to form permanent bricks. In each case the bricks were crumbly after two weeks, though those prepared with the Doppel fluosilicate were not quite so crumbly as the others.

These results are of considerable importance, as it is claimed for these fluosilicates that in the case of calcareous sandstones, the resulting complex precipitate of fluosilicate of lime and zinc and magnesium hydroxides forms a reliable cementing material.

Any supposed preservative which depends for its success as a binding material on attacking the calcite which the stone contains should be regarded with profound suspicion. The results do not justify the use of a solution which is to depend upon its success in attacking the calcite existing within the stone, destroying the natural cement of the stone, in order to create another. If a solution is to be selected with this aim in view, it is difficult to imagine anything better than the fluosilicates, and it is for that reason that we have tested them so carefully, and have come to the conclusion that they are not reliable.

Such a process must be distinguished from that of Sir Arthur Church, in which case he is attacking the sulphate of lime in the stone, which is already a decomposition product, and therefore is not injuring the structure of the stone by the treatment to which it has been subjected.

The second conclusion which we have arrived at from these experiments is that a very large number of colloid precipitates, such as silica and alumina, cannot be regarded as cementing materials.

The direction, therefore, that the enquiry should take should be, if possible, to find an inorganic substance which is insoluble in rain water and the acids usually found in rain water, which, without requiring to attack in any way the natural combining material of the stone, will form a successful cement for particles of quartz.

#### *The conditions of precipitation within the stone.*

It is evident, from what has been already stated, that we have to look for our successful preservative in one of two directions, *i.e.*, we must either find a single solution or liquid which will solidify within the stone and form a successful combining material without showing any tendency to come to the surface, or we must obtain the result by a process of double precipitation, by the action of one solution upon another. The difficulties of obtaining such a solution are sufficiently obvious, but it must not be ruled out of the ultimate possibilities. At the same time, it is very likely that the right method is to use a double solution, and therefore experiments on the precipitation within the stone itself require to be undertaken.

In order to test this question, we decided again to select precipitates, in the first instance, which, on account of their colour, would be fairly visible on the stone, without any reference to their suitability as stone preservatives, in order to get some guiding principles in the matter of such a precipitation.

The main difficulty of successful precipitation in the interior lies in the fact that the first precipitation will take place across the mouths of the capillaries, forming a diaphragm which will be more or less impervious to the passage of the salt molecules in either direction. At the same time we might expect to find the molecules of one of the salts might penetrate very much more easily than the molecules of the other, and therefore that the order in which the treatment of the stone took place would prove of considerable importance.

In most of these experiments we did not allow the stone to dry completely before applying the second solution; in some cases applying the the second solution directly, before evaporation could have taken place; in other cases allowing partial drying before its application. We also in certain cases sprayed the stone lightly with paraffin wax on both surfaces, so as to tend to keep the first solution within the stone and give the precipitation a better opportunity.

The experiments on spraying the unwaxed stone showed that in every case the precipitation took place on the surface layer of the stone, and there was no proper penetration. The only successful experiments have been those made by means of the poulticing method, which has already been described. These results show that it depends very largely upon which salt solution is applied first, a fair precipitate being obtained inside in certain cases, while, when the treatment with the salts is reversed, practically no precipitation takes place at all. One of the necessary conditions, therefore, of success is to decide in which order the solutions are to be applied. It was decided to test the use of two different solvents for the salts. The idea of these experiments was to select two salts, which will result when mixed in a precipitate, one soluble in water and alcohol, the other insoluble in alcohol. If the stone is first treated with the water solution and allowed to dry leaving the salt precipitated throughout it, and if it is then treated with the second salt dissolved in alcohol, the second salt will be able to penetrate freely into the pores, owing to the fact that the precipitation will be delayed owing to the insolubility of the first salt in alcohol, and therefore we may hope to get the precipitation taking place within the stone.

*Further experiments on precipitation of substances inside stone.*

Further experiments in this direction lead to the conclusion that the decisive factor in precipitation in the stone is the order in which the stone is treated with the solutions causing precipitation. Practically perfect precipitation was produced in the stone when potassium ferrocyanide was the second solution used, whereas when it was the first solution there was practically no precipitation in the stone.

The use of different solvents has certain advantages in so far that no waiting is required between treatment with the two different solutions. Waiting, however, is no real disadvantage owing to glycerin, etc. being able to keep the first substance in the interior of the stone.

the precipitation of one solution by another—must be a material which is not readily attacked by air, water, or the acids usually present in rain water.

(3) None of the solutions used should act upon the natural cementing material of the stone, or should depend upon that action in order to produce a binding material.

(4) The resulting solid—whether produced from one or from two solutions—must be one which has been proved by severe tests to act as a cement for particles of silica.

(5) If two solutions are to be used it is very important to study carefully the right order of application in order to ensure proper penetration, or to use two different solvents in the way already described.

Stone.	Treated first with:	Drying period.	Treated with:	Result.
Waxed	Lead nitrate (aqueous saturated) until crystals at back.	1 day	Potassium chromate (saturated) for 6 days.	Practically no precipitate inside. All round outside. 1.1th inch.
Unwaxed	Borax.	3 days	Copper sulphate (saturated).	No precipitate inside. All round outside.
Unwaxed	Lead nitrate (aqueous saturated) until crystals at back.	1 day	Potassium chromate (saturated) for 6 days.	No precipitate inside. All outside. 1/16th inch.
Unwaxed	Potassium chromate (saturated) until crystals at back.	1 day	Lead nitrate (saturated) for 6 days.	Fair precipitate inside stone (irregular).
Waxed	Potassium chromate (saturated) until crystals at back.	1 day	Lead nitrate (saturated) for 6 days.	Fair precipitate inside stone (irregular).
Unwaxed	Copper sulphate (saturated).	1 day	Potassium ferrocyanide for 6 days.	Small amount of precipitate in layers in stone.
Waxed	Copper sulphate (saturated).	1 day	Potassium ferrocyanide for 6 days.	Precipitate going through stone in layers.
Unwaxed	Potassium chromate.	1 day	Lead nitrate.	No precipitate in stone.
Unwaxed	Copper nitrate.	3 hours and then waxed	Potassium ferrocyanide.	Little or no precipitate in stone. It penetrated but only gave streaks.
Unwaxed	Copper sulphate (aqueous).	None	Alcoholic caustic potash for 2 days.	Black copper oxide well through stone.
Unwaxed	Caustic potash (alcoholic).	2 hours	Aqueous copper nitrate.	No precipitate in stone.
Unwaxed	Copper chloride (alcoholic).	None	Aqueous sodium sulphide.	Poor precipitate of copper sulphide in stone.
Unwaxed	Sodium sulphide (aqueous).	None	Alcoholic copper chloride.	Very good precipitate in stone.
Unwaxed	Ferric chloride (alcoholic).	None	Aqueous potassium ferrocyanide.	Very fine precipitate in stone.
Unwaxed	Potassium ferrocyanide (aqueous)	None	Alcoholic ferric chloride.	Very poor precipitate in stone.

The result, then, of these experiments on precipitation is to show that, in the first place, methods of spraying with two solutions in water seem to be very unlikely to yield satisfactory results; and that some method of poulticing is apparently essential for success, so as to ensure deep penetration of the solutions. In the second place, if prevention of the salt crystallising on the outside can be obtained, it is possible to get a fairly uniform precipitation within the stone. And, in the third place, even where salts are used which tend to crystallise on the outside, the use of the two solvents results in a very uniform precipitation if the stone is not allowed to dry between the two treatments.

While we do not pretend for a moment that these results can be regarded as more than preliminary, we think that they have been sufficient, in the first place, to show that the problem has not been successfully solved by the existing stone preservatives, and also to show the complex nature of the problem, and the directions in which a solution of it is to be looked for.

These are as follows:—

(1) The solution with which the stone is treated—or if two solutions are to be used, the first solution with which it is treated—must be of such a kind that the resulting solid is uniformly distributed throughout the stone, and does not tend to come to the surface. It must, therefore, be a solution in which the surface tension tends to diminish instead of increase on concentration.

(2) The resulting cement—whether due to the evaporation or solidification of one solution, or to

**Yorkshire Section.****THE LOSS OF INDICAN ON DRYING INDIGO LEAF.**

BY E. R. WATSON.

(This Journal, 1918, 81 T.)

**DISCUSSION.**

Professor A. G. PERKIN said that it was, of course, well known that the indican content of the freshly-gathered indigo plant in its moist condition rapidly deteriorated, but there was no previous record so far as he was aware of the behaviour in this respect of the leaf when dried under ideal conditions. Indeed, it was, and might still be, the case in some parts of India to air-dry the leaf before employing it for the manufacture of indigo, and should only a small amount of colouring principle thus be lost, this procedure would have much to recommend it. As this matter was interesting to him at the time, he had approached Professor Watson, then living in India, and he had very cordially undertaken to carry out the work, an account of which was contained in the paper. It had long been suspected that indigo brown, a well-known impurity of commercial indigo, was derived from indoxyl itself, and thus represented a loss of colouring matter. There was evidence from the work of Rawson and others that this was the case, but unfortunately the matter was difficult to prove with certainty. In



conjunction with Bloxam he had shown that when pure indican was digested with moderately strong hydrochloric acid at the boil, a brown compound termed indoxyl brown was produced in which approximately all the nitrogen originally contained by the glucoside was present. A similar product was also obtained by submitting the leaf extract to the same operation, and in this the whole of the indican should be represented by indoxyl brown. He had recently found that "seeth" water, that is the liquid remaining after the settlement of the indigo, yielded some quantity of a product similar to indigo brown, and he felt that in case during the preparation of a small batch of indigo on the large scale he could obtain samples of the leaf, of the finished indigo, and of the "seeth" water for analysis, a solution of this problem might possibly be arrived at. The results of Professor Watson's experiments now made it evident that an investigation of this character could only be satisfactorily accomplished on the spot.

During his work with Bloxam at the Leeds University some years ago, numerous samples of air-dried indigo leaf obtained for them by the Indian Government had been examined, and it was interesting now to know that the poor indigo-yielding content of many of these, which was approximately 0.6%, did not by any means represent the capacity of the growing material. On the other hand one specimen of *I. sumatrana* leaf had given the surprising amount of 3.13% of indigotin, and it would thus appear that in its fresh condition a considerably larger quantity had been present.

It was interesting to note that the air-dried leaf even enclosed in closely stoppered vessels very gradually suffered alteration, and he had in his possession a sample of this originally rich leaf which when last examined gave but 0.5% of indigotin. The alteration in this case he had ascertained was not due in the main to the formation of indigotin in the leaf substance, but, curiously enough, of indirubin, which was evidence that in these circumstances a portion of the indoxyl was converted into isatin, and then reacted with indoxyl subsequently liberated. A certain amount of brown matter was also formed during this deterioration of the contents of the leaf, but no attempt had yet been made to ascertain its character.

## Discussions on Papers.

### LIGHTING A CHEMICAL BALANCE.

Mr. H. DROOP RICHMOND writes as follows:—  
On page 95 T of the issue of the Journal of March

30th, Mr. B. D. Porritt describes a "Convenient Arrangement for Lighting a Chemical Balance." This arrangement is almost identical with that which has been in use in the Laboratory of Messrs. Boots Pure Drug Co., Ltd., for some years past, which was devised by my predecessor, Mr. T. F. Harvey.

### THE ANALYSIS OF ALUMINIUM ALLOYS.

In a contribution to the discussion on the above paper, by B. Collitt and W. Regan (this J. 1918, 91 T), Mr. A. W. WILLIS writes that the estimation of manganese in aluminium alloys may be performed even more simply and expeditiously than described by the authors, as 1 gm. of practically any of the alloys will dissolve readily in 25–30 c.c. of nitric acid of sp. gr. 1.20, if a single drop of hydrofluoric acid be added. This immediately causes much effervescence and rapid solution of the alloy. The solution is then oxidised with bismuthate, cleared and cooled, oxidised cold, and filtered through asbestos as usual. The hydrofluoric acid has no detrimental effect on the results, and enables one to work with a much smaller bulk of solution which is better for the oxidation by bismuthate. For nickel estimation, the glyoxime method gives the most satisfactory results, the process being somewhat simpler than that given in the paper, viz., the alloy is dissolved in dilute hydrochloric acid with the addition of a drop or two of nitric acid to get the copper residue into solution, avoiding excess. Then the solution is diluted and about 10 grms. of tartaric acid added, and when dissolved a suitable quantity of dimethylglyoxime solution, and the liquid made ammoniacal and allowed to stand. It is then filtered through counterpoised papers and the precipitate dried and weighed as usual. The aluminium, copper, etc., are kept in solution by the tartaric acid and ammonia, and the nickel precipitate is quite clean. The manganese and nickel processes as above have been in continuous use in a large works laboratory for some years, and are quite satisfactory for ordinary works purposes. Mr. Grisley's suggested method of titrating the copper with potassium cyanide was tried some three or four years ago and the method found to be quite satisfactory with care, but for rapid works check purposes (mostly performed by junior workers) it was found that the iodide method gave a much better end-point, as the cyanide discharges the copper blue colour somewhat slowly and hence requires slower and more careful titration.

## Birmingham Section.

Meeting held at Birmingham University on Thursday,  
March 21st, 1918.

MR. L. P. WILSON IN THE CHAIR.

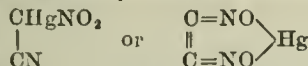
## FULMINATE OF MERCURY AND ITS DETERMINATION.

BY G. S. HEAVEN, B.Sc.(LOND.), F.I.C.

Fulminate of mercury was discovered by Howard about 1800, and was described by him in a paper read before the Royal Society. He prepared it by the action of mercuric nitrate on alcohol and also by treating mercury with alcohol in the presence of nitric acid.

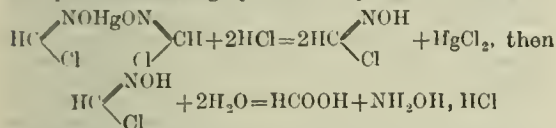
Liebig, in 1823, analysed the silver salt and found it to have the same composition as silver cyanate and cyanurate,  $\text{HgC}_2\text{N}_2\text{O}_2$ .

The constitution of the mercury salt could therefore be represented by either



When fulminate is treated with concentrated hydrochloric acid, it decomposes in accordance with the equation:—

$\text{Hg}(\text{CNO})_2 + 2\text{HCl} + 4\text{H}_2\text{O} = \text{HgCl}_2 + 2\text{NH}_2\text{OH} + 2\text{HCOOH}$   
and Schott, in 1891, explained the formation of hydroxylamine and formic acid by attributing to fulminic acid the carbyloxime formula, the addition compound breaking up into formyl chloridoxime:—



Nef (Annales Chimie et Pharm., 1894, 268), described the synthesis of mercury fulminate, by the action of sodium nitromethane on mercuric chloride. This appears to verify the cyclic constitution in view of the formation of the formyl chloridoxime, as above, instead of hydrocyanic acid, when fulminate is treated with hydrochloric acid.

Fulminate is manufactured by a method closely resembling that used in its preparation by Howard. The following is the outline of a published method. A cooled solution of mercuric nitrate, prepared by dissolving 500 grms. of purified mercury in an excess of concentrated nitric acid (4.50 kilos.), is poured into a large flask containing 5 litres of 94% alcohol at a temperature of about 25°C. In about 20 minutes the reaction is complete and the crystals begin to separate. The mixture is poured into a small quantity of water and the fulminate which separates is washed by decantation. It is then sifted through a fine net filter which serves to remove large crystals and finally washed on a finer mesh filter till free from acid.

The fulminate is stored in calico bags under water, and for transportation in bulk the bags containing 20 lb. each are packed in a barrel which is filled with water; this is headed and placed in a large cask likewise filled with water.

The powerful oxidising action of the nitric acid gives rise to various oxidation products such as aldehydes, nitrites, and nitrates. The aldehydes may subsequently reduce the fulminate and give rise to free mercury and to soluble mercurous salts.

For use the fulminate is washed with distilled water, through a sieve of 40-mesh, into a calico bag, the wash water being tested with litmus to ascertain that washing has been complete. Excess of water is then removed by pressure applied either by hand or mechanically, and the fulminate is then dried on fulminate drying cloths in one pound lots or less.

Drying is performed on tables heated either with water or with steam, or on cloths fitted to wooden frames in a current of warm air. The temperature of drying is usually about 83° C., 65° C. being the maximum temperature allowed. The drying fulminate smells of alcohol.

When the fulminate is dry it is cooled to the temperature of the room and the lumps pressed to powder by means of a soft pad, or pressed through the muslin cloths by the hands. The fulminate is then stored in well varnished papier-mâché pots.

The copper tables are attacked by the wet mercury compounds, and the bags on which the fulminate stands, and the copper sieves through which the fulminate passes, become green. Copal varnish has been found to be the best preservative for the tables, and several thicknesses of flannel or of cloth help to keep the mercury fulminate free from copper salts.

**Crystalline form.** Owing to the smallness of the crystals, which are on the whole microscopic, the fulminate appears to the naked eye as a sparkling powder, more or less fine according to the conditions of manufacture. Personal observations of numerous commercial samples show that the largest of the crystals, or of a repeated series of apparently twinned crystals existing as an individual, does not exceed 1 mm. in length. An average sample, of a coarse make, gave the following results on shaking the dry powder through a series of sieves:—100% passed through an 80-mesh sieve, 40% remained on a 120-mesh sieve, 30% remained on a 200-mesh sieve, and 20% remained on a fine calico. The 10% which passed through the calico could be still further subdivided in size by settling in water, an impalpable powder being finally obtained which under a magnification of 440 diameters showed no crystalline form. Analysis showed it to contain 85% fulminate. This result emphasised the need of thoroughly washing the fulminate.

Another sample gave the following results:—20% remained on a 120-mesh sieve, 30% remained on a 200-mesh sieve, and 30% remained on calico.

It will be seen that even from samples of the same make, there is considerable variation of size which from the point of view of the detonator maker is undesirable.

The colour of commercial samples varies from pale cream to dark brown. Some crystals are colourless and like glass, others have the colouring matter evenly distributed, whilst in others—and this occurs frequently—there is a concentration of colour ranging from a deep pigmented band along an edge to deep spots of colour occurring adventitiously in or on the crystal.

As some manufacturers habitually produce coarse dark powders, and others finer and paler, it might be inferred that the dark coloration of a coarse powder is due to special ingredients, but that there is a definite connection between colour and size of grain in fulminate prepared as described above, is shown by the similarity of coarse powders produced by different makers, as also by the large crystals separated from the bulk of a given sample being darker than the fine. It has been stated that the colour is due to resinous substances, so that the difference may be due either to well-known optical effects or else to the longer contact of large crystals with the mother liquor.

To remove the colour, copper and hydrochloric acid are sometimes added to the reaction

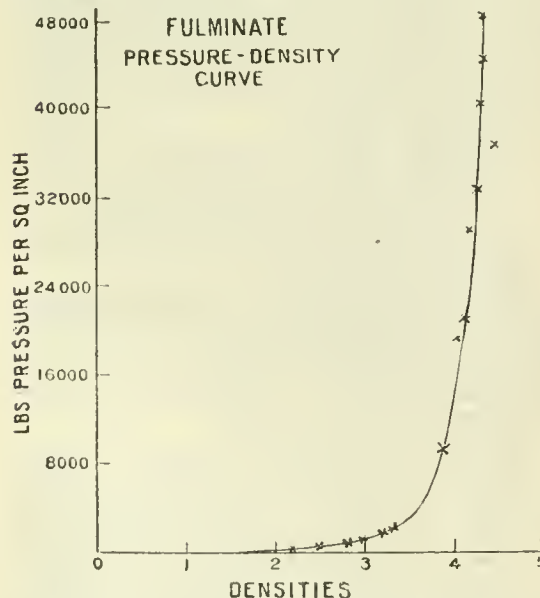


bath, or the fulminate is subsequently bleached in such a mixture. Bleached fulminate is thought to resist moisture when pressed into detonators better than unbleached; this may be due to an alteration of the organic coating which, when damp, tends to reduce the fulminate.

The crystalline form is that of an octahedron; twinning is very common, and takes place in a variety of ways. In each of these ways, successive stages may be traced which form an interesting series. Repeated twinning is also common, and under these conditions the general outline resembles that of the individual crystal, but with serrated edges. Ladderlike forms frequently lead to the clustering round them of crystal fragments forming lumps in the dry powder which give trouble in detonator filling. Crystals with defective outline are found very frequently. Though the substance is very brittle, such outlines cannot be attributed to fracture. Many of them are doubtless due to solution action.

**Specific gravity.** The specific gravity is given as 4.42, but the gravimetric density is much lower. Gravimetric densities are notably difficult to measure owing to the influence of rate of pouring, tapping, etc., but the following values were obtained by following the same regular procedure:—Very fine pale powder, 1.31; very slightly darker, 1.36; darker and coarser, 1.46; another coarse make, 1.50; darkest and most coarse, 1.57. The size of the crystal has therefore a direct relation also to its gravimetric density. This has an important bearing on the charge plates or scoops used in detonator filling, since equal volumes contain different masses when successive batches of fulminate are used. A graded series of plates is advisable where fine limits of weight of charge have to be worked to.

A series of experiments was made to obtain a relation between gravimetric density and pressure. 1.5 grms. of fulminate was pressed into a cylinder 0.36 inch diameter. In the figure pressures are given in lb. per sq. inch.



The density of fulminate pellets and of detonators in general decreases from the surface downwards, and the degree of crushing follows the same order as the density.

**Sensitivity.**—The figures given by different authorities for sensitivity vary considerably as the type of apparatus used greatly influences the

figures quoted; relatively valuable results can, however, be obtained from a given apparatus. It is generally considered that coarse powders are more sensitive than fine, though some doubt has been expressed as to the correctness of this opinion. From personal experience I have satisfied myself that there is no room to doubt the fact that large crystals are more sensitive to impact than small ones. But friction tests under certain conditions seem to show that fine crystals are more sensitive.

The following figures were obtained by dropping a 2-oz. weight a distance of 11½ inches. The powder was placed on a hardened and ground steel anvil, and covered with a hardened steel cylinder. One sample of fulminate was taken and the portions separated by sifting.

Coarse powder remaining on a 120-mesh sieve .....	fired	100
Intermediate, i.e., between 120- and 200-mesh .....	"	50
Fine, passing fine calico .....	"	24

Another sample:—

Coarse powder .....	fired	85
Intermediate .....	"	45

Very fine powder, washed through calico, failed to fire. The fulminate value of the latter was 86%.

The nature of the surface affects the tests considerably, and the presence of fine mercury smoke makes the fulminate very sensitive.

The behaviour of fulminate under friction tests is remarkable. I have found it very difficult to fire fulminate by friction tests between metal surfaces, although a combination of friction with impact, i.e., glancing blows, will fire the substance. Fulminate composition behaves similarly, although it is far more sensitive to impact test. If the fulminate is wetted and then allowed to dry, it becomes sensitive to friction. These facts may be explained by taking the view that rupture of a cake is necessary. On the other hand, fulminate compositions have been repeatedly fired:—(A) When the composition is spread on well painted surfaces—by rubbing with a cloth rag. (B) On American cloth by brushing with a camel hair brush. (C) On leather, using a camel hair brush. (D) On linoleum by rubbing with rubber or leather instruments. In all these cases the surfaces were non conductive, and I am inclined to attribute the explosions to electrical effects.

The fact that the crystals are crushed in detonators, explains why coarseness of powder does not affect the sensitivity of detonators.

**Solubility.**—Fulminate is soluble in cold water, put more so in hot, but is also partially decomposed by hot water, yellow basic salts being formed by the hydrolysis. It is soluble in alcohol, ammonia, pyridine, potassium cyanide solution, and with decomposition in alkalis, thiosulphate solution, etc. Sodium thiosulphate is used to destroy waste fulminate, but if the fulminate is in excess, comparatively large individual crystals of fulminate consisting of numerous twinned crystals of fulminate are copiously deposited. Their length is often from 4–5 mm., and they are 1–2 mm. in width. They are generally pyramidal at either end, and it is interesting to note that it is under these conditions that the crystals develop the tabular habit of growth. From saturated solutions in thiosulphate masses of snow-white fulminate crystals, very regular in shape and of a large size—2 mm. or more in length—are formed. Twinning is very common with these, and a flocculent white precipitate resembling cotton wool, and consisting of long needle-shaped crystals, is frequently deposited. The fulminate crystals so deposited appear to contain water of crystallisation.

Mercury compounds have been generally considered to be inimical to protoplasmic growth, but



I have found fungi growing luxuriously in and under solutions of fulminate of mercury and upon wood, calico, and even the crystals themselves. I am indebted to Miss Lorrain-Smith (of the Natural History Section, British Museum) for kindly identifying the fungi as *Tricoderma viride* and *Acrostalagmus albus* Preuss, respectively, the former growing upon wood and the latter on the calico bag and hence in contact with the crystals.

It is interesting to note in this connection that of the many workers engaged upon the operation of washing and drying fulminate, none of them was ever observed to suffer from the lines on the gums characteristic of mercury poisoning.

These facts accord with the chemical properties of mercury fulminate in that this substance does not give the ordinary reaction for mercury ions.

**Estimation.**—Various methods have been proposed from time to time for the estimation of mercury fulminate, and for the analysis of cap and detonator compositions. Of these the greater number are concerned with the determination of the mercury content of the salt, and in consequence are similar to the standard schemes for analysis applicable in general to mercury compounds. The following examples are typical:—

1. The fulminate is dissolved in warm, dilute hydrochloric acid, and phosphorous acid is added to reduce the mercuric chloride to mercurous chloride, which is filtered, washed, dried at 105° C., and weighed.

2. The fulminate or composition, which may contain antimony sulphide, is decomposed by warming with dilute hydrochloric acid, with subsequent addition of concentrated hydrochloric acid. If antimony is present a little tartaric acid is added, the solution diluted and filtered to remove silica, etc., made alkaline with ammonia, and ammonium sulphide added. The whole is digested for a time to allow the solution of the precipitated antimony sulphide, filtered through a Gooch crucible, and the precipitate washed, treated with alcohol, followed by carbon bisulphide, then alcohol, dried, and weighed as mercuric sulphide.

Divers and Kawakita (J. Chem. Soc., 1884, 13) proposed to decompose the fulminate with a known amount of concentrated hydrochloric acid, and to titrate with alkali the formic acid generated in the decomposition, allowance being made for the hydrochloric acid.

Jones and Willcox (Chem. News, 1896, 283; this J., 1897, 67) proposed to estimate fulminate, potassium chlorate, and antimony sulphide in detonator compositions by mechanical means. Their method was to extract the fulminate by ammoniacal acetone, then remove the chlorate by solution in water, and finally weigh the insoluble antimony sulphide.

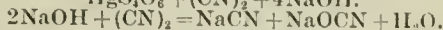
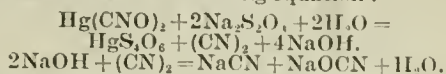
Reechi (Gaz. Chim. Ital., 1904) showed the method of Jones and Willcox to be inaccurate. He suggests treating the composition with water, heating the solution and decomposing the fulminate with hydrogen sulphide. The sulphides are digested with ammonium sulphide, and mercuric sulphide subsequently precipitated with hydrogen sulphide. In the filtrate the potassium chlorate is converted into chloride and so estimated.

H. W. Brownson (this J., 1905, 382) described a method for the analysis of cap compositions based on the reaction between sodium thiosulphate and fulminate in which alkali is generated. His method was to purify the fulminate by solution in potassium cyanide, and precipitate pure fulminate with dilute nitric acid. 0.04–0.05 grm. of this substance was placed in 50 c.c. of water in a flask and 1 grm. of pure thiosulphate added. When the fulminate is dissolved the solution is diluted to 100 c.c. and 25 c.c. is titrated with acid

in the presence of methyl orange. This serves to standardise the acid in terms of fulminate.

For cap compositions of 15–20% fulminate 0.25 grm. is weighed out in order to keep the weight of the fulminate below 0.05 grm., and hence keep the resulting alkali from reacting with the antimony sulphide. Brownson calls attention to the necessity for titrating at once as secondary reactions set in and lead to low results.

Marshall, in his book on "Explosives," states that the reaction has been studied by Philip and, by G. Peace and himself, and that it is in accordance with the following equation:



The cyanides and cyanates produced can be titrated with acid.

Marshall gives the secondary reaction as follows:

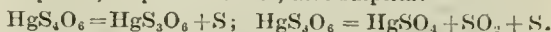


thus accounting for the diminution of the alkalinity when the solution is allowed to stand. He adds that excess of thiosulphate or of potassium iodide restrains the back reaction; for example, ten times as much potassium iodide as fulminate causes only 12% of the alkalinity to disappear in 100 minutes.

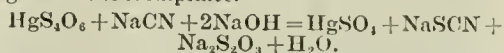
Philip (Z. ges. Schiess- u. Sprengstoffw., 1912, 7, 180, 198, 221; this J., 1912, 606) describes a method in which potassium iodide and thiosulphate are added, the alkalinity titrated as usual with N/10 acid, and the residual thiosulphate titrated with iodine.

It will be observed that the equation given by Marshall for the secondary action is arithmetically incorrect, hence it can only be regarded as a statement in symbols. Further it is evident that one molecule of caustic soda remains unaccounted for.

The reaction is in reality more complex than is indicated by Marshall, as various products are obtained according to the conditions. Thus when fulminate is added to a concentrated cold solution of thiosulphate in a proportion greater than 1 mol. to 2 mols. of thiosulphate the resulting solution is faintly opalescent; after about 1½ hours a grey precipitate consisting of fulminate and of mercuric thiocyanate is formed, and the solution smells strongly of ethereal salts and of cyanide. After further standing the smell of cyanide lessens, a mass of fine needle-shaped crystals forms, the precipitate darkens, and mercuric sulphide is formed. The sludge then develops various colours, from green to scarlet, the composition of the mass being indeterminate. The solution still contains thiosulphate, and I consider a portion of the back reaction to be due to the re-formation of this salt. Thus the tetrathionate breaks up into trithionate which then gives rise to mercury sulphate, sulphur dioxide, and sulphur.



One of the sulphur atoms forms thiocyanate with the sodium cyanide, whilst the other helps to regenerate thiosulphate.



The formation of double thiosulphates of sodium and mercury would account for the non-precipitation of mercury salts from dilute solution with subsequent decomposition into mercury sulphide.

In order to test the effect of various concentrations of thiosulphates on the velocity of the back reaction, quantities of 0.3 grm. of mercury fulminate were dissolved in 50 c.c. of thiosulphate solutions of 2%, 10%, 20%, 50%, and 75% concentration respectively, no potassium iodide being used. The fulminate value of the fulminate used was found in the usual manner, i.e., 0.3 grm. was dissolved in 100 c.c. of water containing 3 grms.



of potassium iodide, and 50 c.c. of thiosulphate added. After 5 minutes the solution was titrated with sulphuric acid, using methyl orange as an indicator.

As a mean of three tests 42.2 c.c. was required, and a blank titration required 0.2 c.c., therefore 42 c.c. of acid was equivalent to the fulminate used. 1 c.c. of acid = 0.007115 grm. fulminate.

Time.	Concentration of thiosulphate.				
	2%	10%	20%	50%	75%
	c.c.	c.c.	c.c.	c.c.	c.c.
5 mins. ....	39.2	—	—	—	—
8 " ....	38.7	41.6	42.0	42.0	42.3
15 " ....	38.5	40.4	41.7	41.5	41.9
30 " ....	35.4	40.0	41.0	41.0	41.3
60 " ....	30.0	39.3	40.2	40.0	40.8

It should be remarked that with 75% concentration it is difficult to get a correct blank owing to the decomposition of the thiosulphate by the acid used in the titration.

From these tests it is evident that the restraining action of thiosulphate is a function of its concentration, but that for practical purposes nothing is gained by using concentrations greater than 20%.

The quantity of fulminate usually taken, *i.e.*, 0.3 grm., does not dissolve readily in the *N*/10 thiosulphate, and if solution is hastened by warming, very erratic results are obtained as the inverse reaction is greatly accelerated. The result of using potassium iodide was of great interest; 0.3 grm. of fulminate when treated with 6 grms. of potassium iodide in 50 c.c. of water required 21.1 c.c. of *N*/10 sulphuric acid to neutralise the resulting alkali, *i.e.*, one half the quantity necessary in the thiosulphate method. With excess of fulminate a white precipitate is formed which yields mercuric iodide on heating or on treatment with acid.

It is desirable to use less expensive reagents than potassium iodide, and I made tests with potassium and sodium chlorides. Six grms. of potassium chloride was added to 50 c.c. of *N*/10 thiosulphate and 0.3 grm. of fulminate was used. In 8 minutes 29 c.c. of acid was required, but it was impossible to get a definite end-point owing to the great rapidity of the secondary reaction. The effect of sodium chloride was similar, but this salt was less energetic in its action.

The experiments described below were performed to see whether acids indifferent to methyl orange could be used, as it seemed possible that by reducing the concentration of the "OH" group, the back reaction might be restrained.

0.3 grm. of fulminate was dissolved in 50 c.c. of *N*/10 thiosulphate, with the addition of 1 grm. of succinic acid; after standing for 8 minutes and titrating, 20 c.c. of *N*/10 sulphuric acid was required, and a further quantity after standing 60 minutes took only 9 c.c. of *N*/10 acid. With salicylic acid similar results were obtained. Boric acid was next tested, 3 grms. being added to the usual quantities of fulminate and thiosulphate. In 8 minutes 41.8 c.c. of *N*/10 acid was used and in 60 minutes 39.8 c.c. The effectiveness of boric acid being apparent, a series of titrations was made and their general agreement proved that boric acid had a marked restraining action, and that it could efficiently replace potassium iodide.

Sodium dihydrogen phosphate is neutral to methyl orange, but acid to litmus, and experiments were therefore made with this salt in order to test the view I had formed, *i.e.*, that it is the presence of free sodium hydroxide which determines the back reaction. As before, 0.3 grm. of

fulminate and 50 c.c. of thiosulphate were used in each experiment.

Weight of phosphate.	Times of standing.			
	8 min.	15 min.	30 min.	60 min.
gm.	c.c.	c.c.	c.c.	c.c.
0.17	38.7	37.4	34.3	32.7
0.5	42.0	42.1	41.6	41.2
1.0	41.95	42.1	42.1	42.0

The end point is not very distinct with the greater concentration, but the results tend to confirm the view I have put forward.

I have often found the need for a complete method of estimating the constituents of detonators when only one sample was available. It is possible to remove the composition from the shell by deforming the latter in two directions at right angles to one another, and to estimate the fulminate by means of thiosulphate, the antimony sulphide as insoluble matter, and the chlorate by difference. But methods by difference are not usually satisfactory, save when dealing with a fairly standard product. Further, it is frequently necessary to determine whether age or exposure has caused a deterioration in the fulminate; for example, it may have reacted with the other constituents, or with the metal envelope. In such cases it is advisable to determine the total mercury in order to find the weight of fulminate originally used, and also the fulminate value to serve as a measure of its decomposition, and finally estimate all other constituents such as antimony sulphide, potassium chlorate, organic substances, etc.

It seemed to me that the first step to be taken was to find the fulminate value by the thiosulphate method and then to remove mercury quantitatively, leaving the solution in a suitable state for an estimation of chlorate.

Vollhard's method of separating the mercury, *i.e.*, by precipitation with fresh ammonium sulphide, treatment with caustic soda and destruction of the sulpho salt with ammonium nitrate, was unsatisfactory and other methods were sought for.

I then tried removing the mercury from the thionate-thiosulphate mixture by means of hydrazine sulphate or the corresponding hydroxylamine salt. With hydrazine sulphate a very clean separation was possible, for example:—0.32 grm. of pure fulminate and 0.1082 grm. of potassium chlorate were dissolved in hot water, 20 c.c. of *N*/10 sodium hydroxide solution was added, followed by 1 grm. of hydrazine sulphate; on heating a black precipitate was formed, leaving a clear solution which filtered readily. The precipitate was washed, dissolved in concentrated nitric acid, heated to oxidise the mercury completely, cooled, diluted, and titrated with thiocyanate in presence of ferric sulphate indicator. 0.224 grm. of mercury was found (theory, 0.226 grm.).

The chlorate in the filtrate was at the same time reduced; the weight found by Vollhard's method was 0.1072 grm., some loss having been occasioned by the oxidation of the solution by means of nitric acid.

No difficulty was experienced in the hydrazine method and concordant results were obtained, but as a further supply of this material could not be obtained the method had to be superseded.

The next reagent used was soda-glucose.

(A) Reduction of chlorate alone. 0.3 grm. of potassium chlorate was dissolved in 50 c.c. of water, 100 c.c. of a mixture of 10% glucose and 10% caustic soda was added, and after boiling for 30 minutes the mixture was cooled, the remaining glucose oxidised with 25 c.c. of nitric acid, and the



chloride precipitated by means of silver nitrate. In three experiments in which 0.3 grm. of chlorate was used the amounts found were 0.295, 0.296, and 0.293 grm. respectively.

(n) Reduction of chlorate in presence of sodium thiosulphate. 50 c.c. of thiosulphate was added to 0.3 grm. of chlorate and 100 c.c. of soda-glucose and treated as before. It was noticed that some sulphur was precipitated on addition of the nitric acid. In three experiments the amounts of chlorate found were 0.298, 0.298, and 0.297 grm.

(c) Reduction of chlorate in presence of mercury fulminate. 0.3 grm. of potassium chlorate and 0.3 grm. of fulminate were treated as above; after reduction the precipitate of mercury and mercury sulphide was filtered off, but some difficulty was experienced in completely freeing the filtrate from mercury compounds.

The results obtained in successive experiments were 0.294, 0.296, 0.298, 0.32, 0.31, and 0.301 grm. (the last two precipitates were found to contain mercury). Unfortunately the mercury could not be accurately estimated owing to the precipitate running through the filtering medium, and all the results were low and not sufficiently accurate to justify the method.

Ferrous sulphate was next tried as the reducing agent, the following method being used:—0.3 grm. of fulminate and 0.3 grm. of chlorate are dissolved in 50 c.c. of thiosulphate, and 3 grms. of boric acid added, and the whole well shaken for three minutes. The fulminate value is then determined by titrating with *N/10* sulphuric acid which has been standardised against pure fulminate in presence of boric acid, the indicator used being methyl orange. If antimony sulphide is present it is filtered off on a Gooch crucible, washed, dried, and weighed. To the solution 100 c.c. of 10% ferrous sulphate, containing 1 c.c. of free sulphuric acid, is added, and it is boiled for 30 minutes. After standing for a short time, it is filtered through a small paper, and the precipitate well washed with water. An excess of nitric acid is added to the filtrate to oxidise the ferrous sulphate, 50 c.c. of *N/10* silver nitrate is added, and the excess titrated with *N/10* ammonium thiocyanate. The reduced mercury precipitate, which contains some sulphide, is dissolved off the paper with *aqua regia* and the solution diluted, neutralised with ammonia, and made slightly acid with hydrochloric acid. The sulphide is now precipitated with hydrogen sulphide, filtered in a Gooch crucible, washed with hot water, followed by alcohol, carbon bisulphide, and alcohol again, dried at 105° C., and weighed as mercury sulphide. The free sulphuric acid in the ferrous sulphate quantitatively precipitates mercury sulphide from the mercury thionate solution.

The method I now use for the complete analysis of detonator composition serves to determine fulminate value, total mercury, antimony sulphide and insoluble matter, and potassium chlorate.

The detonator to be examined, if of the closed-over type, has its lugs cut by means of dental forceps; if of the "obturator" type careful squeezing by means of a shielded press, followed by manipulation with forceps, serves to open it. The composition is then cracked by deforming the shell across two diameters at right angles, when the bulk of it is readily removed. The portions remaining adhering to the walls are loosened by the use of a splinter of wood, using alcohol if necessary. By weighing the detonator before and the cleaned shell afterwards the weight of charge is found. The dried and weighed composition is then broken by moistening with alcohol and gently triturating with a glass rod.

The quantity of thiosulphate to be used is dependent on the weight of the composition. For quantities up to 0.3 grm. 50 c.c. of approximately

*N/10* thiosulphate is used; for larger weights such as 1.5 grms. 50 c.c. of *N/1* thiosulphate should be taken. 0.3 grm. of boric acid is added and the whole well shaken, using a glass rod to aid solution. After 3 minutes a few drops of methyl orange are added and sulphuric acid added till neutral. Any antimony sulphide is filtered off, washed, and weighed, but if powdered glass is present the antimony is estimated volumetrically.

The solution containing the mercury compounds, etc., is next treated with 100 c.c. of 10% ferrous sulphate solution containing 1 c.c. of free sulphuric acid. It is boiled for 30 minutes, the precipitated mercuric sulphide is filtered off, washed, dissolved in *aqua regia*, the solution diluted, neutralised with ammonia, acidified with hydrochloric acid, and precipitated with hydrogen sulphide.

The precipitate is washed, the final washing being done with alcohol, carbon bisulphide, and alcohol again, dried, and weighed. The filtrate containing the reduced chlorate is oxidised with nitric acid, excess of silver nitrate added, and titrated with ammonium thiocyanate. An alternative method for the mercury sulphide precipitate is to dissolve in *aqua regia* containing sulphuric acid, evaporate until fumes are evolved, to remove hydrochloric acid, dilute, and estimate volumetrically with thiocyanate.

I wish to thank Messrs. White and Poppe for their permission to communicate these results, and Miss G. M. Leeson for her helpful observations.

#### DISCUSSION.

Dr. D. F. TWISS, referring to the estimation of mercury fulminate, enquired whether the author had tested the practicability of the alkalimetric titration of a mixture of sodium cyanide and cyanate with standard mineral acid and methyl orange; he was of the opinion that on account of the hydrolysis of cyanic acid into ammonia and carbon dioxide, sodium cyanate would neutralise more than an equimolecular proportion of a monobasic acid such as hydrochloric acid and that the mixture of sodium cyanide and cyanate would therefore not possess a neutralising power equivalent to its content of sodium. With reference to the constitutional formula attributed in the paper to mercury fulminate, he suggested that fulminic acid was now more generally accepted as being an isomeride and not a polymeride of cyanic acid; according to this view there was no direct linkage between the carbon atoms in the acid radicals of mercury fulminate and these atoms were regarded as examples of bivalent carbon, their only attachment being to the nitrogen atom by means of a double bond.

Dr. E. B. MAXTED asked for information as to the reaction between mercury fulminate and potassium iodide. What happened to the CNO? Were stability tests against heat made? In the case of mercuric nitride this was easily done in a capillary tube in a glycerin bath, and it occurred to him whether a similar method could be adopted in relation to mercuric fulminate.

Dr. H. W. BROWNSDON asked for information regarding the preparation of the detonator sections. He doubted the author's conclusion as to the breaking down of the crystalline fulminate into an amorphous variety under heavy pressure and preferred to consider the differences observed as due to crystal size only. When he had first noticed the formation of alkali on treating fulminate with thiosulphate and examined the possibilities of using this reaction as a basis for the quantitative estimation of fulminate, the interference of secondary reactions had been the chief disturbing factor which had to be overcome in order to obtain reliable results.



Mr. F. C. A. H. LANTSBERRY expressed the view, with regard to the breaking up of crystals, that pressure was not likely to cause crystalline material to become amorphous, because generally the crystals occupied a larger space.

Mr. F. H. ALCOCK did not think there was any objection to dealing with the mercury content by precipitation with hypophosphites; it was a reliable method in most cases.

Dr. TROTTER enquired if the colour of fulminate had any bearing on the question of efficiency. There was an idea that the lighter the colour the greater the efficiency.

Mr. HEAVEN, in reply, regretted that he could not answer Dr. Twiss's question. He could not explain the course of the reaction between mercury fulminate and potassium iodide. If a small quantity of the former were used the iodide dissolved it with great readiness. It indicated the presence of a double compound. With an excess of fulminate, however, a point was reached when a fine white precipitate containing iodine was obtained. As to what the actual reaction was he did not know, but alkalinity was developed. The heat test was not followed as a matter of routine or use. He had failed to get a sharp melting point. There was evidence of mercuric vapours being given off at temperatures approaching  $150^{\circ}$  C. He had used the term "amorphous" rather loosely; there was no evidence of the crystalline form up to magnification of 400 diameters. Dr. Brownson's analytical scheme had been of great service to him. Bar tests had been employed on detonators on the Hopkinson principle. The question of whether a relationship existed between colour and efficiency remained unsettled. The balance of opinion was that there was no relationship.

### Nottingham Section.

Meeting held at Nottingham on Wednesday, April 24th, 1918.

DR. R. M. CAVEN IN THE CHAIR.

#### A MODIFIED MERCURIAL VISCOMETER FOR DETERMINING THE VISCOSITY OF VOLATILE LIQUIDS.

BY F. M. LIDSTONE.

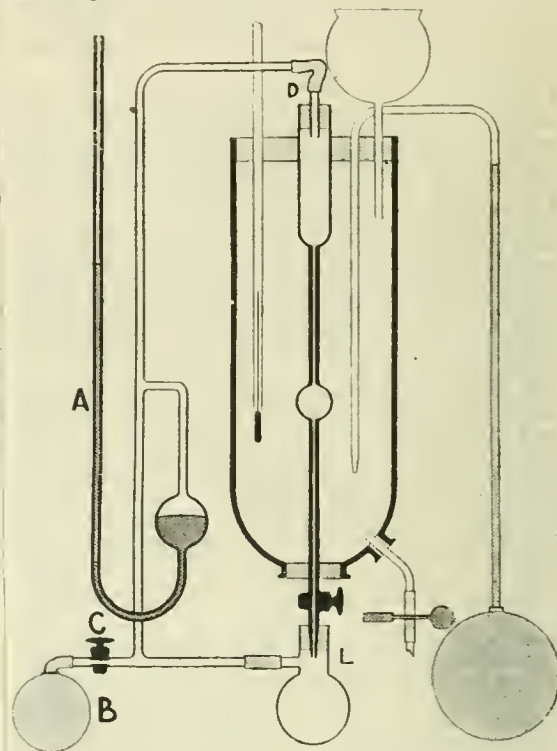
In a paper read in January, 1917 (this Journal, 1917, 270), a description was given of a mercurial viscometer and its application to the measurement of the viscosity of lubricating oils. Since that paper was written, however, liquids other than oils have been at different times run through the instrument, with the result that it has been found that the apparatus as there described is not adapted to the testing of liquids of high vapour tension, inasmuch as a cushion of vapour forms at the junction of the mercury and liquid being tested.

On consideration it will be seen that this takes place when the vapour tension is greater than  $P - H$ , where  $P$  is the atmospheric pressure and  $H$  the head of mercury in the viscometer.

The apparatus to be described here was designed to remedy this defect in the readiest and simplest manner by means of an attachment which would not interfere with the ordinary working of the viscometer and would also allow the usual constants of the instrument to be used without any alteration.

A is a mercury manometer which registers the increase of pressure, which is applied equally to

the top and bottom of the moving column in the viscometer. If this pressure be  $p$ , the atmospheric pressure  $P$ , and the head of mercury in the viscometer  $H$ , then if  $P + p - H$  be equal to or exceed the vapour tension of the liquid, at the temperature of the experiment, there will be no separation of the mercury and liquid taking place during the running.



The pressure is conveniently applied by means of a hand bellows, B, and the stopcock, C, is then closed. If the rubber connections are properly airtight there will be no need to pump in any more air during the running; also it will be seen that no matter how great is the pressure,  $p$ , the ordinary constants of the instrument remain unchanged.

Examples of liquids which cannot be run at ordinary temperatures through this viscometer without the application of increased pressure are:—Ether-alcohol solutions of nitrocellulose, acetone solutions of cellulose acetate, solutions of rubber in volatile solvents, etc. It can be used for all volatile liquids at high temperatures; for example, with this apparatus the viscosity of ether may be determined at temperatures much above its normal boiling-point.

There are other useful purposes which this attachment can be made to serve. Thus thin and quick-running liquids can be made to take a longer time by applying a known pressure at the end L, whilst the end D remains open; if the decrease in the head is accurately measured by the manometer the readings can easily be converted. Conversely, a known pressure can be applied at D, L remaining open, and a thick and slow-running oil may thus be made to run more rapidly. When pressures are applied in this manner, care must be taken to tap the manometer frequently in order to overcome the peculiar lag in the mercury and to get a correct measure of the pressure which is being applied. A globe or flask of large capacity must also be interposed in order that the small change in pressure due to the volume of the mercury which is leaving the viscometer may not have any

sensible effect. Of course, when the pressure is applied simultaneously to top and bottom, as in the case of volatile liquids, these precautions are not necessary. After a thick oil has been run, and the reading taken, it also saves time to force the oil through the viscometer by applying pressure at the top of the column instead of waiting for the oil to drain out. The tube is then washed with ether as usual.

The author takes this opportunity of correcting a mistake which appeared in the previous paper (*loc. cit.*). The error referred to was in the formula there given to correct for the time taken by the mercury in the case of very mobile liquids. As will be seen, it is impossible to give a simple formula which is mathematically correct for all cases, but a very close approximation is arrived at as follows:—

Let  $t$  = time taken by mercury flowing freely,  $v$  = mean velocity of mercury flowing freely,  $T$  = time taken by mercury retarded by oil,  $V$  = mean velocity of mercury retarded by oil, and  $m$  = mass of mercury.

Then  $\frac{1}{2}m(v^2 - V^2)$  = work spent on oil. Hence when  $V$  is made infinitesimally small,  $\frac{1}{2}mv^2$  represents the total available work which the mercury can do. The time,  $T$ , can then be converted into the time which would be taken if the whole of the available work were spent on the oil, this time being:—

$$T \frac{\frac{1}{2}m(v^2 - V^2)}{\frac{1}{2}mv^2} = T \left( \frac{1}{t^2} - \frac{1}{T^2} \right) = T - \frac{t^2}{T}$$

This time would then be proportional to the absolute viscosity if the mass and kinetic energy of the liquid could be neglected, or, on the other hand, if  $t$  could be taken to represent the time which the mercury would take together with an imaginary liquid of unit density, but with absolutely no viscosity, the equation could be written:—

$$\eta = k \left\{ T - \frac{\left( t \frac{H+h}{H+hd} \right)^2}{T} \right\} \frac{H+hd}{H+h}$$

where  $H$  is the mean head of mercury,  $h$  the mean head of liquid of unit density, and  $d$  the density of the oil.

Now by making the bore of the tube below the bulb relatively large, the time,  $t$ , becomes small and its correction negligible. The equation then becomes:—

$$\eta = k \left( T - \frac{t^2}{T} \right) \frac{H+hd}{H+h}$$

or by putting  $\frac{H}{h}$  equal to  $n$  and  $\frac{k}{1+n}$  equal to  $K$  the equation can be written:—

$$\eta = K \left( T - \frac{t^2}{T} \right) (d + n)$$

where  $K$  and  $n$  are constants,  $T$  is the observed time of running,  $t$  is time taken by mercury alone, and  $d$  is specific gravity of the oil.

In calibrating the viscometer the density,  $d$ , is taken as unity for water at 60° F., as in actual practice specific gravities of liquids are usually referred to water at this temperature, and thus a number of small corrections are obviated.

My thanks are due to Mr. L. Archbutt for permission to make the necessary experiments and the apparatus itself in the Chemical Laboratory of the Midland Railway Company at Derby, and also to Mr. R. W. Lunn for kindly preparing the sketch.

#### DISCUSSION.

Mr. J. M. WILKIE said that when using the ordinary Redwood viscometer it was necessary

to employ considerable quantities of oil, and the operation required the expenditure of considerable time. As a result of Mr. Lidstone's work an apparatus has been devised which gave results comparable with those obtained by the Redwood apparatus and furthermore made it possible to obtain results in a much shorter time and with the use of considerably less material. The apparatus had been adopted in Messrs. Boots' Analytical Laboratory, and the results had been entirely satisfactory.

Mr. Woodhouse said that at present he was using the Coleman-Archbutt glass apparatus and had found that it gave reliable results. He had made one slight modification of this apparatus by surrounding the efflux tube with a glass bulb which was immersed in water, so as to maintain the oil inside the tube at the same temperature as the oil outside the efflux tube prior to leaving it. By this means he had obtained results which compared favourably with figures obtained with the Redwood apparatus.

#### THE INFLUENCE OF SMALL QUANTITIES OF SOME METALLIC ELEMENTS ON THE MECHANICAL PROPERTIES OF BRASS.

BY E. MILLINGTON.

The experiments to be described in this note were carried out to ascertain the effect of some metallic elements on the mechanical properties of a certain type of brass.

Shepherd, Carpenter, Edwards, Hudson, Hoyt, and others have made exhaustive investigations into the constitution of brasses, and as a result various diagrams have been framed embodying their conclusions. It is by the aid of these diagrams that the mechanical properties can to some extent at least be predicted.

For certain purposes the strength of ordinary brass is insufficient and it becomes necessary to modify the alloy in accordance with the conditions to be met. It is well known that the addition of other elements may profoundly modify the physical properties of the pure brass, either by exerting their own peculiar properties or by introducing an additional phase, or phases. It is, therefore, necessary so to adjust the mixture by selecting the element or elements to be added which are the most suitable for the purpose in view.

In the case of copper-zinc-tin alloys the theoretical and practical side has been worked out by Hudson, Hoyt, Thurston, and others, and from their results it is possible to predict what effect is likely to be produced in given circumstances. But as many other elements are employed and as the information concerning them was not available, it was decided to investigate the matter as far as the immediate requirements were concerned.

In the alloys to be described the choice of metals, other than copper and zinc, was limited to tin, iron, manganese, and aluminium. Owing to the restrictions in the use of pure metal for industrial purposes it was only possible to use materials of commercial purity.

Since the object of these tests was to represent castings as such, without any subsequent treatment, the specimens to be tested were cast both in sand and iron chill moulds, and since the work had to represent ordinary foundry conditions nothing further than foundry practice was observed in the preparation of the alloys, but at the same time every care was taken with the melting and the casting of the metal.

The actual figures as obtained from tests are given in the tables and also plotted in Fig. 3. The effect of different rates of cooling, as produced when the metal is cast in sand or in iron chill moulds, is clearly shown in this figure.



The range covered by the alloys was Cu 56—60%, Zn 37—41%, the difference being made up by the addition of a third element. It will, therefore, be seen that the brass was of that type known as Muntz metal.

From the constitutional diagram of the copper-zinc series, a portion of which is shown in Fig. 1, it will be seen that for varying composition corresponding phases are met with in normally cooled or annealed solid alloys. Thus in alloys containing not less than 63% of copper, the system is one of single phase, viz.,  $\alpha$ ; with less than 63% and more than 53% of copper, the system is duplex  $\alpha$  and  $\beta$ . In the interval of

selves in any given alloy. The  $\alpha$  constituent for example is the one having the greatest ductility, while  $\beta$  is the one imparting the greatest strength. The presence of either  $\gamma$  or  $\delta$  in a brass may seriously affect the ductility and also the strength of the alloy.

As an example illustrating the effect of the  $\gamma$ -phase the following figures, which have been kindly furnished me by Mr. Archbutt, may be quoted:—

#### Analysis of the alloys.

	No. 1. %	No. 2. %
Copper .....	60.70	50.69
Tin .....	0.01	0.02
Lead .....	2.06	1.22
Aluminium .....	0.06	0.49
Iron .....	0.04	0.03
Nickel .....	0.07	0.03
Zinc .....	37.02	47.46
	99.96	99.94
Yield point, tons per square inch .....	9.2	8.4
Maximum stress, tons per square inch .....	24.4	16.7
Elongation % on 2-inch .....	41.0	4.0

The explanation for the remarkable difference in physical properties of the two alloys is to be found in their micro-structure, the good properties being associated with the duplex  $\alpha + \beta$ , while the inferior strength accompanies the  $\beta$  and  $\gamma$  formation.

Thus, the subject resolves itself into one having for its object the reinforcing of the  $\alpha$  and  $\beta$  duplex by means of the addition of other elements in suitable proportions which shall give the necessary strength, at the same time avoiding the formation of the two deleterious phases  $\gamma$  and  $\delta$ .

Owing to the similarity in appearance of the two constituents  $\gamma$  and  $\delta$  when viewed through the microscope it is difficult to distinguish with certainty one from the other in a ternary alloy, since they both present a bluish colour after etching and for this reason they will be referred to as a blue constituent.

The results embodied in the diagram, Fig. 3, will now be considered, commencing with the copper-zinc alloy which forms the basis of this series. The composition of this alloy is Cu 59.72 and Zn 40.18%. The mechanical properties in the sand-cast specimen are yield-point 8.4, and maximum stress 22.7 tons per square inch, with an elongation on 2 ins. of 47.7%. These figures are notably increased in the chill-casting as will be seen from the diagram.

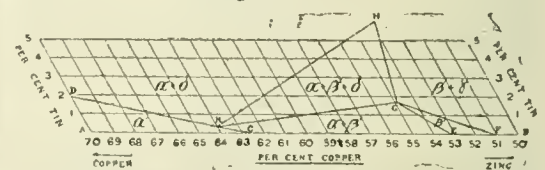


FIG. 2.

#### Copper, zinc, tin, ternary diagram.

Since a similar relationship holds good in sand- and chill-cast specimens it will only be necessary here to consider the results in one case. Selecting the sand casting for this purpose, it will be seen that the introduction of 1.11% of tin, which in this case displaces the copper, produces an increase in yield point of 19 and maximum stress of 11% respectively, at the same time lowering the elongation by 61%. This reduction in elongation does not appear to be due to the presence of a third constituent, as microscopical examination failed to reveal any and this is in accord with the diagram (Fig. 2), which shows at 58.23% of copper only

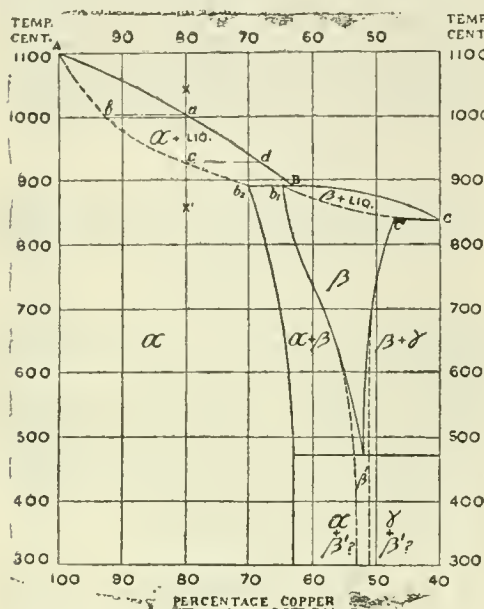


FIG. 1.

#### Equilibrium diagram of copper-zinc alloys.

61%—53% copper, again, the system is one of single phase viz.,  $\beta^*$ , while 51%—40% copper produces the duplex  $\gamma$  and  $\beta$ .

The ternary system of copper-zinc-tin has received some attention by Hudson and Jones. Their diagram (Fig. 2) represents this system with some degree of accuracy, covering alloys from 50%—70% copper and 0—5% tin.

It will be noticed that the three branches  $\alpha$ ,  $\alpha$  and  $\beta$ , and  $\beta$  and  $\gamma$  of the copper-zinc system are represented. The chief features to observe are the varying solubility of tin and the phase-producing effect in the branches referred to. Commencing at 70% copper it will be seen that the solubility of tin below the line D—C decreases with the decrease of copper down to 63%. Below this line the  $\alpha$  phase is retained while above it the duplex  $\alpha + \delta$  makes its appearance. On the other hand in the branch lying below the line K—G the solubility of tin increases with a still further increase of copper.

Above this line the three-phase system  $\alpha$ - $\beta$ - $\delta$  is found.

In the area G.E.F. the solubility of tin again falls, giving rise to the single-phase alloy  $\beta$ ; while above the line C—F the duplex  $\beta + \gamma$  occurs.

In using the constitutional diagram it is important to remember the respective properties of the possible phases which may present them-

\*Carpenter and Edwards regard this constituent as a duplex  $\alpha + \gamma$  (see J. Inst. Metals, 1911, 5). For practical purposes the author uses Shepherd's diagrams (J. Inst. Metals, 1901, 1).

the normal duplex  $\alpha + \beta$  possible. The only conclusion is that other causes are present and this is probably due to the hardening influence of the tin itself. Further increase of tin to 2.13% raises the yield point by 33%, and the maximum stress by 4%, and reduces the elongation by 83% compared with the copper zinc basis alloy. In this case the blue etching constituent was found to be present, which is supported by Fig. 2, showing that with copper at 57.51% (the copper content of the alloy) the three phases  $\alpha$ ,  $\beta$ , and  $\delta$ , would be present.

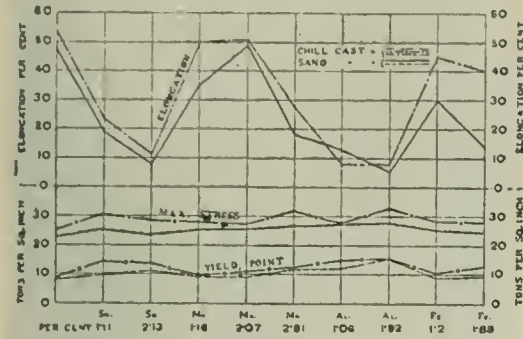


Fig. 3.

Ternary brass alloys.

Displacing tin with 1.16% manganese increases the yield and maximum stress by 11.9% and 12.3% respectively, with a decrease in elongation of 25.5% compared with the original brass. An increase in manganese to 2.07% results in the same tenacity being retained but gives a much improved elongation, being actually 2.7% greater. In this instance in the chill cast specimen elongation is less by 6% than in the corresponding basis alloy. A still further increase of manganese to 2.8% has a very marked effect by considerably raising the strength, the yield and maximum stress increasing by 42.7% and 18.9% respectively; at the same time the elongation is reduced by

lites in large  $\beta$  crystals, giving every evidence of brittle material.

The presence of 1.92% of aluminium notably raises the yield and maximum strength, viz., by 88.9% and 23.3% respectively, and simultaneously reduces the elongation by 88.4%. Microscopical examination revealed the presence of the blue etching constituent in a structure which is otherwise apparently a single phase  $\beta$ . This occurs in the presence of 59.06% of copper, which makes the case more remarkable as in the ordinary brass such a condition would normally not be found with more than 51% of copper. It is not, therefore, surprising that the ductility should fall.

The effect of 1.2% of iron is to increase the yield point 9.5% and the maximum strength 12.7%; at the same time the elongation, while being much superior to that of the aluminium alloys, is reduced by 37.1%. The presence of 1.88% of iron notably raises the yield point but slightly reduces the maximum stress compared with 1.2% alloy; at the same time the elongation is seriously impaired. While microscopical examination failed to reveal any blue etching constituent the 1.88% alloy contained this phase and this probably accounts for the loss in ductility.

It will, therefore, be seen that the addition of the third element to the binary copper-zinc alloy has been limited to 2.0%, with the exception of manganese, which was carried to 2.81%, and as this served the immediate purpose the investigation was concluded.

The author is fully aware of the limited scope covered by the paper. What is now required is to extend the investigation into the still more complex systems and this as time and opportunity permits is being carried out. However the results of this extension up to the present do not admit of conclusions being drawn and much requires to be done before it can be completed.

Before concluding reference may be made to an experiment which has some bearing on the subject of greater complexity. An alloy having the composition, Cu 54.10, Pb 0.42, Al 0.40, Fe 2.51, Mn 0.89, Ni 3.58, Zn 38.03%, gave in the testing machine:—

State.	Tons per square inch.		Elongation, % on 2 in.	Contraction of area, %.
	Yield.	Max. stress.		
Cast in sand.....	11.6	23.7	13.5	23.1
Cast in iron chill mould.....	13.2	32.5	43.0	42.0

62.2%. A microscopical examination failed to show anything but the duplex  $\alpha + \beta$  structure. The remarkable reduction of elongation in the case of 2.8% manganese can only be accounted for by the hardening effect of this element itself and this is confirmed by the corresponding increase in strength.

The addition of 1.06% of aluminium results in a very marked effect on the ductility of the alloy, reducing this property by 72.7% in the sand and 85.2% in chill casting respectively, at the

These figures present two striking features, the first being the great difference between the sand cast and the chill cast specimens and the second the superior results obtained by the latter method of casting.

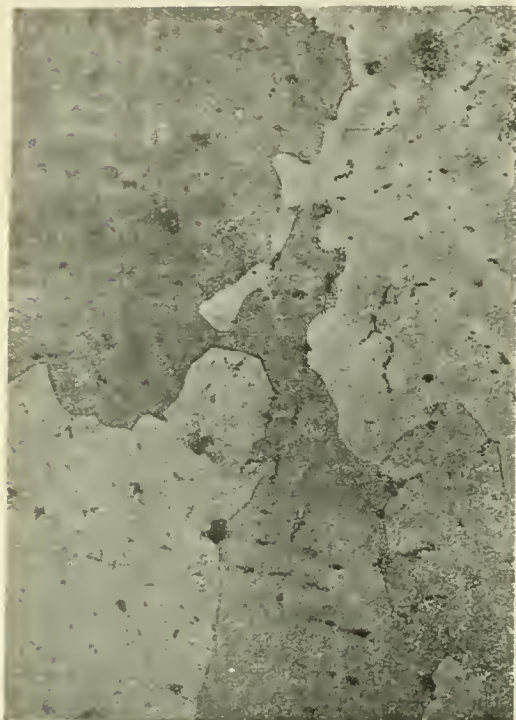
With the object of increasing the strength of this alloy tin was added and the following is the analysis of the resulting alloy:—Cu 53.86, Sn 0.88, Pb 0.43, Al 0.38, Fe 2.31, Mn 0.84, Ni 3.33, Zn 38.05%; on testing the following results were obtained:—

State.	Tons per square inch.		Elongation, % on 2 in.	Contraction of area, %.
	Yield.	Max. stress.		
Cast in sand.....	18.0	26.4	5.5	15.2
	16.8	29.5	7.5	15.2
Cast in iron chill mould.....	19.8	35.8	13.0	21.6
	19.2	35.7	15.0	21.6

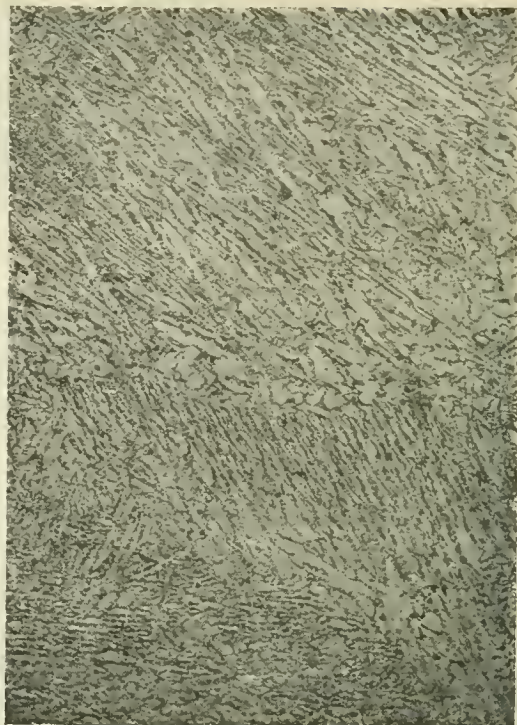
same time increasing the strength. It will be noticed that 1.0% of aluminium has an effect similar to that produced by 2.13% of tin. The structure of this alloy was coarse, acicular crystal-

These figures require no comment; the strength is very much improved, particularly the yield point, but at the expense of a serious loss of ductility.

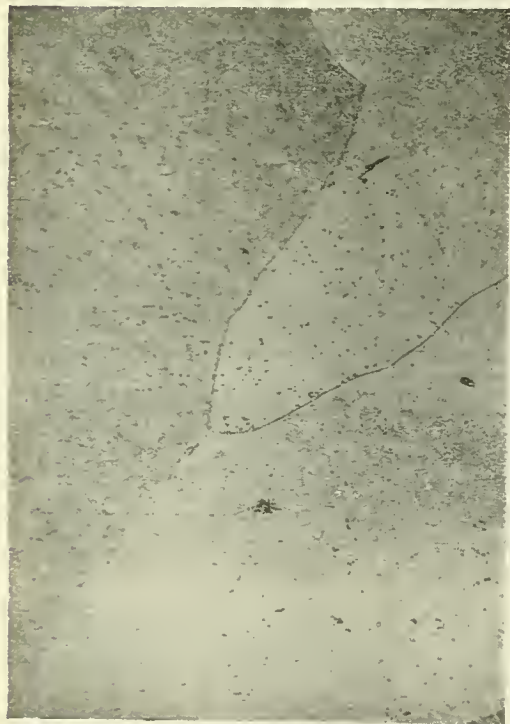




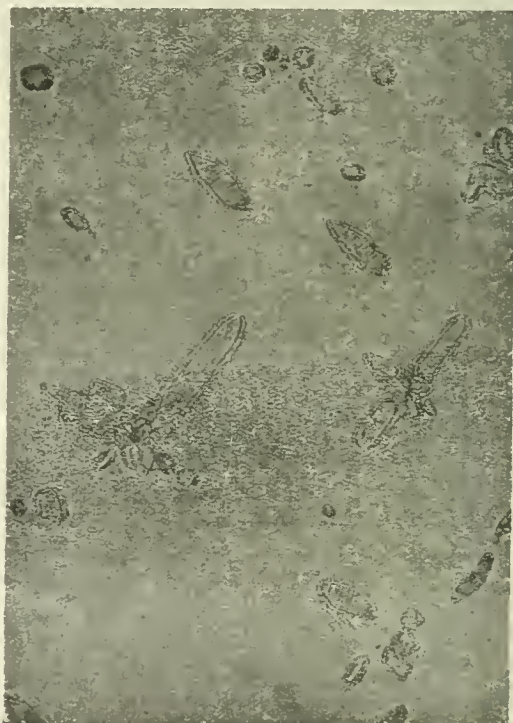
Cu 70, Zn 30% approx.  
Cast in sand mould.  
Mag. = 50 dia. V.  
 $\alpha$  cored structure.



Cu 60, Zn 40% approx.  
Cast in sand mould.  
Mag. = 50 dia. V.  
 $\alpha + \beta$ .

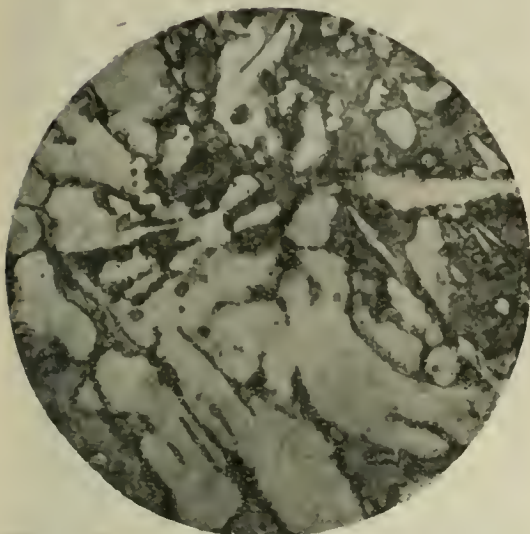


Cu 55, Zn 45% approx.  
Cast in sand mould.  
Mag. = 50 dia. V.  
Typical  $\beta$  area.



Cu 50, Zn 50%  
Mag. = 300 dia. V.

$\beta + \gamma$



Cu 57.51, Zn 40.06, Sn 2.13%  
Cast in iron chill mould.  
Mag. = 1000 dia. V.  
 $\alpha + \beta$  + blue constituent.



Cu 56.74, Zn 40.17, Pb .23, Mn 2.81%  
Cast in iron chill mould.  
Mag. = 1000 dia. V.  
 $\alpha + \beta$ .



Cu 60.23, Zn 36.91, Pb .49, Fe 1.88%  
Cast in iron chill mould.  
Mag. = 1000 dia. V.  
 $\alpha + \beta$  + blue etching constituent.



Cu 59.06, Zn 37.98, Pb .78, Al 1.92%  
Cast in iron chill mould.  
Mag. = 500 dia. V.  
 $\beta$  + blue etching constituent.



Time has not permitted a microscopical examination being made on these alloys, in which case the explanation for the difference in the ductility would most likely be found, especially having regard to the effect of tin and iron previously discussed.

In conclusion I desire to thank my friends at the Midland Railway Company for their kind assistance, particularly Messrs. Archbutt and Adams, the former for the analyses of the alloys and the latter for the photomicrography.

*Results of tests compared with basis (simple) copper-zinc alloy.*

Element.	%	Cast in sand. % Difference.			Cast in iron chill. % Difference.		
		Yield point.	Maximum stress.	Elongation.	Yield point.	Maximum stress.	Elongation.
		%	%	%	%	%	%
Sn .....	1.11	+19.0	+11.0	-61.2	+77.5	+19.4	-57.4
Sn .....	2.13	+38.0	+4.0	-33.0	+73.7	+13.8	-79.2
Mn .....	1.16	+11.0	+12.3	-25.5	+20.0	+10.3	-7.4
Mn .....	2.07	+11.0	+12.4	+2.7	+37.5	+8.7	-6.0
Mn .....	2.81	+42.7	+18.0	-62.2	+60.0	+26.2	-49.0
Al .....	1.06	+50.0	+20.6	-72.7	+88.7	+9.1	-85.2
Al .....	1.92	+83.0	+23.3	-38.4	+97.5	+30.0	-84.8
Fe .....	1.20	+9.5	+12.7	-37.1	+30.0	+12.7	-16.6
Fe .....	1.83	+14.3	+8.6	-70.6	+60.0	+11.0	-26.0

Original brass. (Cu 59.77, Zn 40.18, Pb 0.03 %)	Tons per square inch.		
	Yield.	Maximum stress.	Elongation, % on 2 in.
Cast in sand .....	8.4	22.7	47.7
Cast in chill mould .....	8.0	25.2	54.0

#### DISCUSSION.

The CHAIRMAN asked if the metals indicated in Fig. 3. were added so that only one was present at a time in addition to the zinc and copper.

Dr. E. P. HEDLEY remarked that the equilibrium diagram (Fig. 1) differed from that originally published by Shepherd, a horizontal line having been inserted from the point representing 37% to 60% Zn at a temperature slightly below 500° C. He asked the reason for this alteration. From the appearance of the first photomicrograph it might be imagined that there was more than one phase present, whereas the photograph was intended to represent the homogeneous  $\alpha$ -phase. The paper emphasised the importance of considering the effect of mere traces of metal. He referred to the amount of work that would be required to obtain equilibrium diagrams for the complex alloys referred to in the latter part of the paper, containing seven and eight metals respectively. The difficulty of making a complete metallographic study of a system of alloys increased very rapidly with the number of component metals and a quaternary system would necessitate several thousand determinations being made.

Mr. MARSHALL asked for some information as to the best method of treating swarf.

Mr. WYER asked if any special temper was given to the samples used in the experiments referred to. He also mentioned that his firm had made an attempt to increase the temper of an ingot metal by the addition of ferromanganese but that the result had been a failure.

Mr. MILLINGTON, in replying to Dr. Caven, said that Fig. 3 was intended to show the effect of adding one metal at a time to the mixture of copper and zinc. The line referred to by Dr. Hedley in the equilibrium diagram had been omitted

by Shepherd in his diagram. There was, however, no doubt that such a line existed and should be embodied in the diagram as it represented a series of heat evolutions originally observed by Roberts Austen (4th Report to the Alloys Research Committee, Inst. Mech. Eng., 1897, p. 36, plates 2 and 3), and the line had recently been determined by Carpenter and Edwards (J. Inst. Metals, 1911). There was some doubt as to the real significance to be attached to this line. Carpenter and Edwards considered that at this

temperature (470° C.) the  $\beta$ -phase underwent decomposition into  $\alpha + \gamma$ , whereas Hudson (J. Inst. Metals, 1914, 88-99) considered it to be due to a polymorphic transformation of the  $\beta$  constituent and to represent the areas below the line as composed of  $\alpha + \beta$ ,  $\beta$ ,  $\beta + \gamma$  respectively. For practical purposes it was sufficient to recognise the existence of this line and to work on Shepherd and Hudson's conclusions regarding the phases present. For these reasons in Fig. 1. Carpenter and Edwards' results were shown in full lines while Shepherd's conclusions were given in dotted line. The reason for the heterogeneous appearance of the  $\alpha$  phase in the first photomicrograph was owing to the fact that during the solidification of the alloy the final condition was not at once arrived at, as the central part of each individual crystal of such a solid solution when first formed contained considerably less of the solute than the liquid alloy from which it was deposited, and as solidification proceeded the "core" of relatively dilute solution was surrounded by successive layers of more concentrated solution. The result was the appearance of these "cores" within the polyhedral crystals. If such an alloy were annealed the "cores" disappeared and the alloy then appeared perfectly homogeneous. However, if the annealing had been accompanied by mechanical work the disappearance of the "cores" was accompanied by the formation of numberless "twinned" crystals. In the preparation of equilibrium diagrams it was essential that the component metals should be as pure as possible if the diagrams were to be of any practical value. The addition of a third component might result in the precipitation of one of the other components which had been previously in solution, whereas the addition of another might result in the whole becoming a homogeneous mass.

In connection with the melting of swarf (the turnings obtained from the working of brass) he stated that the operation could be accomplished by employing any of the standard forms of heating apparatus. It was advisable to melt a small quantity of the swarf at the commencement and gradually to add the remainder to the liquid, in small quantities at a time, until all was molten. By the addition of an amount of aluminium equal to 0.25% of the weight of metal treated, the resulting alloy was much superior owing to the deoxidising effect of this metal.

In the preparation of the complex alloys referred to, the chief point to be borne in mind was first to melt the metal or preparatory alloy which possessed the highest melting point and then to add the other constituents in the order of their melting points. By this means overheating of the mass was obviated and oxidation reduced to a minimum. For good melting the molten metal should be kept covered with charcoal and a suitable flux; one that had given good results was composed of two parts of common salt and one part of borax.

## Yorkshire Section.

*Meeting held at Queen's Hotel, Leeds, on Monday, April 15th, 1918.*

MR. W. MCD. MACKEY IN THE CHAIR.

### PRELIMINARY NOTE ON THE PRESENCE OF BISMUTH IN THE HUMAN BRAIN.

BY T. FAIRLEY, F.I.C., AND B. A. BURRELL, F.I.C.

Some time ago we received the viscera of a man whose death was suspected to be due to slow poisoning. The subject, who was in middle life, had been ill for some months and during the illness had taken considerable quantities of preparations of bismuth, which had been prescribed by his medical attendant. During the course of the analysis we were therefore not surprised to find this element present in the intestines, stomach, liver, spleen, kidneys, and bladder, as Hale White says it is very slowly absorbed and excreted chiefly in the urine, and may be found in the liver, kidneys, spleen, and nervous system.

Amongst the organs examined was a portion of the brain; this also showed distinct evidence of the presence of bismuth, and we hope at some future date to give the quantity present in this and other organs.

We have not been able to find any recorded instance of bismuth being detected in the human brain, and it is an interesting speculation as to what action this metal will have on the brain substance and therefore on the mental condition of those who are taking a long course of this widely-used drug.

The extensive use of bismuth preparations is shown by the following, quoted from Martindale and Westcott's "Extra Pharmacopoeia," 8th Ed., 1895:—"Analysis of the ingredients of 25,500 prescriptions showed Tinct. Nucis Vom. to occupy the highest place, having entered into 2168 prescriptions, and bismuth preparations, including the nitrate, carbonate, and Liq. Bism. et Am. Cit., to be ninth on the list, having entered into 1112 prescriptions.

The method used for the extraction of bismuth was that usually given in works on toxicology. A very delicate test for bismuth (which will detect 0.00025 grm. of Bi in copper) is that of Abel and Field.

### DISCUSSION.

Mr. JAMES MILLER asked whether the lead iodide test was regarded as more delicate than potassium iodide.

Mr. BURRELL replied that it was advised by Abel and Field for the detection of bismuth in copper, and that he certainly thought it the most delicate, after comparing it with other tests.

### FILTERING MEDIUM USED IN THE ESTIMATION OF WOODY FIBRE.

BY T. FAIRLEY, F.I.C., AND B. A. BURRELL, F.I.C.

In a recent note (Analyst, 1918, 43, 31), J. A. and E. W. Voelcker emphasise the desirability of some agreement being made as to the strength of the acid and alkali used in the determination of woody, or, as it is sometimes called, crude fibre in cattle feeding stuffs. In common with what we believe to be a large majority of chemists, we use the 1.25% strength of acid and alkali, as against the 2% strength recommended by Messrs. Voelcker.

We think that the filtering medium used in this empirical method is of almost equal importance as the strength of the acid and alkali. The majority of chemists use a piece of fine linen (Frankland's "Agricultural Analysis" says fine muslin) as recommended by most of the text books on agricultural analysis. Wiley states that the first filtration (acid) may be made through linen, glass wool, or asbestos, and the second (alkali) through a Gooch crucible. We submit that "fine linen" is an expression capable of a wide and variable meaning, and it follows that the mesh will also be variable; for instance comparable results could not be obtained by using in one instance a fine linen pocket handkerchief and in another a fine linen tablecloth as the filtering medium, though both of these are equally "fine linen."

In the dry state, the mesh of a fine linen handkerchief in several we have examined ranged from about 90 to 110 to the linear inch. We have not made any determinations when wet as it would be if used as a filter, though obviously it will be altered by the swelling of the fibre, and there is also the action of the dilute acid and alkali to be considered.

Owing to the uncertain character of "fine linen" we have discarded its use as a filtering medium and use instead copper gauze with 100 meshes\* to the linear inch.

The results obtained with such gauze are somewhat lower than when linen is used, as the following figures show:—

	Linseed cake.	Barley meal.	Palm kernel meal.	Bran.
	%	%	%	%
Gauze .....	8.3	6.1	18.4	10.6
Linen .....	9.5	6.9	20.9	12.2

### DISCUSSION.

The CHAIRMAN asked if the gauze itself was unaffected.

Mr. BURRELL replied that it was. It could be used for months if carefully washed and dried.

Mr. F. W. RICHARDSON did not think it would be possible to have a process that would be applicable to all substances, because the fibres varied greatly in different materials. He had tried various processes, but now he always used an electrical centrifuge, working at a very high rate of revolution. This saved the whole process of filtration, and, of course, avoided the problem of the size of the mesh. In the case of flours he emulsified them and then treated them with malt extract at 155° F., afterwards adding the equivalent of 0.2% of hydrochloric acid and using a very pure solution of pepsin. He thus got a very complete digestion without the action of acids or alkalis, and obtained a good idea of the amount of indigestible cellulose. He thought the copper gauze would not eliminate the whole of this, since it was possible that it would

\* The same size as is used for testing the "fineness" of basic slags.



not prevent the more finely divided particles from passing through.

Dr. H. INGLE said it was well known (Ingle, this J., 1904, 1197) that corky matter could only be saponified in a rather strong solution, and he thought the 1½ or 2% mentioned by the authors would not do for such matter. The so-called indigestible matter might contain cork, and he asked if there was any chance of its remaining unsaponified. Personally he thought it would.

Major MARPLES said it seemed to him that there was very little to choose between the two methods. In using a centrifuge he had found difficulty in the washing, and this he thought was quite as objectionable as the trouble of filtration.

Mr. BURRELL, replying, pointed out that the method suggested was purely an empirical one, and was capable of improvement, but still it served a useful purpose. The point suggested by Dr. Ingle was a very interesting one, because—with reference to the Voelckers' note—the usual strength of solution used in this country, on the Continent, and in America was 1½% acid, followed by 1½% alkali. In their laboratory they had used 1½% solutions for thirty years, but the Voelckers affirmed that this was not sufficient to effect hydrolysis and that 2% was necessary.

## Communications.

### THE COMPARATIVE EFFECT OF ORGANIC AND INORGANIC ACCELERATORS IN VULCANISING RUBBER.

BY HENRY P. STEVENS.

Various statements have been made regarding the comparative accelerating effect of inorganic accelerators, such as litharge and magnesia, and the more modern organic accelerators.<sup>1</sup> It has been stated that relatively large quantities of the former are necessary to produce an accelerating effect comparable with that obtained by means of small quantities of the latter. It is also stated that the physical properties of the rubber vulcanised with inorganic accelerators differ from those obtained with organic accelerators, and it appears to be generally held that, of the two inorganic accelerators, litharge is more powerful than magnesia. My experience led me to question some of these statements, and I give the results of a number of vulcanising experiments with 90 parts of ordinary pale plantation crêpe rubber, 10 parts of sulphur, and small quantities of the following accelerators:—(1) litharge, (2) magnesia,<sup>2</sup> (3) isonitrosodimethylaniline (Ac. I), (4) a basic amino derivative (Ac. II). All the compounds were vulcanised in steam for one hour at 35 lb. pressure (138° C.). Portions of the vulcanised compounds were extracted for one week (say 40 hours) with boiling acetone in a special extractor,<sup>3</sup> and the sulphur remaining in the rubber was estimated. In the case of the litharge compounded samples the specimen after extraction was placed in ether acidified with hydrochloric acid for a few hours to decompose the lead sulphide formed.<sup>4</sup> A second treatment sufficed for the complete removal of the lead. The combined sulphur is given, calculated as a percentage on the raw rubber taken (coefficient of vulcanisation). Rings were also cut from the vulcanised compound and the load required to elongate the specimen to 9 times its original length was determined.<sup>5</sup> The figures are the average of tests on five rings and give the load in terms of grms. per sq. mm. cross-sectional area.

#### (1) Litharge.

% Accelerator.	Coefficient.	Load, grms. per sq. mm.
nil	1.26	237
0.1	1.25	244
0.25	1.27	248
0.5	1.37	282
0.8	1.75	370

#### (2) Magnesia.

% Accelerator.	Coefficient.	Load, grms. per sq. mm.
nil	1.40	152
0.1	2.66	390
0.25	3.31	755
0.4	3.68	1120
0.75	4.08	1320

#### (3) Isonitrosodimethylaniline.

% Accelerator.	Coefficient.	Load, grms. per sq. mm.
nil	1.40	152
0.091	1.90	320
0.182	2.27	430
0.364	2.97	827
0.726	4.24	1675

#### (4) Basic amino derivative.

% Accelerator.	Coefficient.	Load, grms. per sq. mm.
nil	1.40	—
0.45	2.62	—
0.90	3.42	—

There being insufficient of the original raw rubber for the litharge "mixings," which were made after the others, it was necessary to use another sample of crêpe for this purpose. The coefficient and load for this latter sample differed a little from those of the crêpe rubber used for the

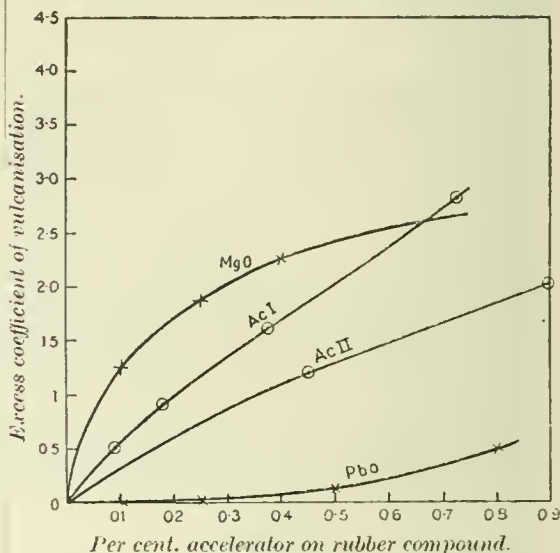


FIG. 1.

other three experiments; thus, for the one hour cure a coefficient of 1.26 was obtained instead of 1.4 and a load of 237 instead of 152. This, however, does not affect the conclusions to be drawn from the experiments. The relationship between

the amount of accelerator used and the coefficient is shown in Fig. 1. The curves start at a zero obtained by subtracting the natural coefficient of the rubber from that found after adding the accelerator. The conclusions may be summarised as follows:—

(1) Of the inorganic accelerators, magnesia is much more powerful than litharge, particularly in small amounts.<sup>6</sup> The form of the curves would, however, lead one to expect that in larger proportions litharge would tend to approach magnesia in accelerating power.<sup>7</sup> The curve for magnesia is convex to the ordinate, that for litharge concave. The action of litharge is frequently complicated by the withdrawal of part of the sulphur which reacts with the lead. This, however, does not affect the present experiments owing to the large proportion of sulphur and small proportion of litharge in the "mixings."

(2) Magnesium oxide is more powerful than the organic accelerator Ac. II. (basic amino derivative) in all proportions up to at least 1% of accelerator on the rubber. It is also more powerful than the organic accelerator Ac. I. (nitrosodimethylaniline) in proportions up to about 0.6%. The graphs for the organic accelerators are almost straight lines in contrast to the strongly curved graph for the magnesia. Therefore the effect of

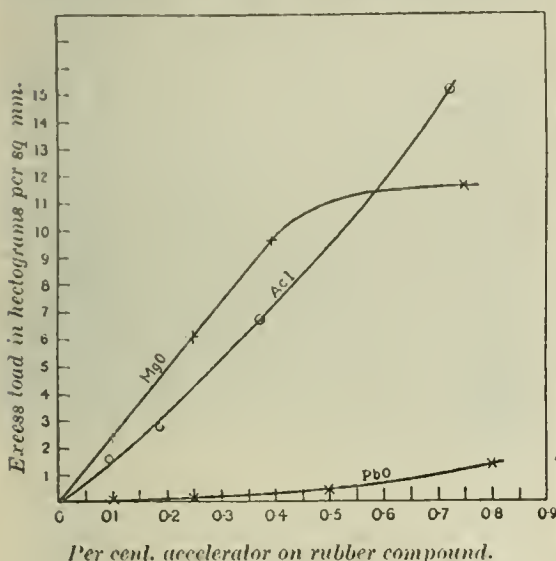


FIG. 2.

increasing the proportion of the organic accelerators is similar in both cases but different from that of the magnesia which is also different from that of the litharge.

Fig 2 gives the loads at a constant elongation after subtracting the load of the control for each. These figures define the position of the stress-strain curve which has been used by some as an index of the degree or state of cure.

It will be seen that the graph for the organic accelerator is practically a straight line, as also is that for magnesia up to a certain point which corresponds to a coefficient of vulcanisation of about 3.7. It therefore appears that, when a magnesia compound is appreciably over-cured, it becomes rapidly harder and less distensible the higher the coefficient. This effect is not noted with the organic accelerator. The graph for the litharge is slightly curved and closely resembles that in Fig. 1. Fig. 3 gives the coefficients plotted against the loads. The graphs for the organic accelerator and litharge compounds are only

slightly curved, consequently we may conclude that the coefficient and load are approximately proportional within the limits of the experiment. The graph for the magnesia compound is appreciably curved and the load increases more rapidly the higher the coefficient. It is therefore evident that, with compounded rubbers, even those containing less than one per cent. of mineral or organic

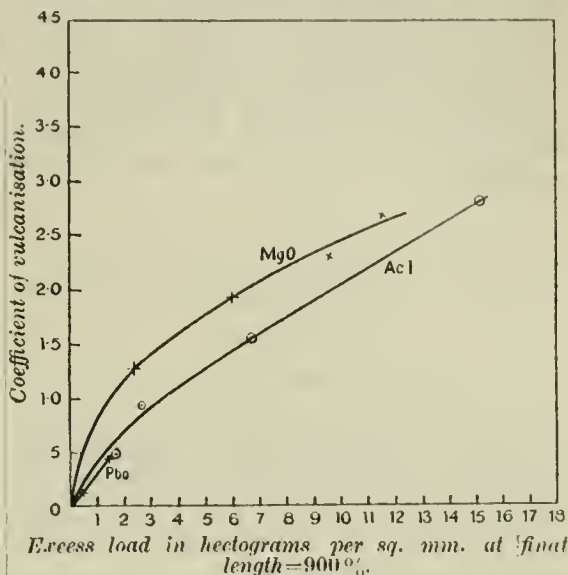


FIG. 3.

accelerators, the load is seldom if ever exactly proportional to the coefficient and consequently the estimation of the rate of cure cannot be safely based on the loads, that is to say, the stress-strain curves, without checking these against the coefficient of vulcanisation. Even small quantities of quite inert ingredients appreciably affect the load corresponding to the same coefficient. Thus, in some earlier experiments<sup>8</sup> it was shown that  $\frac{1}{2}\%$  of zinc oxide in a rubber and sulphur mixing decreased the extension for a given load by about 11%, while  $\frac{1}{2}\%$  of talc increased the extension by about 9%. These changes were accompanied by little or no increase in the amount of combined sulphur. The experiments in this paper are confined to the effect of very small proportions of accelerators such as are used in practice in the case of the organic accelerators. Inorganic accelerators such as litharge and magnesia are often used in larger proportions which cause a considerable modification in the properties of the vulcanised rubber, apart from the accelerating effect.

<sup>1</sup>D. Speace, *J. Ind. and Eng. Chem.*, 1918, **10**, 115.

<sup>2</sup>B. J. Eaton, *this Journal*, 1917, 1225.

<sup>3</sup>The precaution was taken to ignite the magnesium oxide shortly before incorporation with the rubber.

<sup>4</sup>*Analyst*, 1913, **38**, 143.

<sup>5</sup>In accordance with the author's method of estimating sulphur in combination with rubber (combined sulphur) in the presence of metallic sulphides and sulphates. *Analyst*, 1915, **40**, 275.

<sup>6</sup>That is, load when the elongated specimen measures 9 units, the original length of the specimen = one unit.

<sup>7</sup>This disproves Eaton's contention (this *J.*, 1917, 1225) that lead oxide is a more powerful accelerator than magnesia. Consequently Eaton's explanation of the effect of litharge on the stress-strain curves is inadequate. Otherwise magnesia should have obscured the difference between fast and slow vulcanising rubbers more completely than litharge. The explanation I put forward is based on previous work (this *J.*, 1915, 524; 1916, 874), in which I showed that the effect of litharge is to withdraw the sulphur which would otherwise be available for vulcanising the rubber. Litharge therefore acts in a twofold capacity. Firstly, as an accelerator and secondly, by reducing the proportion of sulphur available for vulcanisation, it has a retarding action on the rate of vulcanisation of the compound. The retarding effect produced by a reduction in the percentage of sulphur is apparent from Eaton's own curves (this *J.*, 1916, 1049). Compare for



Instance the graphs 146A and 146C. The latter corresponds to a mixing where the sulphur has been reduced by one-half. The 2 and 5 hour curves for this mixing (146C) are not further apart than the curves for 1 and 1½ hours of the mixing 146A. Consequently, a difference of 3 hours in the mixing 146C produces no greater effect on the curves than a difference of ½ hour in the mixing 146A, that is to say, the rate of vulcanisation as shown by the stress-strain curves on which Eaton relies is reduced to one-twelfth by reducing the percentage of rubber in the mixing by one-half. In manufacturing practice, 5% of sulphur on the rubber is a much more usual proportion than 10% and, if the former had been taken as the basis for comparison of plantation rubbers, the apparent variation in rate of cure would only be approximately one-twelfth of the figures which have been obtained by Eaton and published as representative of plantation rubber.

In the same article a number of other statements are made by Eaton in reply to my criticisms. Some of these statements are sufficiently refuted by previously published work. Others will be dealt with by the publication of experimental evidence at a later date.

<sup>7</sup>These conclusions do not necessarily hold for dry-heat vulcanising.

<sup>8</sup>This Journal, 1911, 1421.

## ON THE QUALITY OF GLYCERIN FROM WHALE OIL.

(A correction.)

BY A. H. SALWAY, D.S.C., PH.D.

In a recent communication (this J., 1918, 123 T) on the above subject an account was given of a method of investigating crude glycerin for the presence of trimethyleneglycol, and an approximate estimation of the amount of the latter was based on a determination of the specific gravity and apparent glycerol content, using the equations,

$$\begin{aligned} \text{sp. gr.} &= 1 + 0.00263x + 0.00053y \dots\dots\dots (1) \\ \text{apparent glycerol content} &= x + 0.81y \dots\dots\dots (2) \end{aligned}$$

where  $x$  and  $y$  are the percentages of true glycerol and trimethyleneglycol respectively.

The attention of the author has now been drawn to the fact that equation (1) is correct only when  $x$  and  $y$  are percentages by volume. In equation (2)  $x$  and  $y$  represent percentages by weight, and therefore the two equations cannot be used in conjunction for the calculation of  $x$  and  $y$ . Equation (1) should be:—

$$\text{Sp. gr.} = \frac{100}{\frac{x}{1.263} + \frac{y}{1.053} + (100 - x - y)} = \frac{100}{100 - 0.2082x - 0.0503y}$$

When this equation is used for the calculation of the trimethyleneglycol content of crude glycerin it is found that the figures previously given require modification to some extent. The general conclusions are not however affected. The table on p. 124 T should read:—

W hale oil.	Source of the glycerin.	Glycerol.	Trimethylene- glycol.
No.		%	%
0.....	Saponification process	85.0	nil
1.....	Autoclave	78.0	nil
2.....	Twitchell "	93.3	0.004
2.....	Autoclave "	80.0	0.01
3.....	Saponification "	77.5	0.01
4.....	Autoclave "	—	0.94

## DEOXIDATION OF COPPER AND COPPER ALLOYS.

BY W. R. HODGKINSON, C.B.E.

In a paper read recently before the American Electro Chemical Society by G. F. Comstock (see this J., 1918, 210 A), reference was made to the use of zinc and silicon as deoxidisers in the production of copper castings. The object of the present note is to call attention to the valuable properties of calcium carbide in this respect (see this J., 1914, 445). A mixture of calcium carbide with borax or salt is a more efficient reducer of cuprous oxide in copper or its alloys than any of the deoxidising agents usually employed, and it has the additional advantage of leaving the brass or bronze free from carbon. In the treatment of cupro-nickel a small amount of carbon may be introduced into the metal, but this will not affect its use for most purposes.

The electrical conductivity of the metals is unaltered by the carbide process unless the carbide is very much contaminated with sulphide, which is seldom the case; even then, however, this may be counteracted by the use of borax in sufficient quantity. A flux which has proved entirely satisfactory consists of one mol. of carbide to two mols. of borax.

## London Section.

Meeting held at Burlington House on Monday,  
May 6th, 1918.

DR. CHARLES A. KEANE IN THE CHAIR.

THE INHIBITORY EFFECTS OF WATER ON  
THE INTERACTION OF ALUMINIUM AND  
(a) THE FATTY ACIDS; (b) PHENOL,  
CRESOL,  $\alpha$ - AND  $\beta$ -NAPHTHOLS; (c)  
METHYL, ETHYL, BUTYL, AMYL, AND  
BENZYL ALCOHOLS.

BY RICHARD SELIGMAN AND PERCY WILLIAMS.

In a paper read before the London Section of the Society of Chemical Industry (Seligman and Williams, this J., 1916, 88) the action of a number of boiling, concentrated, lower fatty acids upon aluminium was described. It was shown that, whereas these acids when concentrated, in the ordinary sense of the word, were extremely inactive with regard to aluminium, yet, if entirely dehydrated and containing less than 0.05% of water, they rapidly attacked the metal.

Reference was also made to some preliminary experiments with a mixture of higher fatty acids, which appeared to show that these behaved like their lower homologues with respect to aluminium.

These experiments have been continued both with mixed acids and with preparations of "pure" palmitic and oleic acids, and there is now no room for doubt that these acids, in the absence of water, attack aluminium most vigorously, whilst minute traces of water suffice to inhibit the action entirely. If heated in air the higher fatty acids only attack aluminium at temperatures in the neighbourhood of 300°C., whereas if heated in a current of a dry neutral gas or *in vacuo*, interaction starts at very much lower temperatures. This is in all probability due to the fact that before attack by an acid heated in the air can commence, conditions must obtain in which the water produced by the oxidation of the acid is eliminated as rapidly as it is formed, and this is the case at high temperatures. As in the case of the lower acids, a current of an indifferent dry gas has been found most efficacious in removing traces of moisture, and so enabling the interaction to start at lower temperatures, but a current of oxygen protects the metal from attack. This is due to the fact that water is produced by contact of oxygen with the higher acids, whereas the lower acids previously experimented with are not oxidised under the conditions prevailing.

With the conclusion of these experiments certain cases which have occurred in practice in which aluminium has been attacked by higher fatty acids have been satisfactorily explained, and the course or courses to be followed in order to obviate their recurrence have been indicated. Experiments have shown that the composition and physical state of the metal are not without bearing upon the rate of attack, but a definite statement on this point would be premature.

The causes which determine the action of fatty acids upon aluminium having been thus elucidated, an analogous explanation was sought in the case of phenol and similar substances. Numerous attempts have been made to use aluminium stills and condensers for refining phenol and cresol, for which purpose the metal seemed to be particularly suited owing to its cleanliness and the fact that no

discoloration of the refined products was to be feared. The attempts have, however, failed owing to the fact that at high temperatures, which seemed to be quite definite for each substance, the phenols attack aluminium very rapidly. Gladstone and Tribe, as well as Cook, have investigated the interaction of various phenolic substances and aluminium. Gladstone and Tribe (J. Chem. Soc., 1881, 39, 9-19, and 1882, 41, 155-156) state that boiling phenol and cresol attack aluminium very slowly, and  $\beta$ -naphthol "is acted on between its melting and boiling points," but that thymol, benzyl alcohol, and  $\alpha$ -naphthol are not acted on at all except in the presence of aluminium iodide. Cook (J. Amer. Chem. Soc., 1906, 28, 608, and Amer. Chem. J., 36, 543) states that "when commercial aluminium and pure phenol are heated sharply for 2 or 3 hours a violent reaction sets up, precisely the same as in the other methods" (i.e., use of aluminium iodide). In the case of *m*-cresol Cook found that the interaction might be started at the boiling point. In both cases he found that the addition of a small amount of the aluminium compound promoted the attack. He also concluded that "the action of aluminium on phenol is electrolytic in character," and to explain the difference between his results and those of Gladstone and Tribe he suggested that the metal used by the latter was much purer than "the modern commercial article" used in his own experiments. He states that "for the reason that a very pure aluminium would not contain sufficient impurities to set up local action it would be without action upon phenols unless a catalytic agent like iodine should be employed." Experiments made with phenol, cresol,  $\beta$ -naphthol, and  $\alpha$ -naphthol have disproved most of these views, as well as that long held that critical temperatures existed for phenol and cresol respectively, below which interaction did not take place. The first experiments conducted with boiling aqueous solutions of phenol showed that such solutions were for all practical purposes without action upon aluminium,\* and that the same inactivity prevailed in the case of solutions of small quantities of water in pure phenol. Thus, for instance, a sample of aluminium boiled for 2 hours in pure phenol, mixed with a small quantity of water under conditions which precluded the escape of the water, remained entirely unchanged. When, however, the phenol was dehydrated violent action immediately set in. Moreover, the action once started continued even if the temperature was lowered to close to 50°C. As in the case of the fatty acids, it was found possible to inhibit the action altogether by the addition of small quantities of water, no attack taking place when the phenol contained as little as 1% of water.

Similar conditions were found to prevail with regard to a mixture of the cresols and to  $\alpha$ - and  $\beta$ -naphthol, all of which, when anhydrous, attack aluminium violently, although  $\alpha$ -naphthol acts with less readiness than the other substances. It was, however, found more difficult to stop the attack by these substances when once started than in the case of the lower fatty acids, possibly because of their higher boiling points and the consequent difficulty of securing intimate admixture of the water added, but probably also because the rate of attack is very rapid, and consequently much aluminium phenoxide is formed, which entails the addition of considerable quantities of water before complete decomposition can be brought about. The dissolved phenoxides, etc.,

\* If left in contact with aluminium for many months in the cold in the presence of air these solutions attack aluminium in much the same way as water does.



thus tend to keep the phenols anhydrous and so allow the interaction to proceed. These properties also explain the fact observed by Cook that the commencement of the interaction is hastened by the presence of the aluminium compounds. It has been found incidentally that the interaction of the phenoxides and water takes place in at least two stages, the first substance formed being soluble in certain dry organic solvents. The addition of further quantities of water brings about complete decomposition into the phenol and aluminium hydroxide, as described by Cook (*loc. cit.*).

The composition of the metal has been found to affect the rate of attack, but in the opposite sense to that suggested by Cook.

The experiments made with phenol and similar monohydroxy compounds were repeated with various dihydroxy compounds, such as quinol and resorcinol, and the trihydroxybenzene, pyrogallol. With none of these could interaction be brought about by any of the methods used. At this point it is desirable to anticipate somewhat, and to say that similar negative results were obtained with polyhydroxy alcohols such as glycol and glycerol. It may be suggested that the failure of these polyhydroxy compounds to attack aluminium is due to their partial decomposition by which sufficient water is formed to inhibit the interaction. Against this view must be cited the fact that Gladstone and Tribe, using iodine as a catalyst, also failed to form the aluminium compounds of glycol and glycerol, and that similar negative results attended the experiments with amalgamated aluminium on glycols recorded by Tischtschenko (J. Russ. Phys. Chem. Soc., 31, 694—770 and 784—872; Jahresber., 1899, 871). During the course of the present experiments all the polyhydroxy compounds referred to above have been subjected to the action of amalgamated aluminium under various conditions of temperature and dehydration, and although occasional indications of possible incipient attack were noted, it is not considered that these were sufficiently definite to refute the view that no interaction between them and aluminium takes place.

In reading the account of Gladstone and Tribe's experiments it was seen that the device of drying a liquid by a current of hydrogen used by the present authors with acetic acid, etc., had, although unknown to them, been applied to the dehydration of glycerol (J. Chem. Soc., 1881, 39, 31). It is interesting to consider that had Gladstone and Tribe applied the same device to the other alcohols they worked with they would have found the use of iodine unnecessary.

During recent years aluminium has been used on a considerable scale for the manufacture of vessels for the acetylation of various alcohols. From time to time cases have occurred in which these vessels have been attacked. It has been impossible to explain this (*cf.* Boake, this J., 1917, 414), especially in view of the fact that other vessels, apparently identical in construction and composition and similarly used, have not suffered. With the knowledge available of the protection afforded by minute quantities of water the action of anhydrous alcohols upon aluminium was investigated.

It has been known for a long time that aluminium which has been amalgamated with mercuric chloride dissolves in alcohol with the formation of aluminium ethoxide. It has been assumed, however, that the alcohols would not attack the unamalgamated metal. [Meyer and Jacobsen (Lehrb. der organ. Chem., 1907, 212) say that "pure aluminium is only very slowly attacked by boiling ethyl alcohol." This obviously does not refer to conditions such as are about to be described.] Thus Gladstone and Tribe (J.

Chem. Soc., 1876, 153) say "ethyl alcohol was found to have no effect on aluminium alone even when boiled with it for some hours." These authors also experimented with propyl, iso-butyl, amyl, cetyl, and allyl alcohols, whilst reference has already been made (*loc. cit.*) to the negative results of their experiments with benzyl alcohol.

The experiments described in this paper have shown conclusively that where the alcohols can be sufficiently dehydrated they attack aluminium, some of them with extreme readiness, and the form which the attack takes leaves little doubt that herein is to be sought the explanation of the cases of corrosion referred to above. Methyl, ethyl, normal butyl, amyl, and benzyl alcohols when sufficiently anhydrous attack the metal at high temperatures. As in the case of the fatty acids, minute quantities of water are sufficient to prevent the attack or to stop it when once started. The conditions governing sufficient dehydration vary with the different alcohols. Thus amyl and butyl alcohols can be dehydrated by simple boiling in a deep tube and allowing a small fraction to distil off. The alcohols so treated rapidly attack aluminium, and indeed this seems to be a more convenient method of preparing the alkyloxides than that requiring the amalgamation of the metal. In the case of ethyl alcohol this method gave no hope of success, but the desired end was attained by distilling a mixture of 99.5% alcohol with benzene, the water passing over in the earlier fractions in the form of a ternary mixture (*cf.* Young, J. Chem. Soc., 1902, 84, 708). In this way ethyl alcohol was so far dehydrated that interaction with aluminium could be brought about, but the method was not convenient. By distilling ethyl alcohol over calcium and taking precautions to avoid the introduction of moist air into the receiver adequate dehydration was accomplished, and the alcohol so prepared readily attacked the metal. Still greater difficulty was experienced in the case of methyl alcohol. It is interesting to note that Gladstone and Tribe (*loc. cit.*) failed to obtain aluminium methoxide by the method which gave them the aluminium compounds of other alcohols, namely by interaction in the presence of aluminium iodide, a fact which they attributed to the "unstable character of the methylate." The present experiments have, however, shown that by the most meticulous care in the exclusion of water direct interaction of aluminium and methyl alcohol may be brought about. The rate of attack is, however, very slow, a fact which may possibly be due to the slight solubility of the methoxide in methyl alcohol.

As in the case of the fatty acids, the composition and state of the aluminium appear to influence to some extent the rate of attack by anhydrous alcohols.

In the paper referred to above, in which attention was first drawn to the interaction between aluminium and anhydrous fatty acids (*loc. cit.*, p. 89) two hypotheses were raised to explain the inhibition exercised by minute traces of water upon the interaction. It was suggested that acids containing small quantities of water were inactive towards aluminium on account of the formation of a continuous gelatinous, and therefore protective, coating of basic salt upon the surface of the metal, whereas in the absence of water the product of the interaction, being crystalline, would be discontinuous and therefore not protective. That all action ceased as soon as a trace of water was added would then be due to the fact that the crystalline salt formed was incapable of existence in the presence of water. As an alternative, the view was advanced that the rate of dissolution of aluminium in fatty acids is normally very high, but that whenever water is present the

metal becomes coated with the well-known film of oxide which protects it.

As a result of the experiments described in the present paper a combination of these two hypotheses seems to be called for. The view now advanced is that the metal is normally protected by the film which covers it, but that this film breaks down when exposed to the attack of the dehydrated substances experimented with. The presence of even the minutest traces of water in excess is sufficient to inhibit the attack owing either to the fact that the salts, phenoxides, or alkyl oxides, as the case may be, are immediately decomposed by water, aluminium hydroxide being formed, which is insoluble, or because the aluminium exposed by the breakdown of the film is immediately re-oxidised by the water. In one case ocular evidence in support of this view was obtained. A strip of aluminium was boiled with some dehydrated Lulyl alcohol, solution taking place at a single point from which a fine stream of hydrogen bubbles was evolved. Water to the amount of 0.04% of the alcohol was introduced by means of a capillary tube. Within a few minutes the stream of bubbles ceased and the point from which they had been evolved was seen to be coated over by a minute film, presumably of aluminium hydroxide. This hypothesis would, however, demand the gradual though probably extremely slow consumption of the water and the ultimate cessation of its inhibitive action. On this point there is at present neither negative nor positive evidence.

The extreme sensitiveness of the compounds formed to water and their resulting dehydrating tendency appear to explain the fact that the interaction increases in violence as the products of the attack accumulate. It has been observed that the number of points at which the metal is attacked is much larger when the proportion of metal to solvent is large, and consequently the metal content of the solution high, than when small pieces of metal are exposed to the attack of large quantities of solvent. Moreover, the attack by these liquids containing large quantities of metal dissolved cannot be stopped by traces of water. No doubt the bulk of the compound must be decomposed with the production of hydroxide before the water becomes operative in this respect.

#### EXPERIMENTAL.

##### Higher fatty acids.

I. A mixture of higher fatty acids was used, the composition of which was palmitic acid 55%, stearic acid 40%, oleic acid 5%. The aluminium was in the form of sheet 0.5 mm. thick, and its composition was Si 0.13, Fe 0.14, Cu 0.00, Al 99.73%. Before the experiment it was cleaned with dilute caustic soda, as described in the previous paper. Two forms of apparatus were used, according to whether it was intended that the experiment should be conducted in the air or in one or other of the gases experimented with. In the first case the apparatus consisted of a narrow, deep boiling tube, with a small side tube close to the open top. A thermometer dipped into the contents of the tube and the whole was suspended inside a wider tube above a small quantity of boiling bromonaphthalene. For temperatures above 280° C. the outer tube was replaced by a sand bath. Where it was desired to pass a dry gas through the system the boiling tube was closed by a cork, through which passed a glass tube closed at its upper end by a cork through which a thermometer passed into the liquid. At its upper end this tube was, like the boiling tube, supplied with a side tube. Both side tubes were connected to U-tubes containing concentrated sulphuric acid, and the upper U-tube was in its

turn connected with the source of the gas experimented with.

The results obtained may best be indicated in tabular form.

TABLE I.

Temperature	Atmosphere			Vacuum (15 mm.)
	Air (open tube)	Dry hydrogen	Dry nitrogen.	
300°-310° C.	Attack with- in 5 min. of introduction of metal			
280° C. ...	Results regular	Rapid attack immediately on intro- ducing metal		
250° C. ...	No attack in 7 hours	Attack with- in 5 min.	Attack with- in 5 min.	
227° C. ...				Residual at- mosphere air, attack commenced within 30 min.
200° C. ...		Attack with- in 1 hour of introduction of metal		
155° C. ...				Residual at- mosphere air, very slight at- tack during 3 hrs. Re- sidual atmo- sphere hy- drogen, at- tack within 30 min.

In the case of the experiments in air, the formation of a considerable amount of water was observed which at temperatures of 300° C. and above rapidly escaped from the tube, whereas at 250° C. the water formed remained in the tube.

II. *Palmitic acid*, bought as "acid palmitic pure," was used in these experiments, which were carried out with the same metal and in the same apparatus as already described. The results are shown in Table II.

TABLE II.

Temperature	Atmosphere	
	Air (open tube)	Dry hydrogen
300°-310° C. ...	Attack within 5 min. of introduction of sample	
270° C. ...	No attack 1½ hours	
250° C. ...	No attack 1½ hours	Attack within 10 min.
200° C. ...		Slow attack within 1 hour

III. *Oleic acid* bought as "acid oleic pale" was used in this series of experiments. The other conditions were the same as before. The results are tabulated below.



TABLE III.

Temperature	Atmosphere	
	Air (open tube)	Dry hydrogen
300°-310° C....	Immediate and rapid attack	
250° C. ... ..	No attack during 7 hours	Rapid attack within 15 min.
200° C. ... ..		Slow attack within 20 min.

*Phenol, Cresol, and Naphthol.*

*Phenol.* The aluminium used was of the same composition and form as in the experiments already described.

A weighed strip of aluminium was heated with a solution of phenol boiling at 113° C. (uncorr.) in a boiling tube covered with a watch glass. After the heating had been maintained for an hour the metal was re-weighed, but no loss of weight could be detected. The strip was replaced in the tube and the heating continued, but without the cover glass. The boiling point of the phenol solution gradually rose until it reached 177° C. (uncorr.), when rapid attack of the metal commenced.

A further quantity of the phenol solution was heated in the uncovered tube without any aluminium until the boiling point reached 180.5° C. (uncorr.). On introducing a strip of aluminium into the boiling liquid immediate solution of the metal commenced.

In order to make certain that the facts recorded above were not primarily due to temperature, two strips of aluminium were heated with phenol in sealed tubes. The tubes contained phenol which had been dehydrated by heating in a current of dry hydrogen, but to one tube a quantity of water equal to about 1% of the phenol used was added. The tubes were immersed in a vapour bath of boiling phenol. Immediate attack was observed in the case of the tube containing the dry phenol, and on removing the tube from the vapour bath solution of the metal continued so long as the phenol remained liquid. No attack of any kind took place in the tube to which water had been added.

*Further experiments with dehydrated phenol.* In addition to boiling in an open tube, phenol was dehydrated by boiling in a current of dry hydrogen. On placing a strip of aluminium in the boiling anhydrous material, the current of dry hydrogen being maintained, attack commenced within a few minutes and rapidly became most energetic. At a temperature of 128° C. attack developed immediately upon the introduction of a strip of the metal but was relatively slow and showed little acceleration during 20 minutes. At 80° C. slight attack commenced within 80 minutes, and on the phenol being allowed to cool continued until solidification occurred.

*Effect of the addition of water to solutions of aluminium in phenol.* The experiments so far described show that phenol containing aluminium in solution attacks the metal rapidly. These solutions remain clear while hot, but on cooling become cloudy at temperatures well above the freezing point of phenol. They become clear again if the temperature be raised. On the addition of a small quantity of water to such solutions a dense white solid separates, which was found to be insoluble even in boiling phenol and also

in boiling toluene, but readily soluble in boiling dry butyl alcohol. On addition of water to the butyl alcohol solution it was decomposed, aluminium hydroxide being precipitated. Hot phenol containing this solid in suspension was found to attack aluminium readily. On the addition of more water the precipitate changed into a gelatinous mass, similar in all respects to aluminium hydroxide. The phenol now no longer attacked aluminium until it had been dehydrated afresh by heating for several hours in a current of dry hydrogen. Quantitative experiments were not made.

Experiments showed that phenol dissolved in a boiling indifferent substance, such as naphthalene, rapidly attacked aluminium in the absence of water.

*Cresol.* A mixture of cresols, bought as "cresylic acid pure," was used. The experiments were the same as with phenol and gave identical results, except that owing to the cresol being liquid the action of the dehydrated material could be followed during cooling to normal temperature. On adding water to a solution of aluminium in cresylic acid a further difference was noted in that the first product of the interaction was soluble in boiling cresylic acid, whereas the corresponding phenol compound was not soluble in phenol.

*Naphthol.* Gladstone and Tribe (*loc. cit.*, J. Chem. Soc., 1882, 41, 5) state that  $\alpha$ - and  $\beta$ -naphthols differ with regard to aluminium in that the latter attacks the metal whereas addition of iodine is necessary in order to bring about action by the  $\alpha$ -compound. Experiments were therefore made with both  $\alpha$ - and  $\beta$ -naphthols, and both were found to attack aluminium in a manner similar to the other hydroxy-compounds examined, but the rate and form of attack varied as between the two compounds.

*$\beta$ -Naphthol.* At the boiling point  $\beta$ -naphthol was found to attack aluminium extremely vigorously, a strip of metal being dissolved in a few minutes. At 185° C. no attack took place during the course of an hour when a little water was present, but on passing a current of dry hydrogen through the heated liquid a strip of the metal subsequently introduced was rapidly attacked. At 125° C. no attack took place during 2 hours, even in a current of dry hydrogen.

Boiling naphthalene containing 10% of  $\beta$ -naphthol attacked aluminium within 10 minutes of its introduction into the liquid.

*$\alpha$ -Naphthol.* The material used was bought as a "Nobel's standard chemical." It melted at 93.7° C., and gave no ash on ignition. A small quantity was freed from water either by boiling in an open tube or by boiling in a current of dry hydrogen. On boiling  $\alpha$ -naphthol so treated it was found to attack aluminium slowly. The attack was intensely local and on many occasions only took place at one point. In some cases where attack had started at a number of points, all but one point suddenly ceased to give off gas, attack then proceeding much more rapidly at the remaining point. At 185° C. attack was very slow, whilst a boiling solution of  $\alpha$ -naphthol in naphthalene did not attack the metal at all during the course of an hour.

In view of the negative results obtained by Gladstone and Tribe it was thought that the attack might be due to the presence of small quantities of  $\beta$ -naphthol. The  $\alpha$ -naphthol was therefore re-crystallised, but no difference in the behaviour of the purified substance was observed.

In order to make certain that the attack observed was due to  $\alpha$ -naphthol, an amount of aluminium sufficient to saturate 35% of the naphthol used was dissolved in the boiling liquid, by which means

it was safe to assume that any  $\beta$ -naphthol present would be neutralised. The liquid so prepared was found to attack aluminium as before.

#### Alcohols.

**Butyl alcohol.** Pure, normal butyl alcohol containing about 1% of water was used. A strip of aluminium was boiled with this alcohol under conditions designed to preclude the escape of the water. During 3 hours no interaction could be detected. The apparatus was then opened so as to allow the aqueous fraction of lower boiling point to escape. Under these conditions attack commenced within 5 minutes. The rate of attack rapidly increased until the heat developed caused the contents of the tube to boil over. On cooling, solid aluminium butoxide separated from solution.

During a further experiment in the closed apparatus it was noticed that after two hours' boiling interaction started at a single point owing, it was assumed, to the condensation of the small aqueous fraction in the cooler parts of the tube or its accidental escape. When a quantity of water equivalent to 0.04% of the alcohol was added, the stream of gas bubbles flowing from the point of attack ceased within 5 minutes and the point itself was seen to be covered by a flocculent pellicle, presumably of aluminium hydroxide. After a further two hours' boiling attack again commenced at a single point, and was again arrested in the same way. On opening the tube and boiling off the aqueous fraction vigorous action started within 3 minutes.

After the interaction had proceeded for some time and a considerable quantity of aluminium had passed into solution, small quantities of water no longer sufficed to arrest the attack, a definite quantity being first required to decompose the butoxide formed. Similarly it was found that butyl alcohol containing the butoxide in solution attacked the metal with greater vigour and rapidly than the fresh alcohol. From this observation it followed, and was confirmed by experiment, that the rate and form of attack were dependent upon the relative volume of the alcohol to the surface of the metal exposed to attack, since the amount of aluminium dissolved in a small volume would be proportionately greater than in a large volume. It was found that butyl alcohol which had been thoroughly dehydrated attacked aluminium when boiling under a reduced pressure equivalent to 150 mm. of mercury.

**Amyl alcohol.** The material used was bought as "amyl alcohol purified for milk analysis." It was found to be similar in all respects to butyl alcohol with respect to its action upon aluminium, but to attack the metal less vigorously. Attack could be arrested by the addition of 0.04% of water.

**Ethyl alcohol.** The alcohol used was bought as "absolute alcohol 99.5%." On boiling this with a strip of aluminium for 3 hours neither evolution of gas from the surface of the metal nor change in weight of the latter was observed.

**Experiments with dehydrated alcohol.** (a) *Dehydration by distillation with benzol* (cf. Young, loc. cit.). 8 c.c. of 99.5% alcohol, 8 c.c. of dry benzol, and a strip of aluminium were boiled together, 8 c.c. of the mixture being distilled off. A further 8 c.c. of benzol was then added and the distillation continued. When the volume of the liquid had been reduced to 4 c.c. it was found that slight attack took place wherever the surface of the metal was scratched.

(b) *Dehydration by distillation over calcium.* The apparatus used consisted of a 100 c.c. conical flask having a cork stopper fitted with a comparatively wide vertical glass tube about twice

the depth of the flask to serve as a spray trap. The top of this still-head communicated by glass tubing with a boiling tube of the form used throughout these experiments. The side tubulure of the boiling tube was connected to a U-tube filled with glass beads and sulphuric acid. 30 c.c. of alcohol and 3 grms. of calcium were placed in the conical flask. When evolution of hydrogen had ceased about 8 c.c. of the alcohol was distilled over on to a strip of aluminium which had been placed in the boiling tube, and the conical flask and tubing then disconnected and the boiling tube rapidly stoppered. The alcohol in the boiling tube was then heated to boiling point. Within a few minutes evolution of gas was observed from one point, other points of attack developing as the experiment was continued.

The alcohol, which, at the end of the experiment, contained a considerable quantity of aluminium in solution, remained clear. On distilling off the liquid over a water-bath a solid residue remained, which proved to be volatile when heated under reduced pressure, and may therefore be assumed to have been aluminium ethoxide.

**Methyl alcohol.** The methyl alcohol used was bought as "methyl alcohol free from acetone." It was dehydrated in the same way and in the same apparatus as used for the dehydration of ethyl alcohol, but repeated attempts to induce attack proved abortive. The addition of metallic calcium to a tube containing aluminium and dehydrated alcohol also failed to cause attack on the aluminium even when heating was maintained for 8 hours. It was thought that the low boiling point of methyl alcohol might possibly account for the absence of attack, and in order to eliminate this condition experiments were made in sealed tubes. These experiments led to positive results, although temperature is not the governing condition. On the other hand, it was found that the interaction in the case of methyl alcohol is so sensitive to traces of water that only in sealed tubes could a sufficient degree of dehydration be accomplished to enable attack to start.

The first experiment made in sealed tubes was so designed that any water in the alcohol or which might be produced or introduced during sealing of the tube should be taken up by a methoxide which was present in excess. Various attempts were made to use aluminium methoxide for this purpose, but as the only known method of preparing this compound depends upon the presence of mercury, and as it was found extremely difficult to ensure the complete subsequent removal of the mercury, calcium methoxide, which can be prepared by direct interaction of calcium and methyl alcohol, was used. 5 c.c. of methyl alcohol (freshly distilled over calcium), 0.5 gm. of calcium, and a weighed strip of aluminium were placed in a glass tube, the open end of which was then drawn out to a fine capillary and connected to a drying tube (the glass tube used for all sealed tube experiments was boiled with hydrochloric acid in order to remove all traces of alkali). When evolution of hydrogen had entirely ceased, the tube was sealed off and placed in a vapour bath of ethyl alcohol. Under these conditions it was assumed that any traces of water present would be absorbed, and in point of fact definite corrosion of the aluminium and a loss of weight of 0.0028 gm. were observed when the heating had been maintained for 8 hours. Practically identical results were obtained at 100° C. using a steam bath, and at 66° C. using a bath of methyl alcohol vapour, thus showing that temperature was not of determinative importance.

It was thought desirable to obtain similar results in the absence of calcium or calcium methoxide, although it is extremely improbable



that these could play any part other than that of dehydrating agents.

A preliminary experiment showed that 99.5% methyl alcohol, when heated at 100° C. for 8 hours in a sealed tube with aluminium, did not attack the metal.

The experiment made with ethyl alcohol distilled over calcium had already been repeated with methyl alcohol and had given negative results, and sealing off the receiver when the methyl alcohol had been distilled into it similarly failed. This last failure was ascribed to the formation of water during sealing, and the following arrangement, which proved effective, was adopted to obviate this difficulty. The receiver having been thoroughly dried, about 5 c.c. of methyl alcohol was distilled over calcium on to a strip of aluminium. The receiver was then immersed in a freezing mixture to reduce the vapour pressure of the alcohol, and a current of dry hydrogen passed through the upper part of the tube, which had already been drawn out to a capillary. Under these conditions the capillary was sealed off. On immersing the tube in a vapour bath of methyl alcohol attack started within 1 hour, and continued, though very slowly, for 8 hours, when only 0.0002 grm. of aluminium was found to have been dissolved. At each point of attack a minute rod of a white solid was formed, which was subsequently found to be soluble in butyl alcohol and to yield aluminium hydroxide in contact with water. On removing the tube from the vapour bath the reaction stopped, but restarted when heating was resumed. At 100° C. no increase in the rate of attack was noticeable.

*Benzyl alcohol.* Benzyl alcohol was found to mark in some respects a transition stage between ethyl alcohol and butyl alcohol in that, whereas it was much more readily dehydrated than the former, it was more difficult to render absolute than the latter. Its action on aluminium was, however, found to be entirely analogous to that of the other alcohols. A complication was introduced by the well-known propensity of this alcohol to undergo autooxidation, whereby sufficient water is formed to inhibit the interaction under investigation.

On boiling a sample of the alcohol in a long, narrow tube in such a way that the fractions of lower boiling point condensed in the upper parts and thence flowed back again, no attack was observed during the course of an hour. On repeating this experiment in a shorter and wider tube from which the aqueous fraction could escape, the alcohol rapidly became anhydrous and in this condition freely attacked the metal. Similarly on passing a current of dry hydrogen through the liquid which was being boiled in the narrow tube, attack developed as with the other alcohols examined. On replacing the current of dry hydrogen by a current of the same gas which had passed over glass beads moistened with water, a precipitate of aluminium hydroxide was gradually formed, and after this had apparently become complete the alcohol no longer attacked the metal.

It was found possible to dehydrate benzyl alcohol in a stream of dry hydrogen and to render it active with regard to aluminium at temperatures of 185° and 128° C.

Experiments in sealed tubes were made with benzyl alcohol. On heating the alcohol in its original form with aluminium in a sealed tube for 2 hours at 220° C. no signs of attack were observed. The same experiment made with a sample of the alcohol dehydrated by being boiled in a current of dry hydrogen also gave negative results. It was assumed that sufficient water had been produced by the oxidation of the alcohol during the sealing of the tube to inhibit the interaction, and the experiment was therefore

repeated with the modification that the tube was exhausted before sealing and sealed under vacuum. On heating the tube to about 200° C. attack commenced and continued after the contents of the tube had reached normal temperature.

#### DISCUSSION.

The CHAIRMAN asked what degree of dehydration the authors had been able to secure in the case of phenol and the other hydroxylic compounds. In his own experience it was only by means of very special precautions that really anhydrous phenol could be obtained, and as one regarded a third substance, such as water in this case, as an essential agent for reactivity rather than as a deterrent, it would be of interest to know whether it was really excluded. He also asked whether the experiments had been carried out in the presence or absence of air, as possible oxidation by air might be a contributory cause to the reactivity with the aluminium.

Mr. T. H. DURRANS asked whether the authors had studied the action of acetic anhydride on aluminium, and also whether they had made a microscopical examination of the aluminium, because the samples exhibited were perfectly bright and there was no evidence to the eye of any protective film being formed.

Dr. A. F. JOSEPH asked whether the sensitivity of the reaction bore any relation to the quantity of water present.

Mr. C. E. BARRS asked whether the standard of purity of the aluminium had been the same throughout the whole of the experiments.

Mr. H. TALBOT said that in endeavouring to explain why aluminium was or was not attacked the authors had put forward the idea of a protecting layer on the surface of the metal, but they had not given a very clear notion as to whether it was aluminium oxide, hydroxide, or other aluminium compound, or a state of the aluminium. Was the layer material, or was it simply a state of the aluminium? If the former, what was its composition; if the latter, how could it be produced, how could it be altered, and how could it be recognised microscopically?

Dr. SELIGMAN, in reply to the Chairman, said that they did not pretend to have decided the actual degree of dehydration of material that had been obtained; the experiments had been based on severely practical requirements. They did not pretend in any way to have obtained dryness in the sense of the word used in Prof. Baker's communications. They only knew that extremely small additional quantities of water were sufficient to stop this reaction. For instance in the case of methyl alcohol, leaving the end of a tube which had been drawn out to a capillary open for a short time allowed enough water to get in to stop the interaction. He did not know the purity of the phenol used, which was bought as pure phenol. In view, however, of the fact that all the materials that had been experimented with attacked aluminium in the same way, was not it unlikely that in every case they should contain the same impurity? Moreover, in the experiments with lower fatty acids prolonged search had been made for impurities responsible for the phenomenon observed, but without any positive result. In connection with their previous paper the Chairman had suggested one or two possible causes for the attack, amongst them being the formation of aldehyde. That had been investigated and they had found that aldehyde played no part in the interaction. It had been impossible to trace anything responsible for the reaction and they had been faced with the fact, which as the Chairman had pointed out was a most surprising one, that far from water facilitating the reaction, as they had supposed would be the

case, the more the water was removed the more rapid was the attack. These experiments had been tried in the presence of air and they had also been made in hydrogen, in nitrogen, and *in vacuo*, and the same results were found in every case. In regard to the effect of acetic anhydride on aluminium, the pure anhydride would probably be without effect, but it was impossible to obtain it unmixed with acetic acid, and a mixture of acetic acid and acetic anhydride gave anhydrous acetic acid, which produced the conditions which were being discussed. In reply to Mr. Durrans, no protective film could be seen on aluminium by the eye, neither could it on any cooking pan, and yet those who had to deal with aluminium were all quite satisfied that it was there. The standard of purity of the metal in these experiments, except where it was varied to see the effect, had been the same, 99.7%. The Chairman had suggested that possibly impurities might account for the trouble, but the purer the metal the more rapidly it was attacked. That was another curious fact which he left to be explained. With regard to Mr. Talbot's question, he had already expressed his view as to the existence of the protective film, but he had no knowledge as to its composition except that it was generally assumed to be a colloidal hydroxide of the metal. As to its visibility he hesitated to say except that in watching molten metal being poured from a crucible or a furnace he had always been struck with the fact that one saw at certain temperatures an apparently perfectly fluid metallic surface, and at certain moments that surface was covered over by a film, but nothing of the film could be seen after the metal had solidified. That he believed was the only actual tangible and ocular evidence of the existence of the film except that aluminium had to be rubbed somewhat vigorously in order to get good electrical contact.

## HOT WIRE ANEMOMETRY: ITS PRINCIPLES AND APPLICATIONS.

BY J. S. G. THOMAS.

When the velocity of a gas current has to be determined the usual method employed consists in the use of a Pitot tube and a sensitive tilting gauge. The Pitot tube and gauge measure the difference between the dynamic and static heads, and very approximately this difference of pressure is proportional to the square of the velocity of the gas current. In the hands of a skilled experimenter the tilting water gauge is capable of yielding accurate results, but is an instrument which cannot be placed with any degree of confidence in the hands of one unskilled in its use. Apart from this objection to the use of the Pitot tube for general works' purposes, there are certain points connected with the use of the Pitot tube which, in the opinion of some experimenters, make it questionable as to how far its indications are reliable in various circumstances. These objections to the use of the Pitot tube have been very succinctly summarised by Professor J. T. Morris.<sup>1</sup>

The system of anemometry with which this paper deals, depends upon the cooling experienced by a heated wire when immersed in a stream of fluid. The theoretical aspects of the problem have been considered by Fourier,<sup>2</sup> Poisson,<sup>3</sup> Boussinesq,<sup>4</sup> and H. A. Wilson.<sup>5</sup> Experimental work on the sub-

ject of the convection of heat from a heated wire has been carried out, amongst others, by Kennelly and Sanborn.<sup>6</sup> Complete references to experiments on the subject have been published by Langmuir.<sup>7</sup> Kennelly<sup>8</sup> appears to have been the first to suggest that a measurement of the current required to keep a wire at a definite temperature when immersed in a current of air might be used as a means of determining the velocity of the air current. Kennelly, Wright, and Van Bylevelt enunciated the laws of cooling of heated wires in a stream of fluid as follows:—(1) The linear convection is proportional to the temperature elevation of the wire at the same wind velocity; (2) under varying wind velocities (200 to 2000 cm. per sec.) the linear convection increases as the square root of the wind velocity.

The subject was developed independently by Morris<sup>9</sup> and by Bordini.<sup>10</sup> An integrating form of anemometer has been described by Gerdien,<sup>11</sup> and the principle of electric heating of a gas has been employed by C. C. Thomas<sup>12</sup> in his electric gas meter. Experiments in hot wire anemometry were carried out by G. A. Shakespear in 1902. An exhaustive study of the subject has been made by L. V. King,<sup>13</sup> who has succeeded in placing hot wire anemometry on an absolute basis, i.e., he has shown how it is possible to calibrate an anemometer wire in terms of its diameter and thermometric constants, independently of the use of any other wind-measuring instrument.

As has been stated above, hot wire anemometry is based on the cooling effect experienced by a heated wire when immersed in a stream of gas at some lower temperature. The laws determining this cooling effect, its dependence upon the temperature to which the wire is heated, its dependence upon the velocity of the gas stream and upon all the other factors involved, amongst others the emissivity of the wire, the thermal conductivity of the gas, etc., have been thoroughly studied. Practical hot wire anemometry, then, simply requires some method of determining the cooling effect of the gas stream upon the heated wire. Various methods are available and have been suggested and employed by different experimenters.<sup>14</sup> In some cases the temperature of the wire is maintained unaltered, extra current being passed through the wire to compensate for the cooling effect of the gas stream. This is the method employed by King.<sup>15</sup> The platinum wire is inserted in one arm of a Kelvin double bridge, the balancing resistance being composed of manganin, which possesses a negligible temperature coefficient. The pairs of ratio arms of the bridge are so adjusted that a balance can be obtained independently of all connecting or contact resistances. The wire is mounted at the ends of a fork, the tension of the wire being adjusted by means of a fine thread. The pairs of ratio arms are separately adjusted to equality, the bridge being thus balanced when the resistance of the anemometer wire is equal to that of the manganin wire. On inserting the anemometer wire in a stream of fluid, the bridge being balanced before insertion, the cooling of the anemometer wire by

<sup>6</sup> Kennelly & Sanborn. Amer. Phil. Soc. Proc., 1914, 55—77.

<sup>7</sup> Langmuir. Phys. Rev., 1912, 34, 401; Proc. Amer. I.E.E., 1912, 1011; 1913.

<sup>8</sup> Kennelly. Trans. A.I.E.E., 1909, 363—397.

<sup>9</sup> Morris. British Assoc., Sept., 1912; also Engineer, Sept. 27, 1912.

<sup>10</sup> Bordini. Nuovo Cimento, April, 1912, 241—283. Electrician, Nov. 22, 1912, 278.

<sup>11</sup> Gerdien. Ber. douts. physik. Ges., 1913, 20.

<sup>12</sup> C. C. Thomas. J. Franklin Inst., 1911, 411—460.

<sup>13</sup> King. Phil. Trans., 1914, A. No. 520, 373—432.

<sup>14</sup> See, e.g., Morris. Engineering, Dec. 27, 1912.

<sup>15</sup> L. V. King. Phil. Mag., 1915, 556. Also Phil. Trans., 1914, A., 373—432. Eng. Pat. 18,563 of 1914.

<sup>1</sup> Morris. "The Electrical Measurement of Wind Velocity." Engineering, December 27, 1912.

<sup>2</sup> Fourier. Mémoires de l'Académie, 1820, 12, 507.

<sup>3</sup> Poisson. "Théorie Mathématique de la Chaleur," 1835.

<sup>4</sup> Boussinesq. Comptes Rend., 133, 257.

<sup>5</sup> Wilson. Proc. Camb. Phil. Soc., 1904, 12, 413.



the stream upsets the balance, and an adjustment of current through the anemometer wire is made by means of a rheostat until the bridge is again balanced. King has shown that the velocity,  $V$ , of the stream is related to the current,  $i$ , necessary to restore the balance by the equation  $i^2 = i_0^2 + K \cdot V$ ; when  $V = 0$ ,  $i = i_0$ , so that  $i_0$  is the current which produces a balance of the bridge in a still atmosphere. Platinum wire of 2.5 or 3 mils in diameter is found most suitable, that used in platinum thermometry being especially suitable. The wires for use as anemometer wires are calibrated by being whirled round at the end of a rotating arm. In this manner King has shown that the relation  $i^2 = i_0^2 + K \cdot V$  holds rigidly over the range from 17 to 800 cm. per sec., and certain results indicate that it holds over a much more extended range. At low velocities free convection currents from the wire may become sensible, altering the resultant direction of the stream past the wire, but this error can be eliminated if the wire is calibrated suitably disposed in the stream. Using a 3 mil wire an accuracy of 1% can be obtained in the determination of a velocity of 9 cm. per sec. = 0.2 mile per hour. An investigation by King has shown that variations of atmospheric conditions have very little effect upon the velocity determinations. With regard to the effect of pressure, this, with ordinary fluctuations is negligible, and in any case, as the hot wire anemometer measures the mass flow of a gas, i.e., the product of the velocity by the density of the gas, when this is required, no correction is necessary. Using a high temperature—say 1000° C.—King has shown that to the first order of small corrections no correction is necessary for the temperature of the gas stream.

J. T. Morris has suggested various methods of use of a hot wire as an anemometer.<sup>16</sup> Perhaps the most suitable for general use is that contained in his Patent Specification No. 25,923 of 1913. King, it will be remembered, proposed the use of one platinum wire with large temperature coefficient of resistance, the balancing wire being of manganin with negligible temperature coefficient. Morris, on the other hand, according to the specification, proposes to make all the four wires of the bridge—a simple Wheatstone bridge is employed—of the same material, one or alternate arms of the bridge being shielded by means of a tube so that, all the resistances being adjusted to equality at any temperature, the balance is not upset with change of temperature provided there is no relative motion of the anemometer and the surrounding medium. For the purpose of calibration in Morris' experiments<sup>17</sup> the hot wire anemometer was inserted, together with a Pitot tube, in a wind channel, and corresponding readings taken. All four wires, shielded and unshielded, are mounted in a frame and inserted *en bloc* in the stream. Using a galvanometer device in which the deflection is proportional to the square of the current, the deflection produced by the "out-of-balance" current in the bridge is very approximately proportional to the velocity of the stream. In this form the instrument becomes robust, the various parts being such as can be employed for general works' purposes. Essentially, what is required is a Wheatstone bridge as described, a cell or cells to serve as source of current, a rheostat for maintaining constancy of current in the bridge and an indication of such constancy of current, which indication may be furnished by the galvanometer employed as the indicator of the velocity of the stream, a suitable switch being arranged for the

purpose. The bridge wires, if being used for the determination of the total-mass flow of air or gas in a main, can be inserted either at the centre of the main or at any other point where the relation of actual velocity to the mean velocity in the main is known. The indicating instrument can then be calibrated directly to indicate the quantity of air or other gas flowing in the main. Used in this form, the reading of a pointer serves to indicate the rate at which gas is flowing in a pipe. The observer has no delicate adjustment to make. The only adjustment to be made is an adjustment of the bridge current to a certain specified value by means of a rheostat. This is an adjustment which can safely be left in the hands of any workman of ordinary intelligence. A skilled observer is not required. In works where rates of flow of various fluids have to be apportioned to one another the readings of one or more hot wire anemometers will enable this to be done in a rather less empirical manner than is sometimes the case at present. Moreover, the hot wire anemometer can easily be made a recording instrument, so that a record can readily be obtained of the performance of various mains so far as the passage of air or other gas through them is concerned. King<sup>18</sup> has described a form of automatic recorder for use with his type of anemometer. The hot wire of the anemometer can be raised to any desired temperature, the readings of the indicating instrument, of course, being a function of the temperature. King in his experiments commonly used a wire at a temperature of 1000° C. For ordinary purposes such elevated temperatures are quite unnecessary. The correction necessary on account of variation of atmospheric temperature is reduced with an increase of temperature of the anemometer wire, but for general purposes, where extreme accuracy is not required, a temperature of 200° C. or so is ample. Moreover, for the determination of low velocities there is an advantage in the use of a wire at moderate rather than at an elevated temperature, in that the free convection current from the wire is thereby reduced. In any case the use of bare wires heated to anything of the order of 1000° C. is precluded in the case of gases, such as coal gas, containing combustible constituents. The possibility of ignition of the gas by the heated wire must be guarded against, and for use with combustible gases a much lower limit of temperature must be employed. Further, the author has found that using a platinum wire of 0.1 mm. diameter and fusing on to it a coating of glass, the sensitiveness of the arrangement is still such as to permit of the use of the coated wire in anemometry; in fact, the sensitiveness is very little, if at all, impaired by this treatment, if not indeed increased.<sup>19</sup> The reason for this apparent anomaly is analogous to that advanced by Porter in the case of the effect of lagging on the radiation from steam pipes.<sup>20</sup> Porter has shown that coating a wire with glass up to about 8 cm. radius increases the radiation loss from the wire as the thickness of the coat increases up to this limit. Generally, Porter has shown that there is a critical radius of lagging equal to  $K/\epsilon$ , where  $K$  is the thermal conductivity of the material of the lagging and  $\epsilon$  its emissivity, such that for values of the radius less than this critical value an increase of thickness of the lagging increases the radiation loss from the heated interior. Although the greater part of the heat loss in the anemometer wire is due to convection, similar considerations hold, and the increase of surface due to the glass coating serves to increase the

<sup>16</sup> Engineering, Dec. 27, 1912.

<sup>17</sup> Morris, *loc. cit.*

<sup>18</sup> Eng. Pat. 18,563 of 1914.

<sup>19</sup> Eng. Pat. 111,015 of 1917.

<sup>20</sup> Phil. Mag., Sept., 1910, 511-522.

effect, the wire being thin and below the limiting radius indicated by theory. This glass-coated anemometer wire is especially serviceable for use with gases easily decomposed by heat or such as are liable to contain soot or carbon from decomposed hydrocarbons. The catalytic activity of glass being in general considerably below that of platinum, such decomposition is prevented by the use of a glass-coated wire. Directions in which the hot wire anemometer can be employed are numerous. Its use where the velocity of a gas stream is required is at once obvious, and has been discussed above. Since the action of the instrument depends upon electric current, this places at our disposal all manner of recording devices actuated by the same power. Any type of recorder employed in platinum thermometry is equally applicable as a recorder with the hot wire anemometer. Relays can be actuated by the "out of balance" current to effect certain operations, thereby rendering many operations automatic. As an example may be mentioned the operation of a valve by the "out of balance" current so that the amount of naphthalene solvent injected into a coal-gas main may be proportioned to the gas flowing in the main. For the complete specification of the flow of gas in a main, as, indeed, for any vector quantity, not only must we know the amount, but also the direction, of the flow. A slight modification of the anemometer enables us to determine whether gas is flowing in one direction or the other in a main. By the use of two bare or glass-coated wires placed across the gas stream, so that one of the wires is shielded from the cooling effect of the stream by the presence of the other wire, the wire on which the stream first impinges is cooled more than the other, the "out-of-balance" current in the bridge displacing the pointer in one direction, which may be marked "up." On reversing the direction of flow of the gas stream, the pointer is displaced in the "down" direction, the shielding effect now being reversed. Owing to the extreme sensitiveness of the hot wire anemometer and its high resolving power, i.e., its power to detect minute variations of gas velocities over small distances, it is a most responsive instrument in the examination of conditions of turbulent flow. King suggests the possibility of using the hot wire anemometer in order to obtain a record of the performance of a steam turbine analogous to the indicator diagram of the ordinary reciprocating engine.

As an example of the employment of the anemometer in the laboratory may be mentioned the result of an investigation suggested to the author by Dr. Charles Carpenter, viz., the determination of the ratio of the volume of gas to the volume of the primary air induced in an inverted incandescent burner, and the variation of this ratio during the period when steady conditions are being established in the burner. For the purposes of experiment a Metro No. 3 inverted incandescent burner was taken. A hot wire anemometer consisting of a piece of platinum wire 0.1 mm. in diameter, glass-coated, and of about 0.6 ohm resistance, was inserted in the supply tube to the burner at some considerable distance from the heated portion of the burner and insulated from the supply tube by a plug of ebonite. The wire formed one arm of a Wheatstone bridge, the ratio arms being adjusted to 1000 : 1. The balancing resistance was 637 ohms, with a maximum variation of  $\pm 1$  ohm during an experiment. The current in the bridge was supplied by a storage battery, and was accurately adjusted throughout by means of a rheostat to 0.8 amp., the magnitude of the current being determined by a sensitive Siemens milli-ammeter

with appropriate shunt. A sensitive dead-beat Paul galvanometer was employed as indicator. The ammeter was calibrated as follows: The gas supply to the burner passed through a gas meter and sensitive balance pressure governor. The bridge was balanced when no gas was being supplied to the burner. The gas supply was then turned on and adjusted to any desired value. The steady out-of-balance deflection of the galvanometer was observed, the bridge current being adjusted to 0.8 amp. A series of observations, galvanometer deflections and hourly rates of consumption over the appropriate range of gas consumption enabled a calibration curve of the anemometer to be drawn. With regard to the determination of the volume of primary air induced by the gas, the following method was employed. The supply of primary air to the burner was drawn in through a brass tube about 6 feet long. Some little distance from the air holes of the burner the tube bifurcated, and the two limbs so formed were brazed to a cylindrical chamber

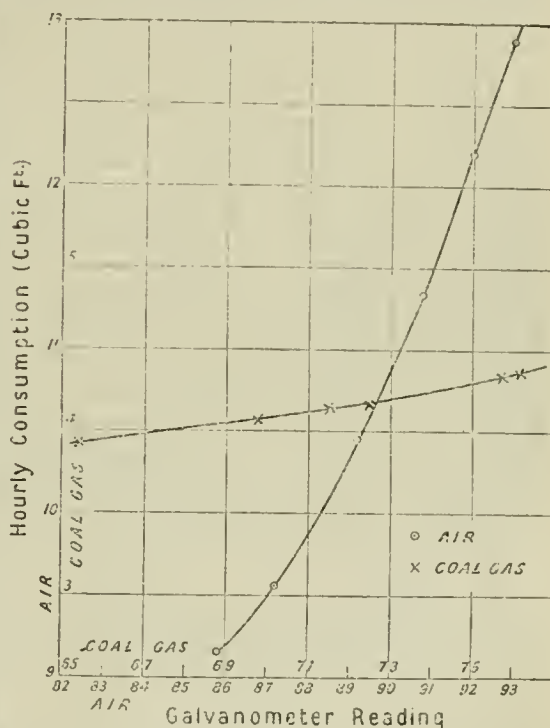


FIG. 1.

surrounding the air holes. In this manner the gas issuing from the injector of the burner received a supply of gas uniformly distributed on all sides. A hot wire anemometer was inserted in the brass tube from which it was insulated by a plug of ebonite. The calibration of the anemometer was performed in the manner described above, the air being derived from the laboratory high-pressure supply and being measured by means of a wet gas meter. All volumes were corrected to 15° C. and 30 in. pressure. The calibration curves are shown in Fig. 1. Both calibrations were made with the burner alight. The marked increase of sensitiveness of an anemometer when used in coal gas as compared with air is obvious from these curves. This increased sensitiveness arises from the very much greater thermal conductivity of the hydrogen contained in the coal gas as compared with that



of air (hydrogen =  $31 \times 10^{-4}$  c.g.s., air =  $5 \times 10^{-4}$  c.g.s.) This factor makes the hot wire anemometer—particularly the glass-coated variety—eminently suitable for use with coal gas or any gas containing hydrogen.

The anemometer having been calibrated as above, the meter connected to the air supply was removed, its place being taken by a closed vessel containing water; the air induced by the burner passed over the water, so that the anemometer was, both in calibration and in use, subject to the cooling action of air saturated with water vapour. The burner was now lit and readings of the respective galvanometer deflections taken at 1-minute intervals, the bridge current being throughout maintained at 0.8 amp. The result is shown in Fig. 2. Certain deductions from these curves are not without interest. In the case of the air and the coal gas it is seen that the consumption diminishes, reaching steady values

directly proportional to the square root of the respective densities. If we assume that the gas obeys Charles' law, we are enabled to estimate the temperature of the gas at the injector, for we have:—

$$\frac{Q_1}{Q_2} = \sqrt{\frac{273 + t_2}{273 + t_1}}$$

$t_1$  and  $t_2$  being the respective temperatures at which the gas consumptions are  $Q_1$  and  $Q_2$ .

In the present case the galvanometer deflection obtained with the coal gas anemometer fell from 75.7 to a steady value 69.8. The respective gas consumptions are 4.36 and 4.05 cub. ft. per hr. The atmospheric temperature being  $57^\circ \text{F.} = 14^\circ \text{C.}$ , we see that the temperature of the gas at the injector is  $60^\circ \text{C.}$  In this manner it is possible to determine the temperature of the gas in its passage through the injector, and a knowledge of this temperature is of no inconsiderable value in connection with burner design. Passing now to the diminution in the primary air induced into the burner, we notice that with the burner in question the final value is not attained by a continuous decrease. There is a preliminary decrease to a lower value than the final steady value, followed by an increase to the final steady value. This effect is explained by the following considerations. Owing principally to the decrease of density of the heated coal gas, and its consequent diminished air-inducing capacity, together with the decreased consumption of gas in the heated condition, the volume of induced air decreases as the burner becomes heated. As indicated at P, the value of the galvanometer deflection throughout was slightly unsteady. This unsteadiness was a characteristic of the decreasing deflection throughout, but cannot be indicated on the remaining part of the diagram owing to the smallness of its amount. The air consumption does not fall steadily, but in a series of oscillations. It will be noticed that the air consumption falls much more rapidly than the gas consumption. The presence of the oscillations and of the final descent and subsequent ascent of the curve at P can be described as an "inertia" effect. The gas and air consumption are being reduced. At any instant of time there is a certain gas and air consumption. The burner is at a certain temperature. At the subsequent instant the gas rate is reduced. The burner, however, does not cool sufficiently rapidly to enable the air rate to attain immediately its value appropriate to the new gas rate. There is a lag in the cooling, and the air consumption is reduced by an amount greater than is appropriate to the new consumption of gas, owing to the fact that the temperature of the burner is higher than its appropriate value. This will account for the dip at P and the series of "oscillations" by which the air consumption attains its final value. From the calibration curve we find that the initial and final consumptions of air, i.e., when the burner is respectively cold and hot are 12.91 and 9.80 cub. ft. per hr. The air to gas ratio is therefore  $12.91 \div 4.36 = 2.96$  when burner is cold, and  $9.80 \div 4.05 = 2.42$  when burner is hot.

The well-known sharpening of the inner cone of an inverted incandescent burner as the burner becomes heated from its cold condition is therefore to be attributed to the direct effect of the pre-heating of the gaseous mixture and not to any increase in the percentage of air in the gaseous mixture, this percentage being, in fact, less in the heated than in the cold condition of the burner.

The author desires to express his gratitude to Dr. Charles Carpenter for his kind provision of every facility for carrying out the above investigation.

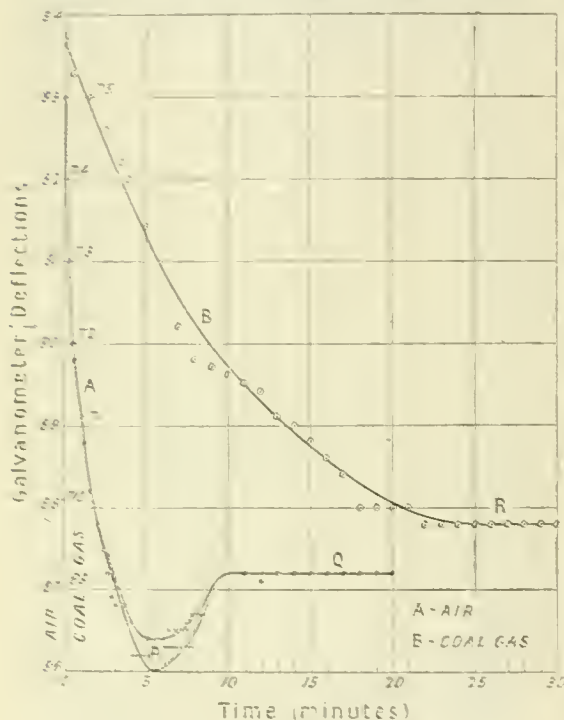


FIG. 2.

represented by Q and R respectively. The reason for the diminution of the gas consumption is not far to seek. Initially the burner is cool, but it becomes heated during the course of the experiment. The pressure at the injector is throughout maintained constant. The suffix 1 referring to the gas in its cool condition and the suffix 2 to the gas in its final heated condition, we have,  $V_1$  and  $V_2$  being the respective velocities of efflux from the injector,  $\rho_1$  and  $\rho_2$  the respective densities—

$$\frac{V_1}{V_2} = \sqrt{\frac{\rho_2}{\rho_1}} \text{ approximately.}$$

Now, the respective volumes consumed per hour measured at same temperature and pressure are in the ratio—

$$\frac{\rho_1 V_1}{\rho_2 V_2} = \sqrt{\frac{\rho_1}{\rho_2}}$$

Hence the respective gas consumptions are

## DISCUSSION.

Captain C. J. GOODWIN said that years ago, before the hot wire anemometer was available, he had had occasion to measure the velocities of gases, and he had used a windmill anemometer calibrated on a rotating arm and also against a Pitot tube. These instruments had to be held more or less at the centre of the pipe that was being used, otherwise an accurate reading was not obtained. The velocity at the centre of a pipe was greater than at its periphery, and although the ratio between these velocities was known with some accuracy, and it was possible to arrive at a mean, these facts complicated the issue in the case of the hot wire anemometer. The wire occupied a good part of the diameter of the pipe, and therefore there was an average cooling effect on the wire, and the reading of the instrument would probably be different in a pipe, say, 20 in. diam. from that in a pipe 6 in. diam. Hence before such an instrument could be used for general purposes it must be calibrated for various diameters of pipes, and the use of a standard diameter pipe for each particular investigation was desirable. It did not appear possible to adapt these anemometers for use in corrosive gases—for instance, nitric oxide. The Pitot tube, on the other hand, could be made of glass. Personally, he had found the Pitot tube reasonably accurate when used in conjunction with an inclined balance level with micrometer adjustment and telescope sight; its use did not require any great skill. The author had said he did not know of any other type of anemometer which would indicate the direction in which the gas was flowing, but both the windmill anemometer and the Pitot tube did that, because if the gas flow was reversed the reading of the instrument would also be reversed. It would seem that many precautions had to be taken with the hot wire anemometer, because in the test on a gas burner the author had found it necessary to reproduce the condition of the air with which the anemometer had been calibrated, *viz.*, to have a certain definite quantity of moisture. If similar precautions were necessary in all cases when testing gas flow in a chemical works, he thought that these, as well as the cost of the instrument, would militate against its adoption.

Mr. J. W. HINCHLEY said that the Pitot tube was a most unsatisfactory instrument to use, even in conjunction with Threlfall's micrometer devices for measuring levels. Mr. Thomas' contribution to the hot wire anemometer, *viz.*, the glass coating of the wire, got rid of many of the objections which Captain Goodwin had raised. The electrical methods mentioned by Mr. Thomas were not necessarily those that would be used in a works, and it would be quite possible, by means of an ammeter and sensitive voltmeter, to measure the change of velocity across the wire by passing a constant current through the wire, and so make the instrument quite accessible for ordinary works' practice. By placing the wire right across the pipe it would be possible to determine the average velocity in the pipe. It seemed to him that the hot wire anemometer offered an advantage over the Pitot tube, although there was the possibility that eddies in the tube would be recorded with the velocity. At the same time, the hot wire anemometer might by suitable means get over the difficulties which were met with in recording the heat exchange between gases, and he hoped the author would continue his researches so that this anemometer could be made of the greatest possible use in the chemical factory by simplifying it in such a way that chemical manufacturers would not be averse to purchasing it.

Mr. G. N. HUNTLY asked as to the range within which the instrument could be calibrated.

Mr. P. J. NEATE commented on the very marked difference in the diagrams between the readings for air and the readings for coal gas, and also on the fact that the readings for moist air and the readings for dry air were appreciably different. Did not that go very much to prove that it was necessary to calibrate the instrument with the gas or air that was being measured, and that a very small change in the condition might make a very considerable difference in the reading?

Dr. H. C. GREENWOOD asked whether Mr. Thomas had extended the range of his measurements to velocities over the "critical velocity" of the gas and whether in ordinary practice he had confined his measurements to stream line motion. It was well known that at the "critical velocity," *i.e.*, the velocity at which the motion changed fairly abruptly from stream line to eddying or turbulent motion, there was a very rapid increase in the heat interchange between a gas and a metal surface. From the same point of view it would be of interest to know how much trouble was caused by the proximity of the measuring instrument to bends in the pipe. Had the author deduced any definite general law connecting the linear velocity of the gas with the heat loss from the wire, and did it follow the usual law of being proportional to about the 0.7th power of the linear velocity? Did the difference between coal gas and air which had been pointed out hold also when high velocities were used; in other words, when the motion was very definitely turbulent? In a series of experiments carried out by Messrs. Acland, Nobbs, and himself on the heat interchange in high-pressure gases, using pressures up to one ton per square inch, it was noticed that there had been practically no difference between the heat interchange resulting with gases of very different thermal conductivities. With nitrogen and hydrogen, for example, almost identical values were obtained for similar conditions as regards velocity. Another rather interesting phenomenon noticed in the same experiments was that the heat lost from a given surface under similar conditions was, over a wide range, practically independent of the pressure; the heat loss could be expressed in terms of the quantity of gas flowing through the tube per unit time without reference to the pressure.

Mr. THOMAS said that the cost of the hot wire anemometer was not such a large amount as might be anticipated from the description which he had given of it. Some little while ago his company had entered into arrangements with a scientific instrument company with regard to the manufacture of these instruments in conjunction with Prof. J. T. Morris, and the estimated cost of the hot wire anemometer after the style of that devised by Prof. Morris had really been surprisingly small. Prof. King had, moreover, suggested methods of use in which the necessary apparatus was reduced to a minimum—an ammeter, rheostat, storage cell, resistance box, and the heated wire. The windmill anemometer he had had to abandon, as in the case of a number of experiments, on calibrating such an instrument against actual velocities, he had found a difference of 50% between the indicated and the actual velocities, and that even after applying the calibration corrections supplied with the windmill. The Pitot tube in the hands of an experienced experimenter was, of course, an instrument of precision, but as used in different works and on different occasions it was questionable as to what the indications really meant. Personally he had had to abandon the Pitot tube because of the difficulties he had encountered, and Mr. Hinchley's experience seemed to have been the same.



As regarded the use of the anemometer in connection with corrosive gases, there was no reason why the whole anemometer should not be coated with glass, or with any coating material which would be suitable for the particular gas in question. He agreed that the direction of the flow of gases could be determined by the Pitot tube, but it did not give a very clear indication, *i.e.*, one visible in all cases without some adjustment being made. What he should perhaps have said was that the double hot wire anemometer was the only method which gave a clear indication that could be noticed just by a pointer. As to the limits of use referred to by Mr. Huntly, the limits as given by Prof. Morris were an inferior one of well under 1 mile per hour and a superior one of at least 40 miles per hour. Using the instrument under the conditions of calibration, there was no reason why these limits could not be materially extended. As to the use of the wire at the centre and at different points in the tube, if the motion of the gas in the tube was stream line motion, then theory indicated that at a certain point in the tube the velocity of the gas was the mean velocity in the tube, and if the anemometer was used at that point, calibration of the anemometer in a 2-inch pipe would make it suitable for use in a 6-inch or any other size of pipe in the absence of turbulence. The difficulty in connection with the use of the instrument with turbulent motion was not insuperable. Prof. King calibrated his instruments by rotating them at the end of a long arm. Prof. Morris on the other hand used a wind channel, whereas his own calibrations had been carried out by the use of gas meters. By rotating the instrument at the end of a long arm there was a tremendous amount of turbulence, and Prof. King was able to obtain the turbulent correction by putting up another wire alongside, not connected with the arm, then rotating the arm and noting the indication given by the fixed wire when the arm was rotated. In that way he was able to measure the correction due to turbulence caused by the arm. That was what he called the "swirl correction," and he had shown that it followed a definite law when the instrument was used in this manner. When used for the measurement of turbulent flow, the velocity measure was what Prof. King called the effective velocity, and experiments by Professors King and Gray had shown that in cases of turbulent flow it was possible with the use of the hot wire anemometer to obtain consistent measurements where other methods were inadequate. The heat loss referred to by Dr. Greenwood was given by an expression similar to the relation between the heat loss from the wire and the velocity of the stream. Users of the electric gas meter designed by C. C. Thomas had found that ordinary variations in the constituents of the coal gas did not materially affect the indications of the instrument. The hot wire anemometer, being likewise based on electric heating, should also yield fairly accurate values for the velocity of a gas stream for ordinary variations of the various constituents. The cooling of the wire was principally effected by convection and this was not materially affected by moderate variations of the constituents. Radiation and conduction in general played subsidiary parts in the cooling of the wire, their influence being more pronounced at low stream velocities. The water vapour in the air supply to the burner in the experiment recorded was introduced so that the anemometer was used exactly as it was calibrated. The difference in the reading caused by the absence of this precaution was less than 1% and of course for general works' use such a precaution would be quite unnecessary.

## THE ABSORPTION OF ATMOSPHERIC GASES BY WATER.

BY J. H. COSTE.

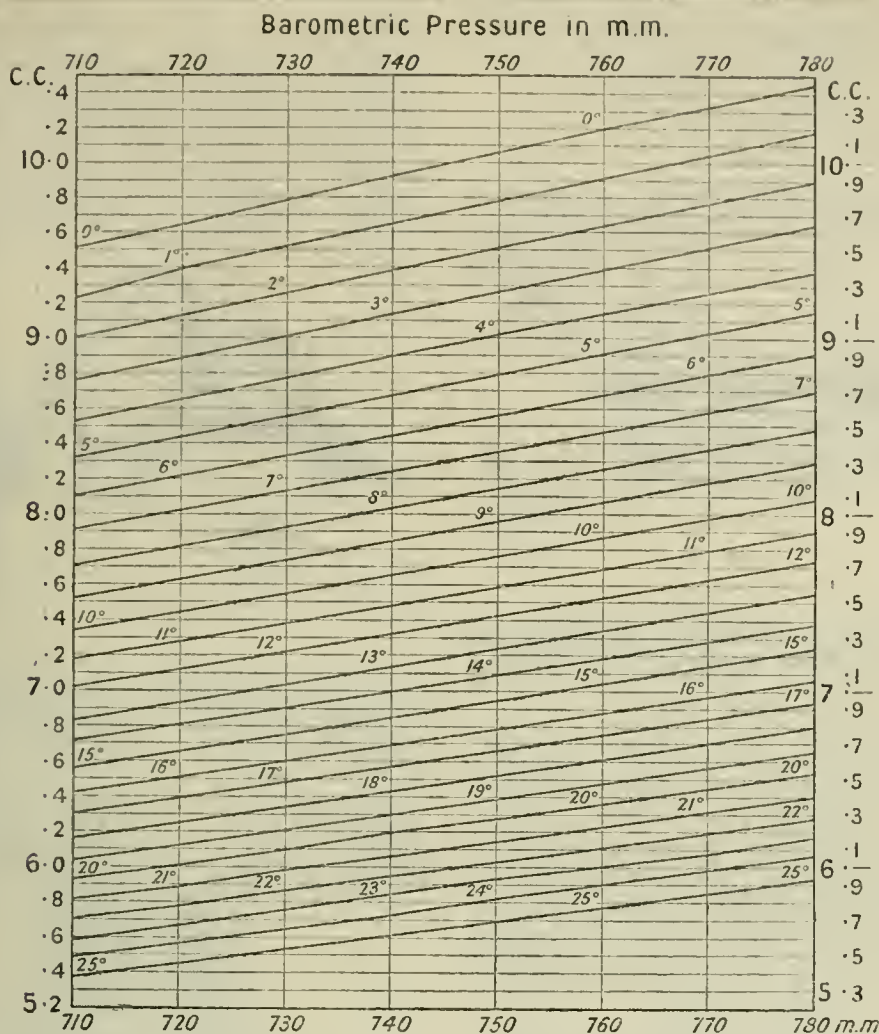
### II.—A DIAGRAM SHOWING THE VOLUME OF OXYGEN DISSOLVED BY WATER AT DIFFERENT TEMPERATURES AND PRESSURES, AND ADDITIONS TO THE BIBLIOGRAPHY.

In calculating the percentage saturation of water with dissolved oxygen it is usual to correct for temperature only, assuming the effect of variations of barometric pressure to be negligible, whereas they may actually affect the results by more than 2% at sea level and by greater amounts in elevated districts. To facilitate correction for both temperature and pressure I have constructed the accompanying diagram, calculated from Winkler's values (19), for *observed* barometric pressures for each 10 mm. from 710 to 780 mm. and for temperatures from 0° to 25° C. To find the volume of oxygen dissolved by a litre of water at any desired temperature and pressure, observe the point where the slanting line marked with the temperature cuts the vertical line indicating barometric pressure. The figure at the side on a level with this point indicates the volume desired, *e.g.*, pressure 730 mm., temperature 13°, volume 7.04 c.c.; pressure 776 mm., temperature 20.5°, volume 6.43 c.c. (in this case visual interpolation is easy).

A few papers may usefully be added to the list given in my previous paper (this J., 1917, 846—853). W. Black and E. B. Phelps (77) in 1911 pointed out the applicability of Fick's law of hydro-diffusion to the absorption of oxygen by still water, and showed that Adeney's "streaming" effect observed in sea water (37) was really due to the formation of a surface layer of more concentrated saline solution by blowing dry air through it. They say, "The property which was washed from the air by passing through the first tube was its dryness." The paper, which is long and very interesting,\* was brought to my notice by Mr. E. Hannaford Richards, whose work on rain-water (78) has brought out the curious fact that rain-water is frequently not saturated with oxygen as at first sight might be expected. The low partial pressure of oxygen at great heights furnishes some explanation of this deficiency. C. J. J. Fox (79) redetermined by an absorptiometric method, and apparently with considerable accuracy, the solubility of oxygen and nitrogen in fresh and sea water and of carbon dioxide in sea water. He discusses the effect of the ocean in maintaining the balance of carbon dioxide in the air, a subject to which Th. Schloesing (80) called attention in 1880. T. Carlson (81) has also redetermined the solubility of atmospheric oxygen in water. His figures and Fox's are close to one another and close to but rather higher than Winkler's. A very good discussion of super-saturation of liquids with gases is given in Ostwald's "Outlines of General Chemistry" (82), in which it is shown that super-saturation must occur if pressure is reduced and gas nuclei are absent. The excess pressure of a spherical bubble appears to inhibit its formation, in theory at least. In practice the "flatness" of a sparkling liquid poured into an already wetted glass is well known, as also is the effect on such a liquid of roughened surfaces.

Vernon (83) has shown that fats dissolve appreciable quantities of oxygen. I called attention in a letter (84) appearing in this Journal to some

\* I owe a copy of this paper to the kindness of Prof. E. B. Phelps.



old work on the inefficiency of layers of oil in isolating water from the atmosphere.

Rideal and Burgess (85) called attention to some aspects of the "dissolved oxygen taken up" test of the Royal Commission on Sewage Disposal.

Many analytical papers on Winkler's manganese process and on the determination of carbon dioxide have been published.

Additions to Bibliography (the numbers follow those on p. 853, Vol. 36):—

(77) Contributions from the Sanitary Research Laboratory and Sewage Experiment Station, Vol. VII. The Discharge of Sewage into New York Harbour. W. M. Black and E. B. Phelps. Massachusetts Institute of Technology, Boston, Mass., 1911. Also The Absorption of Oxygen by De-aerated Water. E. B. Phelps. Trans. Am. Soc. Civ. Eng., 1913, 76, 1624.

(78) E. Hannaford Richards. Dissolved Oxygen in Rain-water. J. Agric. Sci., 8, 3, 331.

(79) C. J. J. Fox. On the Coefficients of Absorption of Nitrogen and Oxygen in Distilled Water and Sea Water and of Atmospheric Carbonic Acid in Sea Water. Trans. Faraday Soc., 1909, 5, 68. Internat. Hydrograph. Comm., Publication de Circonstance No. 41 (1907).

(80) Th. Schloesing. Sur la Constance de la Proportion d'Acide Carbonique dans l'Air. Comp. Rend., 1880, 90, 1410.

(81) Solubility of Atmospheric Oxygen in Water. T. Carlson. Z. angew. Chem., 1913, 26, 713.

(82) W. Ostwald. Outlines of General Chemistry, 1912, p. 317.

(83) — Vernon. Solubility of Air in Fats. Proc. Roy. Soc., 79 B., 366—71.

(84) Coste. This J., 1917, 954.

(85) S. Rideal and W. T. Burgess. The New Standards for Sewage Effluents. Analyst, 1909, 34, 193. Also The Tests for Effluents of the Royal Sewage Commission, Proc. Seventh Int. Cong. Appl. Chem.

(86) L. W. Winkler. Ueber die Bestimmung des im Wasser gelösten Sauerstoffs.

(87) L. W. Winkler. Beiträge zur Wasseranalyse. II. Z. angew. Chem., 1916, 29, 44.

(88) G. Bruhns. Estimation of Dissolved Oxygen by Winkler's Method. Chem.-Zeit., 1916, 955, 1011.

(89) H. Noll. Estimation of Dissolved Oxygen in the presence of Nitrites and Organic Substances. Z. angew. Chem., 1917, 30, i., 105.



## SOME COTTONSEED PRODUCTS IN RELATION TO PRESENT DAY NEEDS.

BY E. C. DE SEGUNDO.

(This J., 1918, 118-123 T.)

### ERRATA.

Page 122 T, col. 2, lines 1 and 2 from bottom, for "crude protein" read "protein and fat."

Page 123 T, col. 1, lines 10 and 11, the figures "18 or 19%" refer to the oil content of average American Upland seed, and the figures "30 to 35%" to the percentage of oil in the meats.

## Manchester Section.

Meeting held at the Grand Hotel on Thursday,  
April 11th, 1918.

MR. WM. THOMSON IN THE CHAIR.

## EVAPORATION IN THE CHEMICAL INDUSTRY WITH PARTICULAR REFERENCE TO THE KESTNER EVAPORATOR.

BY J. ARTHUR REAVELL.

The earliest type of evaporator is that which is direct fired. Even to-day large numbers of evaporators are merely cast iron pots, or steel vessels, which are heated directly by means of coal or coke furnaces, and in some instances are fired by gas. This type is open to many objections, in fact the laws of heat transmission show that such a type does not fulfil a single one of the conditions favourable to efficiency or economy. An instance of this is the common salt pan 60 ft. by 20 ft., with its costly flues and brickwork, requiring gangs of riveters and bricklayers for repairs, etc., to say nothing of the huge area covered relative to its capacity.

The next step was to construct evaporators consisting of a jacketed vessel, in which steam is the heating medium supplied to the jacket. This type retains most of the defects of its forerunner, and may in fact be thermally less efficient.

A later type consists of a simple tank or open pan fitted with a steam coil, for use with high-pressure steam. This makes no advance, beyond the fact that it is easy to construct. With all the foregoing types the rate of heat transmission is low, chiefly because there is very feeble circulation—even with weak liquors—falling to nothing as concentration increases. Further, there are large heat losses by radiation, often accompanied by loss of liquor by entrainment.

### Vacuum evaporation.

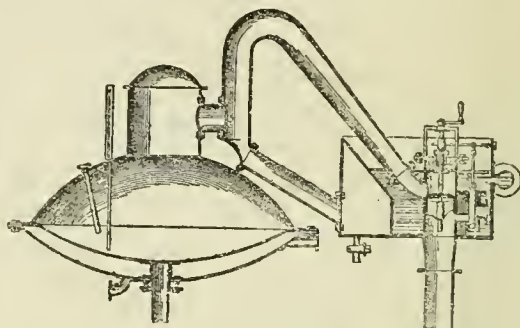
A further development, and one of very considerable importance, was the introduction of the vacuum pan. For the first time advantage was taken of boiling under reduced pressure, and consequently at reduced temperature (see Fig. 1).

It is of interest to note that the original inventor of the vacuum pan was Howard, an Englishman.

The use of reduced pressure led, of course, to the utilisation of exhaust steam, which, previously wasted, now became of value, and gave considerable improvement in economy. The study of evaporation under artificially reduced pressure led to the development of multiple-effect evaporation.

Attempts were made to set up some form of

agitation, with a view to obtaining some motion of the liquor relative to the heating surface. This gave rise to many mechanical devices, which not only agitated the liquor, but the agitators themselves being heated, something approaching a mechanical film was created in this way. Types of evaporator were evolved, such as the Bauer or Wetzel, in which during half a revolution the liquor is in the form of a thin coating on the heating surface, yet this film has little motion relative to the heating surface. Such evaporators

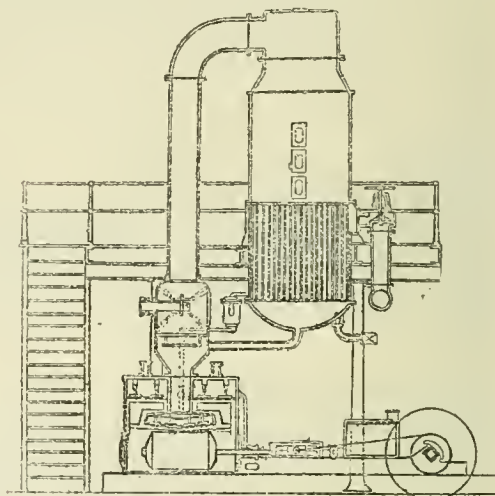


VACUUM PAN.

FIG. 1.

never came into extensive use and the vacuum pan with improvements in detail held the field.

In spite of its many advantages over the more elementary types, the vacuum pan has always suffered from grave disadvantages in practice. Attempts, most of them unsuccessful, though showing a vast amount of ingenuity, have been continuously made to evolve a type of evaporator which would retain the improvements manifest in the pan, but which would be free from the drawbacks which are inherent in this type.



MODERN VACUUM EVAPORATOR.

FIG. 2.

To obtain even moderate circulation and consequent heat transfer, a calandria type is almost essential, and having short tubes the number is great, consequently troubles with faulty expansion and leakage are multiplied. Moreover, large diameters lead to structural difficulties which prevent the use of high pressure or even of what are to-day considered medium pressures. Further, the liquor to be concentrated is present in large bulk, and as a result of this, and of feeble circu-

lation, the ebullition of steam frequently occurs irregularly in the lower portions of the mass, resulting in sudden entrainment or "blowing over."

Secondly, since the rate of evaporation is not constant, but is rapid in the first stages and falls off as concentration proceeds, there must be a compromise when designing the dome and vapour outlet. As a result, if at first the speed of the vapour is too great, continuous entrainment takes place in the form of drops and vesicles.

Again, by evaporating in bulk, every particle of solid in solution is subjected to prolonged heating or "stewing." For this reason expensive vacuum plant is often attached to the pan, simply to keep down the temperature.

There is no economy in the use of a vacuum unless by this means exhaust steam, otherwise wasted, can be made available for heating purposes. As will be shown later, it is better to use, in many cases, an evaporator designed on more correct principles, to employ boiler steam, and to work in single or multiple effect under pressure.

Added to the foregoing, lack of circulation brings in its train quick incrustation.

#### *The film evaporator.*

As the laws of heat were investigated, especially those of heat transfer between liquids and solids, the importance of the movement of the liquid itself relative to the heating surface was more clearly realised.

In 1830, Dubrunfaut set down his idea of an ideal evaporator in as far as sugar liquors were concerned as follows:—"Concentrate the juice as rapidly as possible so as to avoid any alteration; give the liquor to be evaporated great speed,

tilted to one end, so that all the condensed water is drained. The disadvantage of this design is that the film is in no way perfect, because the liquor is formed in large drops on the under surfaces of the tubes, and thus part of the surface is not as effective as the remainder, whilst the same defect occurs, inside the tubes, in the case of the condensed water, greatly reducing the transmission efficiency of this portion of the surface.

The Yaryan (Fig. 4) is also a horizontal type evaporator, but differs from the Lillie in that the liquor to be evaporated is inside the tubes, and

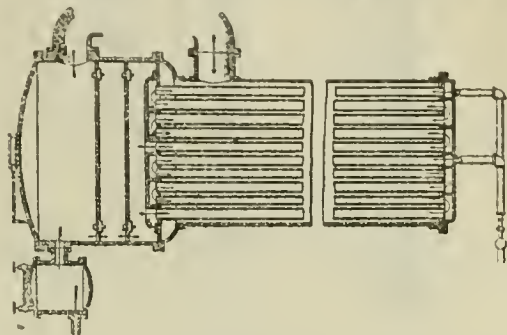


FIG. 4.

the heating steam outside. This has a great many advantages, but the difficulty of feeding a large number of tubes in a horizontal position so that all equally and regularly obtain the same amount of liquor, is obviously great, while the water condensing on the outer surface of the tubes drops from one tube to another, so that the tubes of the lower range are waterlogged and not as effective as those of the upper range.

The Schwager, Claassen, and Greiner evaporators are more or less variations of the same principle, and are not of special interest.

*Kestner evaporator.*—Mr. Paul Kestner, in the course of a large experience in all types of evaporators, had particularly studied the horizontal film evaporator, and found out its great limitations. He therefore set himself to discover, if possible, a means of producing a perfect film, and the principle upon which he founded his design is shown in Fig. 5 herewith.

The tubes are 23 ft. long. The liquor is fed into the tubes from a tank about 3 ft. above the bottom tube-plate, and assuming that this liquor is hot, then on steam being applied to the outer surface of the tubes, ebullition at once commences inside, releasing a large volume of steam which must find its way to a region of lower pressure by passing up the tubes. As a result we have the phenomenon of the climbing film. The liquor and vapour on leaving the top of the tubes meet a fixed centrifugal baffle, where the liquor is separated from the vapour, the liquor passing to the concentrated liquor tank, and the vapour to the atmosphere or to the condenser. The velocity of the liquor itself is thus used by means of centrifugal action to separate the vapour from the liquid.

#### *Heat transfer under actual working conditions.*

In endeavouring to evaporate a liquid, one of the great objects is to transfer the maximum number of heat units per unit area from the heating medium to the liquor to be evaporated.

Based on deductions from actual apparatus, and bearing in mind the results of theoretical and experimental research, the quantity of liquid evaporated in a tubular apparatus heated by steam would appear to vary with the following

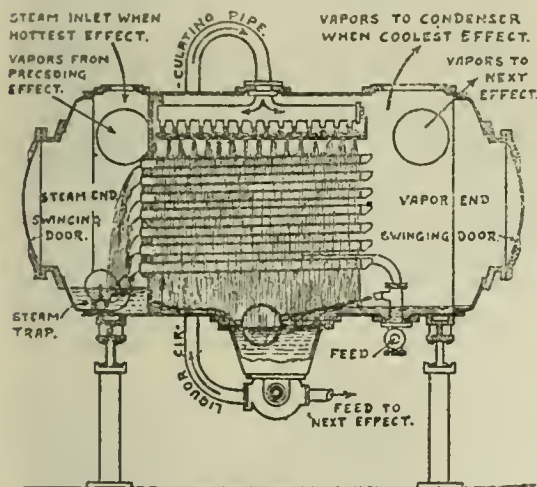


FIG. 3.

and, if possible, accelerate the movement of the liquor so as to evaporate in the form of a thin film." What this authority conceived in 1830 as a theoretical ideal, Paul Kestner has to-day accomplished as an actual fact. The "film idea" has been embodied in different types of apparatus, including the following:—Lillie, Yaryan, Schwager, Claassen, Greiner, Kestner.

The Lillie pan, arranged with horizontal tubes, is shown in section in Fig. 3. The feature of this evaporator is that the liquor is delivered by means of a pump to the top of the nest of horizontal tubes, and is distributed over the outer surface, falling alternately in film and drops into the base of the evaporator again. The heating steam is carried through the tubes, which are slightly



factors:—(1) Area of heating surface. (2) Temperature drop or "temperature head." (3)

Velocity of the liquid relative to the heating surface. (4) Velocity of the heating steam. (5) Specific volume of the heating steam. (6) Viscosity of the liquid.

In practice, of course, the transfer is very far from being the heat conductivity obtained through the ideal cubic centimetre of pure metal. The conditions under which heat is supplied in actual practice to the metal are very different from those in laboratory experiments, whilst the transfer takes place through a combined wall of water, oxide, metal, and incrustation.

The table of conductivity shows the relative values of the different metals.

The next factor is heat drop. Consider a coil pan; the transmission of the heat depends—other things being equal—upon the heat drop, or temperature difference, that is to say, if the steam in the heating coil is at 10 lb. pressure, the temperature of the steam is 115.5° C. Assuming water in the vessel, the boiling point of the water is 100° C., then there is a drop, or temperature head, of 15.5° C.

When vacuum is applied, the object is to increase this temperature drop, and, taking the same figure as above, and with a closed instead of an open pan, by the application of 24 in. vacuum, the temperature of the steam evolved from the liquor would be 60° C. instead of 100° C., so that the temperature drop is increased from 15.5° to 55.5° C.

TABLE I.  
RELATIVE HEAT CONDUCTIVITY.

Grm. Cals. per cm. <sup>2</sup> , per cm. thick, per 1° C. drop per second		Comparative figures, Copper=1000
Copper ... ..	1.041	1000.0
Iron ... ..	0.167	160.0
CaCO <sub>3</sub> ... ..	0.005	5.0
Silica ... ..	0.0035	2.5
Water (not in motion) ...	0.0014	1.4
Air ... ..	0.000056	0.055

Kg. Calories per m. <sup>2</sup> , thickness 1 mm., per hour, 1° C. drop		
Copper ... ..	874,000	
Iron ... ..	60,000	
CaCO <sub>3</sub> ... ..	1,570	
Silica ... ..	935	
Water ... ..	520	
Air ... ..	21	

Theor. kgs. steam condensed per m. <sup>2</sup> , thickness 1 mm., per hour, 1° C.		If the thickness is increased to 2 mm.
Copper ... ..	700.0	350.0
Iron ... ..	112.0	56.0
CaCO <sub>3</sub> ... ..	9.5	1.75
Silica ... ..	1.75	0.87
Water ... ..	0.98	0.49
Air ... ..	0.040	0.020

Now, as the rate of evaporation varies directly as the difference in temperature, a material advantage is gained by this increased temperature head. However, while the rate of evaporation is proportional to the difference in temperature, there may be other factors which militate against this. That other factors do occur is evident, because by increasing the steam pressure the evaporation does not always increase proportionately.

It should be borne in mind, also, that temperature drop has not the same value in any part of the scale. It is of more value higher up in the scale; for one reason the steam is denser, and, therefore, more heat units come in contact with a given unit of area in a unit of time; but in the case of a pan or jacketed tank the designer cannot take full advantage of operating in the higher ranges or with largely increased drops, because constructional difficulties arise in the case of large flat surfaces and to a lesser degree with large diameters, and also because the heating surface is not swept free of the steam bubbles generated.

In a "film" type evaporator high temperatures can be used with advantage, because the surface is so disposed that the diameter is very small, even for great surface areas, whereas, in a bulk evaporator, such as a vacuum pan, a large surface area of tubes means large diameter. Moreover, if high temperature and, consequently, high pressure is employed in this type of pan, many liquors which are sensitive to heat are decomposed, because they are too long in contact with the heating surface. On the other hand, in the "film" type evaporator, which works continuously, the liquor is in contact with the heat for a few seconds only and no "stewing action" takes place.

Heat transfer also depends upon (a) the velocity of the liquor and vapour in relation to the heating surface, and (b) the velocity of the steam in relation to the heating surface.

Speaking broadly, it may be said that the heat transmission varies directly as the square root of the velocity, so that if either velocity is quadrupled, the coefficient of heat transmission is doubled. This is commonly spoken of as the effect of "circulation."

In a tank with heating coils or jacketed tank, there is a large body of liquid which is a considerable distance away from the source of heat, and unless mechanical circulation is adopted, the rate of transmission of the heat to the various parts of the liquor is very low.

In an ordinary type calandria pan the circulation is not as positive nor as certain as in a continuously operating pan. Further, with a large number of short tubes the cross-sectional area of the tubes is large in relation to the heating surface, consequently when movement does take place the velocity is low.

In a vertical climbing film evaporator, the number of tubes is small, the length is great, and there is positive circulation at high velocity, 30 to 60 metres per second; hence the heat transmission is very high, and the time contact exceedingly short—momentary in fact.

Then, again, in a pan the area of the space occupied by the heating steam is large, and the velocity is consequently low; as a result, there is difficulty in evacuating the air and non-condensable gases, whilst the condensed water is not swept from the heating surface.

In a Kestner "film" evaporator the area of the steam space is small, the speed of the steam is high, and the gases are collected definitely in a small space, so that they can be easily removed. It is also found—and this is of the greatest impor-

tance—in regard to high velocity, that the speed of the liquor tends to reduce scaling to a minimum, and has a scouring effect, *e.g.*, a Kestner evaporator remains clean throughout a sugar campaign of many weeks, whereas a pan is cleaned every Sunday. High velocity is also very important in regard to the physical properties of the liquor, in that it gives a short-time contact.

To obtain high velocity, it is necessary to arrange the heating surface so that the ratio of the area of the heating surface to the cross-section of the tubes is as great as commercial construction and theoretical considerations will allow. Fortunately in this case practice can be adapted to theory, and in the climbing film evaporator the tubes are 7 metres long and 50 mm. in diameter, a ratio which has been found most successful in actual use. Other factors, such as the friction of the vapours, come into force and nullify the advantages of using longer and narrower tubes.

Another great advantage obtained, due to this very high liquor velocity in the "climbing film" tube, is that it makes possible a perfect separation of liquid and vapour. The vapour and liquor leave the tubes at about 30 metres per second and enter a separator at this high speed. They are caused to change their direction at right angles, without shock, and by utilising centrifugal force in this way, the heavier particles of the liquor are thrown out, due to centrifugal action, against the side of the separator, allowing the vapour to rise and pass away freed from the liquor.

#### Entrainment.

The prevention of entrainment, known colloquially as "frothing" or "blowing over," is of the utmost importance in evaporation. In the ordinary open tank, wherever the boiling is rapid, a considerable amount of liquid is carried away because the small particles, always projected vertically from the surface of a boiling liquid, do not fall back into the mass but are swept away by air currents. In the ordinary vacuum pan, entrainment trouble, that is, the carrying away of the product with the vapours, is a very common difficulty for the reasons previously stated.

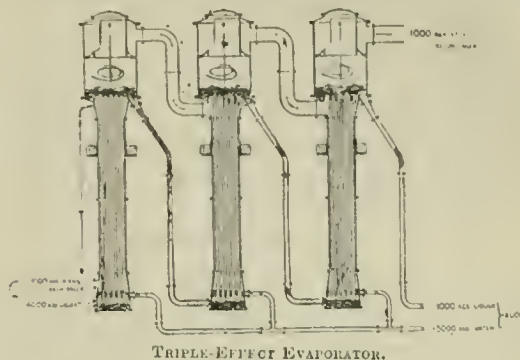
In any properly designed continuous evaporator, the amount of vapour leaving the apparatus is constant. It is, therefore, easy to determine by definite calculation the size of separator required to deal with the vapour, so that a definite speed is attained and no entrainment takes place.

#### Multiple effect.

The principle of multiple effect evaporation is that by alternate evaporation and condensation the latent heat is exchanged successively, as a means of separating water from solids. Thus—neglecting losses and self-evaporation, etc.—if 1 kilo. of steam be condensed in the first calandria of a triple effect through which liquor is passing, 1 kilo. of condensed water would be produced from the liquor in each of the calandrias of Nos. 2 and 3, and 1 kilo. of vapour would be produced in No. 3. If, therefore, an operation commences with 1 kilo. of boiler steam and 3 kilos. of water to be evaporated, then 3 kilos. of condensed water is recovered and 1 kilo. of steam at a lower temperature and pressure than that of the live steam supplied to the first effect. The cycle of operations finishes by producing 3 kilos. of condensed water and 1 kilo. of vapour at a low temperature and pressure—thus showing that what has happened is merely a transfer of heat.

According to theory, in designing a multiple-effect evaporator, when an equal drop is required in each effect, the different effects should have a different amount of heating surface, and although this has been very often carried out, it has been

definitely found in practice that the best average results are obtained, both from the point of view of first cost and operating expense, by adopting the same heating surface in each effect.



TRIPLE-EFFECT EVAPORATOR.

FIG. 6.

The object of multiple-effect evaporation is simply to reduce the cost of removing the water. The following table shows the economy obtained by means of multiple-effect evaporation.

TABLE II.  
COMMERCIAL FIGURES FOR WATER EVAPORATED  
PER 100 UNITS OF STEAM.

Steam 100	Single	Doub'e	Triple	Quadruple
Water ... ..	95	150	220	300

COMMERCIAL FIGURES FOR WATER EVAPORATED  
PER 100 UNITS OF COAL.  
COAL AT 6:1.

Coal 100	Single	Double	Triple	Quadruple
Water ... ..	570	900	1320	1800

Example—1000 kgs. of water evaporated in triple effect.

$$(a) \text{ Steam required} = \frac{1000}{220} \times \frac{100}{1} = 450 \text{ kgs.}$$

$$(b) \text{ Coal required} = \frac{450}{6} = 76 \text{ kgs.}$$

Actual working figures taken from a Kestner triple-effect evaporator concentrating tan extract liquors show that 23 tons of water was evaporated per ton of live steam in the first effect, so that, based on coal at 7:1 in the boiler, 16.1 tons of water is evaporated per ton of coal.

In multiple-effect evaporation assume that the temperature of the heating steam in the first effect at 6 lb. per sq. inch is 110° C., and that the vacuum is 26½"=50° C. in the condenser, then the total available temperature head or drop is 60° C. If this is to be spread over the three units of a triple effect, the average drop on each effect is 20° C.

Now the point that arises with any evaporator is, what is the minimum average drop which can be used for each effect, as this obviously fixes the number of effects—and consequently the economy to be obtained—between any fixed temperature limits?

If economy is of the utmost importance, because of high price of coal, or for other reasons, and where capital cost is of less importance than economical working, with the climbing film it is possible to operate with a greater number of effects between given temperature limits than with any other type of evaporator.

The extremely small drop required by a Kestner climbing film evaporator is illustrated by the



following. According to Messrs. Bell Bros.' patent for the generation of power from slag, molten slag is poured into a vessel, and generates steam at atmospheric pressure. The idea was to carry this vapour direct into turbines of the low-pressure type, working at, say, 28½" vacuum. It was found, however, that the steam contained chemicals of different kinds which attacked the rotor of the turbines. A Kestner climbing film evaporator was therefore installed which would provide clean steam and which would operate efficiently with a minimum loss of temperature drop. The evaporator operated on a heat drop of 5° C. and gave a heat efficiency of 91%, based on average working conditions.

#### *Use of saturated steam, etc.*

The idea that superheated steam can be advantageously used for evaporation is fallacious, because to evaporate quickly the heating steam must condense rapidly. Now, superheated steam acts like a non-condensable gas, and until it is cooled down to the temperature corresponding to its saturation pressure, practically no evaporation takes place. According to M. Normand, the rate of condensation with superheated steam is 20 to 30 times slower than with saturated steam.

There is also quite an erroneous idea of the value of waste gases for evaporation purposes. At first sight the use of waste gases holds out many apparent advantages, but after many years' study of the problem this idea has been dropped except in special circumstances. In the first place, owing to the enormous difference in the number of available heat units per lb. of steam, compared with those available in a pound of hot gas, the volume of gas passing over the outer surface of the evaporating tubes must be very large, and this, consequently, calls for very large calandrias with large spaces between the evaporating tubes. One pound of steam will give about 1000 available heat units, whereas, even assuming 1000° C. temperature drop for the gas, and a specific heat of 0.25, the heat units available per lb. of gas are only about 250.

In addition to this difficulty it must be remembered that the heat transmission between saturated steam and metallic tubular heating surfaces is very much better than the transmission between hot gas and similar surfaces. As a consequence, in order to effect the same evaporation per hour, it is necessary to provide from four to seven times as much area of heating surface when using hot gas as when using steam.

Further, when evaporating to predetermined density, it is very necessary that the temperature of the heating medium should be easily controlled and easily kept constant, two factors which are readily obtained with steam, whilst they are not nearly so readily obtained with hot gases.

#### *High concentration.*

The advantage of a continuous evaporator of the "film" type in regard to high concentration is very great. In this type of evaporator it is possible to concentrate liquors up to very high densities, in some cases to commercial dryness. For instance, in the climbing film evaporator, wood extracts can be concentrated under atmospheric pressure, from, say, 30° B., up to 85% solids. Owing to its temperature, the finished product is liquid, and runs by gravity out of the evaporator. On cooling the extract solidifies.

Any caustic soda liquors, excepting those prepared by the Leblanc process, can be concentrated up to 60% Na<sub>2</sub>O, or 76% NaOH, direct in a Kestner evaporator; this is, in a great many instances, quite sufficient for commercial purposes. This method is much more economical for producing solid caustic than the use of a direct-fired

pot. Moreover, it can be carried on continuously in series with a multiple-effect evaporator.

Owing to high solubility, some inorganic liquors, such as ammonium nitrate solutions, can be concentrated up to a very high density in a Kestner film evaporator, without the use of vacuum, and without precipitation of crystals, to as high as 85% solids. As the liquor is evaporated under atmospheric pressure, it leaves the evaporator at about 136° C., runs into a crystalliser, and yields crystals very quickly. Such high densities mean that a maximum amount of crystals is obtained for a given weight of mother liquor.

The above are merely instances to show the advantages obtained by high concentration in this type of apparatus.

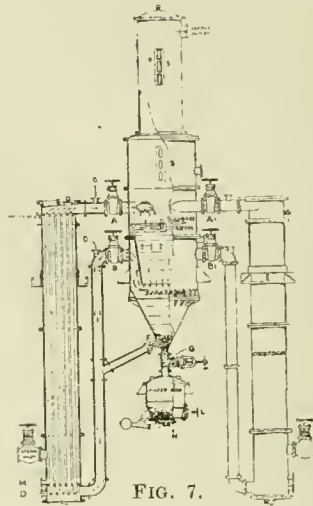


FIG. 7.

There are other types of Kestner film evaporators which have been evolved from the ordinary climbing film, and one, of particular interest at the present time, which has been in very large demand, is that known as the "Salting" type evaporator (see Fig. 7). This apparatus consists of a central separator, around which are arranged calandrias, in which the actual evaporation takes place. There is a circulation of the liquor from the separator into the calandrias, and from the calandrias back into the separator. In the separator crystals are thrown down as illustrated in Fig. 7.

This salting type evaporator is of great value when dealing with liquors containing two salts, one of which, owing to its lower solubility, can be precipitated, whilst the more soluble remains in solution. A typical example of such a liquor is that produced in a potash recovery process. This liquor contains chiefly potassium nitrate and sodium chloride; the solubility of the latter increases very slightly with the temperature and then remains almost constant, whilst the solubility of the potassium nitrate increases rapidly with the temperature. When both salts are present in solution a critical temperature is reached at which practically the whole of the sodium chloride is thrown down, whilst the whole of the potassium nitrate remains in solution.

Such liquids are usually concentrated in a double-effect evaporator. In the first effect in which the temperature is highest, concentration is maintained at such a point that the less soluble salt will not remain in solution but falls down into the bottom cone of the separator in the form of crystals. The liquor from this effect may be either drained out from a salt box and then carried over to a second effect, or in some cases may be

carried over directly from the first effect separator. In the second effect further concentration of the more soluble salt is carried out by means of the vapours driven off from the first effect.

Such problems involving a dual solution must of necessity be carefully examined and dealt with on their merits.

In some cases the more soluble salt is evaporated to crystallising point in the second effect, in others the concentration is only carried to such a point that the liquor may conveniently be taken as a highly concentrated solution to a rotary crystalliser, and thence to a centrifugal machine, from which the mother liquor is returned to the system. Such a method is adopted when the solubility of the second salt is extraordinarily high, such, for example, as ammonium nitrate, or when impurities are present. In the latter case, when the mother liquor becomes sufficiently charged with impurities, it is not returned to the system but treated separately.

#### DISCUSSION.

Mr. F. H. TERLESKI inquired the reason for washing out evaporators of the salting type every 12 hours. Another point was with regard to breakdowns. It might be necessary to put in an evaporator tube, 23 feet long, under a roof supporting a tank. In order to replace that tube the whole calandria would have to be taken down, whereas this would not be the case with an evaporator having shorter tubes. A further question was the number of long tubes in the Kestner evaporator and the possibility of clogging, which was not so serious in the case of the short-tube pan evaporator. The present salting type of evaporator was a distinct improvement upon the early types. With the ordinary multiple-effect pan evaporator where crystals were deposited it was not necessary to remove liquids to tanks outside to crystallise, with resulting heat losses. Crystallisation was continuous, drying was continuous, or practically so, and evaporation proceeded while the crystals were drying; this was not the case in the salting type illustrated. Taking out large volumes of liquid to deposit crystals and sucking back the cold mother liquor with less concentrated solutions would account for a fair amount of heat loss. The velocity of the climbing film was dependent upon the high speed of the vapours. Where entrainment troubles had to be considered there must be complete separation in relatively large volume areas in order to allow the liquid and the vapour to separate. The velocities in the Kestner evaporator were very considerable, and as the liquid and vapour separated at right angles without shock the separation was possibly complete. When, however, there were large quantities of liquid and vapour travelling at high velocities the possibilities of complete separation were greater where the vapour was in the out-take pipe of an ordinary evaporator, and where there was a distance of five or six feet to the header pipe from the calandria the losses should be less, when there was practically only vapour with a minute amount of liquid, as compared with an evaporator in which there was a separation of liquid and vapour practically at the same level, as set out in the Kestner type.

Mr. J. R. HANNAY said that from 80 to 90% of the steam generated in a calico-printing works was used for the evaporation of water from the goods which were running through the works. Anything that would tend to increase the efficiency of the drying plants would go a long way towards reducing coal consumption. Mr. Reavell had referred to the fallacy of the use of superheated steam for evaporation purposes. In the calico-printing trade there had grown up of recent years a very large practice of taking low-pressure

steam back to superheaters in the boiler house and then passing it through the drying cans. An experiment recently made in the works with which he was connected gave very disappointing results, and the opinion had been expressed that the superheaters were not working properly because the use of superheated steam had not resulted in much increased drying power. The problem of drying cloth over cylinders was one which required the attention of heating engineers.

Mr. NAGEL said that in one of the works with which he was connected two of the largest Kestner evaporators in existence had been installed and were giving excellent results.

Mr. THOMAS TAYLOR asked whether, owing to the great speed at which the film climbed, it would be possible to evaporate a liquor, which in the ordinary way would affect the metal of the tube, but might not do so owing to it being in contact for such a short time.

Dr. A. C. DUNNINGHAM inquired whether the Kestner film evaporator had been applied to the evaporation of brine in the manufacture of ordinary salt. He was impressed with the statement that the rapid film prevented the silting up of the tubes. How frequently was it necessary to wash out the tubes, because a statement had been made at one point in the paper that it was only necessary to do so at an interval of something like three weeks in certain cases?

Mr. BOOTH thought there might be a considerable loss of heat in lifting the liquid from a low level up to a comparatively high distance and also owing to the friction against a narrow tube to the high level of three metres.

Mr. REAVELL stated in reply that in the salting type evaporator there was not the same high efficiency of heat transmission as in the case of the film evaporator, dealing with liquors containing no salt in suspension, but even so, the Kestner evaporator maintained its advantages proportionally over the ordinary pan system. In the case of a liquor containing no solids in suspension, and being evaporated in an ordinary climbing film evaporator, there was an advantage of perhaps 50% in the heat transmission, as against the salting liquor in a salting type evaporator. He had not intended to convey the impression that in the case of the salting type evaporator every liquor must be taken out of the evaporator for crystallising purposes. The exact opposite was the case, but there were some instances in which impurities came down first, or where a special size of crystal was required, in which the liquor was evaporated to a very high density in the salting type evaporator, and then crystallised in an outside crystalliser. In a large majority of cases the salt crystals were precipitated directly in the separator of the Kestner salting type evaporator, this separator being quite independent of the evaporating tubes, whereas in a pan, any solids precipitated remained in the same vessel as the evaporating tubes themselves. The crystals were withdrawn from a Kestner salting type evaporator periodically by means of a salt box. By arranging a number of calandrias around a central separator, it was possible to have one calandria free for cleaning and repairing purposes, whilst the others remained continuously at work, whereas in the case of a pan a few leaky tubes meant the closing down of the whole evaporator. The Kestner climbing film evaporator had never been adopted by Mr. Kestner for liquors precipitating crystals. With regard to entrainment, in a pan, the provision of large volumes in the dome would not prevent the continuance of "frothing" once it had begun, but the whole mass of liquor would tend to be lifted as a "frothy" mass into the dome of the pan, and



then carried over to the condenser. The Kestner evaporator, on the contrary, contained a relatively small quantity of liquor at any given moment, and this moving at high velocity was thoroughly separated by means of the centrifugal baffle. It was unlikely that the Kestner evaporator would be put under a roof supporting a tank, especially as, owing to the extraordinarily small amount of floor space occupied by a Kestner evaporator, there was great latitude with regard to position. Concerning the treatment of acid liquors, experience had shown that owing to the high velocity in a Kestner evaporator certain liquors could be dealt with which could not be concentrated in any other apparatus. With regard to the loss of heat in raising the liquor from the low level to the high level of the climbing film evaporator, the work done when expressed in thermal units was relatively small, and the apparatus being lagged, a certain amount of it was recovered again as heat in the centrifugal baffle, and as velocity energy when the liquor passed from one effect to a second.

## Nottingham Section.

*Meeting held at Nottingham on April 24th, 1918.*

DR. R. M. CAVEN IN THE CHAIR.

### DISCUSSION ON BLEACHING AND DYEING UNDER WAR CONDITIONS.

Mr. S. J. PENTECOST, in opening the discussion, mentioned that the bleaching trade had suffered considerably through the withdrawal by the Government of sulphuric acid, which was looked upon as essential in the treatment of cotton goods. This withdrawal had necessitated the use of nitre cake, which had several drawbacks; one of the most serious was that nitre cake always contained iron which resulted in the production of a faint yellow colour if the bath should become neutral, and this was possible, especially in the case of the continuous process for bleaching. In this method the goods to be treated were introduced wet and the strength of the bath was maintained by the addition of a solution stronger than the solution in the bath. Owing to the variations in the degree of acidity of the nitre cake it was possible for the bath to become neutral, especially if a previous bath had been alkaline. In a paper dealing with oil stains on lace (this J., 1897, 409), he had emphasised the fact that these stains tendered the cotton owing to the presence of metal from the bearings, etc., and that the strength of the cotton was reduced as the result of catalytic action being set up in the bleaching solution. Iron deposited in the cotton by nitre cake caused a similar trouble.

A difficulty which was likely to become more acute in the future was due to the increasing scarcity of soap.

A further difficulty had arisen owing to the scarcity of bleaching powder; this had caused bleachers to consider the process of electrolysis salt solutions, and firms were at present prepared to supply plant for this purpose. One firm accomplished the object by electrolysis a salt solution of 26° Tw. (sp. gr. 1.13) with a current of 85 amps. at 110 volts, whereas another emphasised the necessity for employing a low voltage and used 600 amps. at 30 volts. The latter process gave a solution containing 9–10 grms. per litre, whereas the former yielded up to 15 grms. per litre. The makers of the electrical apparatus stated that the electrical process was as cheap as the bleaching powder method. The speaker, however, had not been able to confirm this and the results of

trials had shown that the electrolytic method was slightly dearer. However it had the advantage of cleanliness, and in the matter of the strength of the resulting fibre there was very little to choose between the methods.

In connection with dyeing the chief difficulty had arisen owing to the shortage of colours. However since 1916 British firms had made wonderful progress and were now in a position to supply most of the present needs. Two local firms had put down extensive plant for the production of oxidation Aniline Black, a dye which possessed exceptional qualities as regards fastness to light and beauty of shade. It was formerly produced on cotton hosiery very cheaply by the Germans, and was known as "Hermsdorf" Black.

Dr. E. P. HEDLEY mentioned that in America all the cotton was bleached electrically. He attributed the failure of the process in England to the freight charges on the salt, the cost of which was 15s. per ton in Cheshire and 27s. per ton in Northern Scotland and the South of England. As a source of power in some districts coal was as economical as water in England, owing to our coal deposits. Bleaching powder was objectionable in use owing to its bad effect on the machinery and the workpeople. Further it was not readily soluble and was drawn away on the fibre. There were three possible ways of obtaining bleaching solutions: (a) from bleaching powder; (b) by passing chlorine into sodium carbonate solution; (c) by the electrolysis of salt solution. The first method, besides the disadvantages above referred to, had this disadvantage that if sulphuric acid was used as the souring agent calcium sulphate was precipitated on the fibre and owing to the formation of an oxycellulose there was a weakening of the fibre. This drawback could be overcome by employing hydrochloric acid, which would form calcium chloride and this could be subsequently removed by washing. In the electrical method the compound formed was the sodium salt of hypochlorous acid, and even if sulphuric acid were employed the formation of sodium sulphate was not harmful and did not bring about the "tendering" of the fibre. Also the ash in the case of materials bleached with bleaching powder was considerably higher than that obtained when sodium hypochlorite was employed—1.2–1.5% in the former case and 0.03–0.08% in the latter. As liquid chlorine was now readily obtainable the process of making hypochlorous acid by the action of chlorine on sodium carbonate might be seriously considered by bleachers, and the reaction might be carried on by passing the chlorine beneath a bell-shaped cover immersed in the solution and provided with a stirrer. A suitable plant designed by the speaker was working efficiently.

Mr. J. H. DUNFORD mentioned in connection with the scarcity of sulphuric acid, that at present the Government were releasing sulphuric acid for the purpose of making soluble phosphates.

Mr. J. M. WILKIE asked if there was any marked effect in the products from the different methods of bleaching attributable to the small differences in the ash content.

Mr. PENTECOST replied that in many cases a high percentage of ash was a decided advantage from the seller's point of view and that it was only in the case of cotton cellulose which was to be used in the manufacture of explosives that a low ash content was essential. The bleaching powder process was now firmly established, and so far the results of the electrolytic method had not been exceptionally good. The electrolysis of the salt solution was carried out in slate vats and the cheapest source of power locally was a gas engine coupled to a generator.

## Birmingham Section.

*Meeting held at Birmingham University on Thursday, March 21st, 1918.*

MR. L. P. WILSON IN THE CHAIR.

### UTILISATION OF WASTE NESSLER SOLUTION.

BY WILLIAM CLIFFORD, ASSOC. M. INST. C.E.

In view of the scarcity of iodides, it is important to recover and utilise the mercuric iodide from the solutions remaining after determination of ammonia by means of Nessler's solution. Usually these liquids are thrown away.

A simple method for treating these residues is to pour them into a "Winchester" quart bottle and add a few c.c. of sulphuric acid; mercuric iodide is precipitated, and the supernatant liquid is thrown away. When sufficient iodide has accumulated it is dried and kept for use.

After various attempts to decompose the mercuric iodide, with a view to obtaining sodium iodide, the decomposition was effected by means of iron filings. Metallic mercury and ferrous iodide are obtained, and the latter is treated with sodium carbonate or caustic soda, which precipitates the iron and yields sodium iodide. 200 grms. of mercuric iodide is mixed with 20 grms. of iron turnings in a litre flask and 500 c.c. of distilled water added. The flask is left on the water oven or water bath. The reaction proceeds at once and is completed in about an hour. The green solution is filtered and boiled or allowed to stand overnight on the water bath, when the colour changes to a ruby red. The iron is then precipitated with an alkali carbonate or hydroxide. A second precipitation is needed to bring down the whole of the iron. The filtrate from the second precipitation may be used without further treatment for oxygen absorption determinations. 85% to 90% of the mercury is recovered in the metallic state.

It may not be generally known that sodium iodide and caustic soda may be used in place of potassium iodide and caustic potash in making up Nessler solution.

#### DISCUSSION.

The CHAIRMAN said that a solution of alkali hydroxide and glucose was fairly effective in reducing metallic salts; it would probably give a soluble iodide at once and at the same time precipitate metallic mercury.

MR. HEAVEN enquired whether the final product was fairly satisfactory, or whether it required treatment to decompose the iodate. In trying recently to recover copper iodide precipitates he found that the residue required considerable working up before it was possible to get a reasonably pure iodide.

MR. CLIFFORD, in reply, said he had not tried glucose. No difficulty had been experienced with regard to the utilisation of the iodide, as it was in a fairly pure condition. About 85% of the mercury was recovered as metallic mercury by grinding up the dried residue in a mortar. The whole of the mercury was not removed by this method, minute globules remaining adherent to the iron.

*Meeting held at Birmingham University on Thursday, April 18th, 1918.*

DR. R. S. MORRELL IN THE CHAIR.

### DISCOLORATION OF WHITE PAINT.

BY D. F. TWISS, D.SC., F.I.C.

In the use of a white "enamel" of good quality trouble was experienced in the tendency of the dried films to undergo discoloration, especially when articles painted with it were stored in a fairly warm situation; under these conditions the whiteness was soon replaced by a yellow or yellowish brown colour, the result being much the same whether the painted surface was exposed to the light of an ordinary room or was kept in the dark. The enamel or paint normally consisted of an oil varnish mixed with zinc oxide as pigment, and at first the almost natural tendency was to attribute the discoloration to the presence of some impurity in the pigment; as zinc oxide pigment at the present time is also often adulterated with lithopone, consisting mainly of zinc sulphide and barium sulphate, the zinc sulphide in this, by slowly reacting with lead compounds simultaneously present in the pigment or in the oil, might give rise to a gradual discoloration. Although these impurities were found in some samples of the paint, not only did exposure of dried films in an atmosphere of hydrogen sulphide fail to induce a greatly accelerated colour change, but samples of similar paint were discovered to be free from lithopone and lead and yet to undergo discoloration in a similar manner. A peculiarity of the discoloration was that it appeared to be confined mainly to the outer surface of the films, the under side against the painted article being affected to a relatively slight extent.

It is an interesting fact that some zinc pigments are not above suspicion as to their fastness; lithopone on exposure to light undergoes a reversible colour change, which, however, is hindered by admixture with linseed oil, an extreme and probably exaggerated statement of such behaviour having been made many years ago in a description of the behaviour of a lithopone paint on certain gate posts, which were "black all day and white all night" (Chemical News, 1881, 43, 283). Zinc oxide, however, is relatively insensitive to light, and the fact that light was not essential to the discoloration now in question disposed of the possibility that the trouble was due to the effect of light either on zinc oxide or on any accompanying lithopone.

Another common cause of the discoloration of white paints, which is often not fully realised, is the ease with which the surface becomes stained with dirt from the external atmosphere, and, amongst others, H. E. Armstrong and C. A. Klein (this J., 1913, 329) have drawn attention to the fact that the blackening of white lead paints is frequently due to this cause rather than to the action of atmospheric hydrogen sulphide. In any case the conditions under which the present discoloration occurred rendered this explanation inapplicable.

As the evidence indicated that the source of the discoloration was not in the pigment, attention was turned to the medium, and the seat of the trouble was then located. When a little of the medium was separated from the mixture and was absorbed into paper free from mineral impurities, the dried film, on being kept in a warm place, was found to develop gradually a



marked yellowish brown colour, a very marked change occurring in a few hours at 60° C. Linseed oil free from "gum," either alone or mixed with pigments, was found to suffer a similar alteration to the oil varnish, but in a still more marked degree.

In order to investigate the change further, strips of paper coated with the white paint, and also with linseed oil only, were hermetically enclosed in glass tubes from which the air had been expelled by a current of carbon dioxide; this gas was generated from marble and recently boiled dilute hydrochloric acid, and was then washed with water and dried with sulphuric acid before use. It was expected that under these conditions the discoloration would be prevented, but the unforeseen result was that the development of the yellow or brown colour occurred even more readily. As this colour change was effected in the dried paint film in a few hours at 60° C., in an atmosphere of carbon dioxide, the alteration in the oil would appear to be independent, not only of the oxidation to linoxyn, but also of the polymerisation process for which a temperature in the neighbourhood of 260°–280° C. is necessary (e.g. see Morrell, this J., 1915, 105).

Tests were made as to the conditions under which the yellow or brown discoloration could be removed and the original whiteness restored. It is a matter of common knowledge that such bleaching may generally be effected by exposure to sunlight, but for convenience a Cooper-Hewitt mercury vapour lamp was used as a more readily available source of chemically active radiation. In accordance with the natural expectation, it was found that, unlike the discoloration process, the bleaching process required the presence of air or oxygen. When the discoloured film, sealed in a glass tube containing carbon dioxide, was exposed to the actinic rays of the mercury lamp the discoloration persisted, although it changed somewhat in tint, becoming a shade browner. Bleached films could again be discoloured by keeping in a warm place, and the discoloration and bleaching processes could be repeated many times in succession, the tendency to discoloration then appearing to undergo gradual but only slight diminution. It was not possible to prevent the subsequent occurrence of discoloration in a dried white film by a preliminary prolonged exposure to ultra-violet light with access of air.

During the course of this work the following brief reference to white paints was noted in Lewkowitsch's treatise on "Oils, Fats, and Waxes" (Vol. 3, p. 61):—"The paint oil *par excellence* is linseed oil. In the manufacture of high-class white paints for the use of artists cold pressed walnut oil and poppy seed oil are employed in considerable quantities" (see also *ibid.*, Vol. 2, pp. 100 and 125). Although this statement is not definite with respect to any discoloration, it is suggestive of the possibility that white paints made with linseed oil have a less permanent colour than similar paints made with the other oils named. Tests showed that poppy seed oil, apart from being paler in colour when fresh, certainly has less tendency than linseed oil to discolour in this way, but a distinct, although slower, discoloration was perceptible under comparable conditions, the change in colour being still less obvious, although yet distinct, in the presence of a pigment. The especial tendency of linseed oil to discoloration is masked more effectively by white lead than by zinc oxide, doubtless because of the greater covering power of the former pigment, but even with a mixture of white lead and linseed oil the gradual development of the yellow coloration is observable, the possibility of the colour change being due to external

causes being again excluded by the occurrence of the change in an atmosphere of carbon dioxide in a sealed glass tube. The more feeble covering power of zinc oxide naturally causes the alteration of the colour of the medium to be more perceptible.

As to the nature of the chemical change underlying the alteration in colour, the experiments now described give no explanation other than that it is apparently independent of the well-known phenomena of oxidation and polymerisation of the oil. Probably some minor ingredient of the oil other than the glyceride mixture which forms the main constituent is to be regarded as the cause of the trouble, but further investigation is necessary before any definite conclusion can be drawn in this direction.

#### DISCUSSION.

The CHAIRMAN agreed with Dr. Twiss that the cause of discoloration lay in the oil and not in the pigment. Some time ago he had occasion to remove black paper coverings from varnished white enamels. One of the varnished coatings contained a high percentage of oil, while the other contained a large proportion of gum. The former showed marked discoloration. He inquired whether in Dr. Twiss' investigation traces of manganese had been present, because he was inclined to think that the colour changes might be due to the presence of manganese salts in the oil.

Mr. F. H. ALCOCK suggested that the discoloration might possibly be due to the presence of alkali. His view was that the yellowness of good quality putty was due to the causticity of the chalk. To neutralise the yellowness painters used a little blue.

Mr. L. P. WILSON enquired whether the experiments were made with a pure raw linseed oil or whether a boiled oil had been used. If this were so, it might supply the answer to Dr. Morrell's question. Manganese in minute quantities had a great colouring effect, and it was most evident, of course, when the manganese salts were dissolved in turpentine.

Mr. G. S. HEAVEN stated that in the factory with which he was concerned the white paint showed, on the whole, little discoloration; but when used to paint an imitation uralite it did not dry readily, and it showed a marked yellow coloration. The effect was, however, different on the various panels, but he noticed that a slight yellowing disappeared on exposure to light. He regarded the preliminary darkening, which disappeared on exposure to light, as being due to the linseed oil. He believed other colour evidences were due to free lime in the cement of the uralite. Mineral oil, in place of linseed oil or admixed with linseed oil, had also caused yellowing, but it was not well marked, and did not disappear on exposure to light.

Mr. G. H. HOWSE said that in investigations upon the discoloration of white paint which he had been carrying out for some years it had been found that no pigment or medium gave similar results in various parts of the country. Varying degrees of blackening and discoloration were observed with the same paints, including mixtures made with long oil, short oil, all-gum, spirit, gelatin, and water-glass media.

The action of radium and radioactive bodies as displayed in the spintharoscope, in which zinc sulphide was the substance acted upon, suggested that radioactivity had a good deal to do with the discoloration of white pigments, or, rather, upset the balance of colours which produce a white effect. He had therefore carried out an extended research with an ultra-violet lamp, and the results of these experiments led him to

assume that radioactivity was a factor to be considered, although he had not arrived at any definite conclusion in the matter.

Dr. Twiss, in reply, stated that as not only the "boiled oils" but also the raw oils showed discoloration of the type described the discoloration in question was not due to the presence of lead, and also that as the darkening was rather more rapid in an atmosphere of carbon dioxide than in the air it was exceedingly unlikely that the change in colour could with any degree of probability be attributed to the presence of alkali. In the case of the behaviour of a white paint referred to by one speaker, which contained a zinc oxide pigment and a cobalt drier and showed an especial tendency to darken in certain parts which were liable to be subject to the action of lime, it appeared quite possible that the discoloration might have been due to the formation of cobalt sulphide by the action of zinc sulphide present as impurity in the zinc oxide on the cobalt compound under the influence of the lime. With reference to the suggestion of the possibility that the discoloration could be attributed in some way to a radioactive effect rather than to a chemical change, Dr. Twiss was of the opinion that, whether induced by mere gentle heating or by the influence of radioactive substances, the alteration in colour was the outward manifestation of a chemical change in the medium of the paint.

#### THE TRANSFORMATION OF METHYL α-ELÆOSTEARATE INTO METHYL β-ELÆOSTEARATE.

BY R. S. MORRELL, M.A., Ph.D., F.I.C.

In continuation of the investigation of the properties of thickened drying oils Chinese wood oil (tung oil) was examined, following the methods described in a former paper (Morrell, this J., 1915, 105). The problem is simpler than in the case of linseed oil owing to the fact that tung oil contains a smaller number of component unsaturated glycerides.

The mode of investigation was to thicken the oil at 240° C. for 20 minutes until it commenced to set, and after rapid cooling to remove the polymerised part by treatment with acetone. The thickened oil had a specific gravity 0.9638 (15° C.) and a molecular weight (in benzol) of 1431;  $[GIX]_D^{25} GIX$  requires 1317. The original oil had sp. gr. 0.944 and  $n_D^{20} = 1.5171$  (12.5° C.). The value obtained for the molecular weight would confirm the view that half the oil consists of polymerised molecules. After extraction with acetone the soluble portion (41.5%) was found to have the following constants:—Iodine value 106.3 (1½ hrs.): sp. gr. 0.954 (11.5° C.), mol. wt. 876,  $n_D^{20} = 1.5144$  (11.5° C.). The insoluble portion (47.5%) was a very viscid substance, therefore the iodine value alone was determined; this was found to be 104.3 (1½ hrs.). Treatment of the acetone-soluble part with sodium methoxide (Bull's method) gave a methyl ester, sp. gr. 0.9159 (10° C.),  $n_D^{20} = 1.4958$  (10° C.), mol. wt. (in benzol) 300; iodine value 151.8 (1½ hrs.), 178 (6 days).  $C_{18}H_{32}O_2 \cdot CH_3$  requires mol. wt. 294; iodine value 173. The portion insoluble in acetone was likewise transformed into a methyl ester, sp. gr. 0.926 (12° C.);  $n_D^{20} = 1.49850$  (12° C.); iodine value 138 (1½ hrs.), 168 (6 days); mol. wt. (benzol) 393.

Mention must be made of the iodine values obtained: in the case of the soluble and insoluble glycerides the values are too low and too high respectively and the methyl esters give normal values only after long contact with Wijs' solution.

The action of the sodium methoxide destroys

polymerisation almost entirely, a fact noticed in a previous paper (*loc. cit.*). The methyl esters from the portions soluble and insoluble in acetone respectively were fractionated under reduced pressure (10 mm.). The first methyl ester gave a non-volatile residue (33%) with mol. wt. (benzol) 481, and the distillate (59.7%) came over between 200° and 219° C. (Iodine value 143–160, mol. wt. (benzol) 284–71). On redistillation 92% passed over between 209° and 224° C., corresponding to 55.3% of the original ester. The distillate had sp. gr. 0.900 (12° C.) and  $n_D^{20} = 1.4812$  (12° C.).

The purified ester on saponification in the cold gave a good yield of an acid, m. pt. 66°–69° C., which on thrice recrystallising from 60% alcohol gave β-elæostearic acid, m. pt. 71°–72° C., iodine value 172. On ultimate analysis 0.1196 grm. gave 0.3398 grm. CO<sub>2</sub> and 0.1204 grm. H<sub>2</sub>O: C = 77.49, H = 11.2%;  $C_{18}H_{32}O_2$  requires C = 77.14, H = 11.42.

The methyl ester from the portion insoluble in acetone was treated in the same way with the same results in almost every detail, except that the molecular weight of the non-volatile residue after the first distillation was 716. The acid obtained after saponification of the redistilled methyl ester at the ordinary temperature was β-elæostearic acid, m. pt. 71°–72° C. (0.136 grm. gave 0.3765 grm. CO<sub>2</sub> and 0.130 grm. H<sub>2</sub>O; C = 77.43, H = 11.0%;  $C_{18}H_{32}O_2$  requires C = 77.14, H = 11.42%). The iodine value of the acid was 172.8. The properties of the fractionated methyl esters from the soluble and insoluble forms are practically identical. There are none of the complications occurring in olive, poppyseed, and linseed oils with their mixed and variable glyceride molecules. It is a straightforward case of polymerisation in which the complexes are broken up by removal of the glyceryl radical. At what stage the stereo-isomeric change occurred had to be decided: it might have occurred during the thickening of the oil or during the distillation of the methyl ester. Sodium methoxide might be a factor, but this was very unlikely. In the saponification of the non-distilled methyl ester there was always a large amount of oily acid as impurity so that it was not possible to obtain a sufficient yield of a crystalline acid, α or β, to decide which was present in predominant quantity. Examination of the cerium salt from the undistilled methyl ester obtained from the acetone-soluble part of the oil showed that α-acid was present almost entirely and no β-acid. Cerium α-elæostearate is completely soluble in ether but cerium β-elæostearate is insoluble (Morrell, Chem. Soc. Trans., 1918, 113, 111; see this J., 1918, 130 A).

It was found that Chinese wood oil yielding the α-acid in good quantity was transformed into the methyl ester in the manner previously described. The methyl ester was fractionated under identical conditions and gave a slightly smaller non-volatile residue (15%). The methyl ester was not identical with those described previously and indicated a slightly simpler molecule, but the acid obtained was β-elæostearic acid, m. pt. 71°–72° C., C = 77.7, H = 11.1%.

The conclusion drawn is that stereo-isomeric change has occurred during the distillation of the ester. Comparison of the physical properties of the crude and redistilled methyl ester showed the former to possess a slightly higher sp. gr. (0.9022 against 0.899 at 16° C.) and  $n_D^{20}$  (1.516 against 1.496), but the iodine value shows no marked difference. It is evident that during the rapid thickening of the wood oil (20 minutes at 240° C.) no isomerism has occurred, but stereo-isomeric change ensues on distillation of the methyl ester.

This conclusion does not invalidate the suggestions made as to isomeric changes occurring during the polymerisation of linseed oil, because the



evidence brought forward was very decided; moreover the length of time necessary to produce thickening in the two cases is vastly different (20 minutes against 28–60 hours). The fractionation of the methyl esters takes considerable time, during which the esters are maintained at a high temperature. The fact of the formation of the  $\beta$ -ester confirms the view that molecular rearrangements are most probable when drying oils are heated, moreover the quantities of the non-volatile residues in the distillation of the methyl esters obtained from thickened and non-thickened wood oil are different, which is a fact worthy of remark.

#### DISCUSSION.

Dr. D. F. TWISS asked regarding the polymerisation of Chinese wood oil what proportion of the oil which had been heated just short of "setting" remained soluble in acetone, because he was of the opinion that even in the firm gelatinised oil a considerable percentage still could be removed by extraction with this solvent. He also asked whether the structural change from the  $\alpha$  to the  $\beta$  acid was in the same direction as that induced in oleic acid by nitrous acid.

Dr. E. B. MAXTED stated that with regard to the melting point the result varied, apparently depending on temperature when hydrogenated. He wondered whether any change could have taken place in the acid.

Mr. G. H. HOWSE inquired if Dr. Morrell had taken any records as to the polarity or refractive index of the acids before and after polymerisation. Would any such records help to determine the direction in which the changes mentioned occurred?

Mr. H. G. EVANS pointed out that in the treatment of the oils there were three processes—(1) polymerisation, (2) formation of esters by sodium methoxide, (3) distillation. Dr. Morrell mentioned only the first and last as likely to cause changes in the position of double bonds. Was it possible that the sodium methoxide used in the formation of the esters could have any effect on the double linkages?

Dr. MORRELL in reply stated that the percentage of oil insoluble in acetone in the case of thickened linseed oil was 50% and likewise the percentage of acetone-insoluble oil from thickened Chinese wood oil was 50%. Nitrous acid did not change  $\alpha$ -elaeostearic acid into the  $\beta$  modification, but the transformation readily occurred in sunlight. It was quite possible that sodium methoxide might be a factor in causing stereo-isomeric change. The question of ring-formation on heating was difficult to answer. From the work done on thickened linseed oil the evidence was inconclusive but was generally contrary to ring-formation.

## Liverpool Section.

*Meeting held at Liverpool University on Friday, March 18th, 1918.*

MR. A. T. SMITH IN THE CHAIR.

### NOTES ON OIL PITCH.

BY W. MANSBRIDGE.

Pitch, as met with in commerce, is as various in its properties as the raw materials from which it is produced, and with each variety, especially mineral oil pitches, there are a number of distinct grades each intended to meet some particular requirement. As an instance, one refinery lists no less than fourteen grades, all produced from the

same crude oil and only differing materially in melting point and hardness.

Blast furnace and coke oven pitches are not so well known as that produced from coal tar, but they have similar properties and uses, as one would expect seeing that they are derived from the same material—coal—in each case; they are, however, harder, more brilliant, and freer from smell and can be employed where coal tar pitch would be objectionable.

Next in order of magnitude of production is the pitch made from mineral oil. Its importance dates from the discovery of the Texas oil fields, for although oil pitch, as it is now often called, was made from certain crude petroleum, the quantity was not large and its consistence hardly entitled it to be called pitch.

Later the Galician and Rumanian oil fields were developed and as the oils from both these fields have an asphaltum base they give a good yield of pitch to the refiner. Large quantities also come from Mexico and California, the toughness of the former variety at low temperatures making it exceptionally suitable for road construction and similar work.

It was at first difficult to find a market for the very large quantities of pitch made from the Texas oil and probably many thousands of tons were burned under the stills. In time, however, its valuable properties were recognised and an extensive field was opened up for its use. The yield varies according to the melting point desired and the quality of the crude oil. In a general way commercial grades may be said to follow the melting point, and as this characteristic rises so also the other physical tests are modified; the flash point is raised, the penetration number is lowered, and naphtha-soluble bitumens diminished in amount. The table on p. 183 t, which relates to the products of a single refinery, shows this very well and may be taken as a type of what is obtained from a crude oil with bituminous base.

Oil pitches are now used to a very large extent, especially in America, for architectural, road-making, insulating, and general waterproofing purposes, and as anti-corrosive paints or coatings for steel work. In England they have to compete with cheap coal tar pitch which fulfils many of the conditions required and at about one quarter the price in normal times. Notwithstanding this, oil pitches have found ever-increasing favour for the better classes of work. The properties of toughness and flexibility at low temperatures, resistance to acids and alkalis, the tenacity with which they adhere to metallic surfaces, their high insulation resistance, and in some cases brilliancy of fracture, make them specially useful in certain industries. Some crude oils yield a hard, brittle pitch almost entirely soluble in turpentine and substitutes, which by judicious blending, can be used in high class black Japans such as bicycle enamel.

Closely allied are the mineral bitumens or asphaltum, properly so called, gilsonite, manjak, Syrian, Cuban, and Colombian. Hard, glossy, very black, soluble in all the spirits and oils used for the purpose, these are pre-eminently materials for the varnish maker, and enter into the composition of the highest grade of Japan and black enamel whenever a beautiful finish is desired. Asphaltum is hand-picked and often sold with a guarantee as to its percentage of ash.

*Stearine pitch.* This term was formerly restricted to the residue obtained in the distillation of tallow and palm oil but it has since been extended to include any pitch made from fatty acids, except in cases where a modification of the name designated some advantage. Stearine pitch is now principally used, the hardest varieties

pure, the softer sorts after blending with an asphaltum, such as gilsonite. In some form of varnish making, as it is entirely soluble in turpentine, petroleum spirit, etc. Some varieties, such as the harder kinds yielded by drying and semi-drying oils, require special treatment to obtain complete solution.

Cottonseed pitch although often sold as stearine pitch stands in a class by itself. This material is made by distilling the cottonseed black grease prepared from the "mucilage" obtained in refining crude cottonseed oil. The mucilage, which is a mixture of saponified fat, colouring matter, and neutral oil, is treated with cheap mineral acid to decompose the soap, and the resulting black grease is washed and distilled. The quality of the pitch depends upon the kind of crude oil, the skill of the refiner, and the particular methods of distillation. If the crude cottonseed oil has been crushed from American decorticated seed the black grease is very free from colouring matter, high in neutral oil, and gives on distillation an elastic rubber-like pitch which can be vulcanised. On the other hand the pitch from Egyptian undecorticated seed is very hard, brittle, and difficult to use without special treatment. When crude unrefined cottonseed oil is distilled the pitch obtained is elastic in the highest degree, emerging from the still as a spongy mass very similar in appearance to the rubber sponges of commerce.

Elastic cottonseed pitch after vulcanisation is ex-

actly the true melting point, but rather the point at which the sample under examination becomes soft enough to flow. In one of these, known as the Wendriner method, itself an improvement on the procedure of Kraemer and Sarnov, the movement is assisted by a weight of mercury. A convenient and useful way to carry out this test, and one used by the author for many years with good results, is as follows:—

A small piece of the pitch, weighing about 6 grms., is taken from a clear interior surface of the sample and attached to the bulb of a thermometer, by slightly warming the substance and pressing it on the glass so that it adheres firmly. Not more than one-third of the bulb should be covered. If the pitch has to be made very hot to effect this it should be allowed to become cold again before proceeding with the test. The thermometer is arranged in a 1-inch boiling tube over an ordinary argand burner (not argand Bunsen burner), in such a way that, the tube being vertical, the sample is in the centre of the tube and  $1\frac{1}{2}$  in. from the bottom. The thermometer passes through a cork which closes the tube, and which has a second hole bored through it as a vent for the heated air. The tube is held vertically over the burner by means of a clamp fixed to a retort stand, a piece of wire gauze interposed between the burner and the glass tube serving to moderate the heat and support the tube.

The operation is commenced by so regulating

TABLE A.

*Comparison of different grades of mineral oil pitch from the same refinery (American).*

	Road binders.						Asphalt cements.				Blown asphalts.		
Specific gravity at 60° F. . .	1.035	1.037	1.039	1.043	1.046		1.049	1.055	1.058	1.060	1.023	1.025	1.032
Flash point, closed, ° F. . .	120	165	470	495	175		475	180	451	480	360	383	445
" open, ° F. . .	450	500	500	520	500		500	520	500	500	390	400	572
Fire test, ° F. . .	500	560	580	610	580		590	605	585	580	460	425	590
Solubility in:—													
(a) Petroleum spirit 0.680 s.g. . .	75	75	75	74	72		71	70	70	66	64	61	61
(b) Carbon tetrachloride . . .	99.9	99.9	99.9	99.9	99.9		99.9	99.9	99.9	99.9	99.9	99.9	99.9
(c) Carbon bisulphide . . .	99.8	99.8	99.8	99.8	99.8		99.8	99.8	99.8	99.8	99.8	99.8	99.8
Asb. % . . .	0.2	0.2	0.2	0.2	0.2		0.2	0.2	0.2	0.2	0.1	0.1	0.1
Penetration at 77° F. . .	175	145	125	110	100		85	60	45	35	48	26	20
Melting point, cube, ° F. . .	112	115	126	130	136		140	146	151	160	200	250	300
Paraffin wax, % . . .	0.3	0.3	0.3	0.3	0.3		0.7	0.6	0.6	0.6	—	—	—
Sulphur, % . . .	4.5	4.9	4.9	4.9	4.8		4.8	4.7	5.6	5.6	4.5	3.7	4.0

tensively used in electric cable-making. Such cables are, however, apt to "decentralise" and eventually to break down. If laid in cold, frosty weather the insulation, having become brittle, cracks as the cable is unwound from the drum and breakdown occurs from that cause. The harder kinds of cottonseed pitch are extensively used in the manufacture of waterproof paper, and are applied in the form of a varnish by suitable machinery.

Wool pitch, from crude brown wool fat, furnishes an excellent material for the lubrication of the bearings in hot rolling mills in steel works, etc. When melted, even at a high temperature, it remains viscid and keeps its lubricating power under very severe conditions of heat.

Ozokerit and ceresin pitch are employed as "finishing wax," as in the form of heel-ball; suitable qualities burnish well and take a high polish; they are also useful for many purposes of the electric cable manufacturer and for core vent wick as used by ironfounders.

Bone pitch comes from the tar obtained in bone black making; it is the blackest of all the pitches and is used by the varnish maker to deepen the colour of his enamel.

#### *Commercial testing and analysis of pitch.*

**Melting point.** There are three methods employed for this test, none of which can be said to indicate

the burner that only a very small blue flame is visible, and as required the flame is gradually raised, until the pitch fuses. When the pitch reaches the bottom of the tube, either as a detached drop, or as a viscid thread still remaining attached to the mass on the thermometer bulb, the temperature is noted and recorded as the melting point of the pitch.

The speed with which the test is conducted may greatly affect the accuracy of the result, and in order to ensure uniformity the author allows the temperature to rise at the rate of 3° F. per minute for pitch melting below 140° F. and at 5° F. per minute for samples melting above 140° starting from the temperature of the room. Usually the operation takes about 20 minutes.

Some operators use what is called the "cube" method; in this a 1 cm. cube of the material is suspended on wire in an air bath, the temperature of which is raised at the rate of 5° F. per minute. The bulb of the thermometer is placed at the side of the cube and within one millimetre of, but not touching, the sample.

The "melting point" is most usefully employed to discriminate between different grades of the same kind of pitch, though valuable information may sometimes be gained by the character of the drop. Some varieties melt more completely than others, or more suddenly, and yield a detached drop of liquid material while others give a viscid



string which only really melts on touching the hot bottom of the tube in which the experiment is conducted.

**Solubility.** A rough test for solubility can be made with a distillate of petroleum known as "White spirit," though lighter petroleum spirit will serve if used with caution. About 10 c.c. of the solvent is added, the thermometer lowered to the bottom of the tube, and the contents boiled. When thoroughly mixed or dissolved as the case may be, the whole is allowed to settle for a few minutes and the residue examined; its quantity and appearance are indicative of the procedure to be followed in a complete examination for solubility should such be required.

The general practice is to treat the sample with different solvents in succession, having in view the purpose for which the pitch is intended. This will determine the kind of solvent to be employed and in most cases it will be found that some may be omitted. Ordinarily, petroleum spirit (light motor spirit), "white spirit" (sp. gr. 0.785–0.800), 90% benzol, carbon tetrachloride, and carbon bisulphide are used, and in special cases turpentine or linseed oil. A good method of applying the solubility test is to take sufficient of the sample, as indicated by the preliminary rough trial above referred to (5 to 10 grms. is generally enough), and dissolve this in the solvent by first melting and then boiling, when practicable with enough of the solvent to give a fairly thin solution; this is then transferred to a stoppered graduated measure and made up to 50 c.c. or 100 c.c. as convenient. The solution is allowed to stand overnight; in the morning the clear supernatant liquid can be decanted and if a portion be evaporated the residue of soluble bitumens may be examined. The precipitate is shaken with a further quantity of the solvent, transferred to a weighed filter, washed until the filtrate is no longer coloured, dried, and weighed. A Soxhlet extractor is often recommended for this test but the author prefers the above procedure, and can often obtain sufficient information from an inspection of the graduated measures in the morning to serve the purpose in view.

**Penetration test.** This test is more frequently used in America than in this country, chiefly by the producers of mineral oil pitch to grade their products. The New York Testing Laboratory penetrometer and the Dow penetrometer are the forms in general use. Neither of these instruments is regarded in this country as being satisfactory for the purpose and efforts are being directed towards designing an improved pattern capable of yielding better results. In this test a weighted needle is so arranged that it bears upon a portion of the sample kept at a stated temperature. The needles are of various sections and the load can be varied; the results are expressed by the distance in decimillimetres which the selected needle enters the pitch in say 5 seconds with 100 grms. load at a specified temperature.

**Other tests.** The flash point, open and closed test, and the fire test, are often called for and may be determined in the same way as for mineral oils, using the Gray flash point apparatus or the Pensky-Martens tester.

The specific gravity is conveniently obtained, with sufficient accuracy for ordinary purposes, by cutting a cube from the centre of a block of the sample with a hot, thin-bladed knife and adjusting a solution of glycerin at 60° F. so that the cube when immersed in it neither rises nor falls; no adhering air bubbles are allowed and the sample must be free from cavities. The specific gravity of the glycerin is then taken in the ordinary way. Owing to the difficulty of wetting the pitch this will be found quicker than by weighing in water

as is usually done with minerals. For samples lighter than water a mixture of water and methylated spirit may be used, but this will very seldom be required.

Saponification and titration of the fatty acids in stearine pitches may be carried out by boiling to dryness 30 grms. of the sample with 20 c.c. of methylated spirit and 10 c.c. of a 25% solution of sodium hydroxide; the partially saponified mass is then dissolved in hot water, boiled with dilute sulphuric acid until the soap is decomposed, the pitch layer washed twice, or until free from mineral acid, with water (by boiling), dried, and titrated with  $N/2$  sodium hydroxide, using alkali-blue as indicator. The results are calculated to oleic acid.

The volatilisation test, sometimes specified, is carried out by heating a convenient quantity, e.g., 20 grms., in a circular vessel  $2\frac{1}{2}$  inches in diameter at the surface of the melted pitch, for 5 hours at 350° F. and noting the loss.

Sulphur is determined by the well-known lime or magnesia method, using two crucibles, one inverted in the other, for the ignition.

The electrical tests required by the cable manufacturers call for special apparatus not available in the ordinary works laboratory.

#### *Scheme for the examination of pitch.*

The want of a simple, trustworthy scheme for the examination and identification of pitch must often have occurred to the works chemist or engineer when in search of suitable material or to control supplies. In order to supply this need a description is given below of the methods followed for many years when dealing with samples of unknown origin. It is necessary, however, to point out that the scheme is not intended, except in the most general way, to apply to mixtures; such may, and often do, present problems of extreme difficulty, as in the case of proprietary articles which are specially compounded with that object. If this limitation be kept in mind, the scheme will be found very helpful in most cases.

The scheme is based on the separation of pitches into two classes by their behaviour when treated with alcoholic soda or potash solution. These two groups are then subdivided according to the indications given by treatment with "white spirit" and finally identified by special tests or characters. "White spirit" has been chosen because it can easily be obtained and it is not so volatile as the light petroleum spirits, so that the sample can be more freely boiled; further, if it is not at hand ordinary petroleum burning oil will give nearly the same results. The grade of "white spirit" recommended should have a boiling point near to 140° C. and a specific gravity between 0.785 and 0.8.

The information obtained by working through the scheme will naturally be followed by the special analysis necessary to decide whether the sample answers any particular specification; these requirements are too varied to be dealt with in these notes.

The sample is boiled with alcoholic soda solution until nearly dry; this is best done in a 4 or 5 inch porcelain basin.

**Section 1.** The sample is wholly or partly saponified. Stearine pitch, all varieties. Woolgrease pitch. Bone pitch.

**Section 2.** No saponification takes place. Coal tar group. Mineral oil group. Natural asphaltums. Ozokerit pitch. Wood tar pitch.

**Section 1.** Boil about 0.6 gm. in 10 c.c. of white spirit, allow to settle for a few minutes, decant or filter, and examine the residue.

The substance dissolves—

(a) Stearine pitch from all non-drying fats.

(b) Wool grease pitch. This may be recognised by its tenacity when nearly melted and by the presence of cholesterol.

The substance partly dissolves—

(a) Pitch from drying or semi-drying oils and fats, including soft cottonseed pitch. This is usually oxidised when testing melting point. The insoluble portion is in broken films or scales which powder when rubbed between the fingers.

(b) Bone pitch, giving a characteristic odour of bones when heated.

The substance is insoluble or nearly so—

(a) Hard cottonseed pitch (melts in flame).

(b) Elastic cottonseed pitch, sometimes called "rubber" pitch. Does not melt in flame, will stretch and return.

Section 2. Treat with white spirit as in Section 1.

The sample dissolves—

(a) Soft mineral oil pitch, of treacly consistence or very plastic.

(b) Natural asphaltums, when free from mineral matter (ash). Hard and brittle with brilliant fracture.

The sample is only partly soluble—

(a) Nearly all mineral oil pitch. This is more or less plastic and has very low ash, usually below 0.2%.

(b) Natural asphaltum when containing mineral matter. Hard, brittle and can be powdered; ash high, often visible with a lens in the sample.

(c) Ozokerite pitch. Fracture rough; waxy when heated; will burnish with a warm, bright iron.

(d) Trinidad pitch. Plastic to brittle according to melting point; hard varieties very dull rough fracture, with high ash, not visible.

The sample is insoluble; the spirit is coloured yellow and becomes highly fluorescent—

(a) Coal tar group, including blast furnace and coke oven pitch. These give a characteristic smell when heated.

(b) A few mineral oil pitches, which give an asphaltum smell when heated.

(c) Wood tar pitch, giving a woody smell when heated, like Stockholm tar.

of coal as a fuel have brought about the centralisation of lime-burning, and it is now more convenient for farmers to obtain their lime by rail from these centres. Besides, the use of grinding plant has led to the production of forms of quicklime and carbonate which are easier to handle and to distribute than lump lime.

During the last 50 years the practice of liming soils has fallen off to a very marked extent, and one of the reasons for this falling-off is the ignorance of many farmers as to the significance of the large number of factors responsible for the satisfactory growth of crops, and particularly the varied and essential influences of lime on the productivity of soils. After the introduction of artificial fertilisers it was much too commonly believed that liming could be neglected with impunity where such new manures were used.\* Many artificers call rather for an increased use of lime.

Any one of the factors referred to may become a "limiting factor," and there is no doubt that over large areas in Yorkshire deficiency of lime in the soil seriously limits crop production.

Such soils are said to be "sour," and are recognised in the field by the setting up of certain conditions (not yet fully investigated) which affect the soil physically as well as chemically, and which are unfavourable to the growth of most cultivated crops as well as to useful micro-organisms such as the nodule organisms of leguminous plants, other nitrogen-fixing organisms (*azotobacter*), and the nitrifying organisms.

Heavy soils tend to become sticky and to set hard on drying owing to a delocalisation of the clay. Certain classes of weeds become troublesome, e.g., sorrel (*Rumex acetosa*), and on light land spurrey (*Spergula arvensis*); whilst cruciferous crops are commonly affected with "finger and toe." Most leguminous crops are particularly sensitive to "sour" soil conditions.

In the case of grass land, "sourness" of the soil gives rise to the formation of the well-known "mat" of undecayed organic matter which forms on the surface and does not become incorporated with the soil. Conditions become unsuitable for the more useful grasses, the places of which are taken by inferior grasses and weeds, which usually include sheep's sorrel, field wood-rush, heath bed-straw, and other typically moorland plants.

The soil itself shows a general deterioration which includes a reduction in the number of bacteria, and no doubt gives rise to the condition often spoken of by practical men as "dead."

Originally "sourness" was attributed to the presence of acid substances, partly mineral, but chiefly organic, and produced during the decomposition of organic remains in a soil deficient in bases, particularly lime. Many organic compounds of an acidic nature have been isolated from the "humus" of soils. Later this view was challenged on the grounds that most of the apparently acidic properties can be explained by the colloidal nature of the organic matter and the clay; whilst still more recently "sour" soils have been defined as those which are, for bases, "absorptively unsaturated."

A determination of the amount of calcium carbonate in the soil does not always reveal the true state of affairs, since two similar soils containing approximately equal and insignificant quantities of carbonate may behave very differently towards crops which are sensitive to "sour" conditions, and may respond very differently to dressings of lime.

\* This is perhaps hardly to be wondered at since the production of artificers found soils generally well supplied with lime, so that for some time no deterioration was experienced, and a new generation of farmers had largely arisen before the trouble became acute.

## Yorkshire Section.

Meeting held at the Queen's Hotel, Leeds, on  
March 11th, 1918.

PROF. J. W. COBB IN THE CHAIR.

### LIME, AND THE LIMING OF SOILS.

BY J. A. HANLEY.

Neglect of liming is the primary cause of the unproductive nature of large areas of land in Yorkshire, and the seriousness of the results of this neglect has been emphasised recently, firstly, by the dying off of many acres of grass in the West Riding—usually on coal-measures soils—and secondly by the difficulties attending the ploughing-out and other operations necessary to the conversion of grass into arable land in many parts of the county.

A good deal of evidence has been accumulated recently as to the "lime-requirements" of various types of Yorkshire soils, and the sources of lime available for the county.

Before the introduction of artificial fertilisers, lime and dung were the staple manures, but owing to the more restricted facilities for transport, lime had to be produced nearer the farms and the sources of fuel.

The rapid growth of other industries and the use



Soils which are definitely "sour" are not merely devoid of lime but they require the incorporation of a quantity of lime, sufficient, under natural conditions, to bring them to the point of saturation. Many methods have been introduced for the determination, in the laboratory, of this quantity of lime, which is known as the "*lime-requirement*."

In the Hutchinson and MacLennan method,\* which has recently been applied to a large number of Yorkshire soils of different types, the soil is treated with a freshly prepared solution of calcium bicarbonate and a determination made of the lime absorbed. The method has always given results in close agreement with actual observations in the field and with field experiments.

The following is a list of a few results obtained with a variety of Yorkshire soils:—

However the "sourness" is induced, the ultimate effects on growth of crops, prevalence of weeds, etc., is the same.

Instances of "sourness" produced or increased by the use of sulphate of ammonia are common, and can be seen at many centres where manurial trials are carried out continuously; most marked results have been obtained on the light sandy soil of the Wheat and Barley Plots at the Royal Agricultural Society's Experimental Farm at Woburn, and also on the Meadow Hay Plots at the Leeds University Experimental Farm at Garforth.

The effects of smoke are very noticeable in many parts of the West Riding both on soils originally "sour" and on good soils which originally contained an ample supply of calcium carbonate.

The results of "lime-requirement" determina-

TABLE 1.

*Lime-requirements of Yorkshire soils.*

Type of soil.	Characteristics of soil and crops.	Lime-requirement. CaCO <sub>3</sub> required expressed as percentage of air dried soil.
Carboniferous limestone ..	Deep soils in district of high rainfall. Sorrel and woodrush prevalent in herbage.	0.2–0.3 % CaCO <sub>3</sub>
Magnesian limestone ..	Rather shallow soils. Woodrush prevalent. Clover absent.	0.1 % CaCO <sub>3</sub>
Cbalk soils .. ..	Soil of variable depth. Coarse herbage on grass land: "finger and toe" in turnips on arable land.	0.1 % CaCO <sub>3</sub>
Coal-measures soils ..	Heavy arable soil, difficult to till. Heavy grass land, woodrush, sorrel, and bent grass very prevalent: thick "mat." Lighter soils derived from coal-measures sandstones: grass and arable land.	0.4–0.5 % CaCO <sub>3</sub> 0.5–0.6 % CaCO <sub>3</sub> 0.25–0.4 % CaCO <sub>3</sub>
Millstone grit soils ..	Good grass land, well limed. Unlimed portion of same field, poor grass with thick "mat": woodrush, sorrel, and heath bedstraw ( <i>Galium saxatile</i> ) very prevalent. Semi-moorland sheep pasture with above-mentioned weeds and bilberry.	0.06 % CaCO <sub>3</sub> 0.55 % CaCO <sub>3</sub> 0.44 % CaCO <sub>3</sub>
Moor grit soil .. ..	Reclaimed moorland in N.E. Yorkshire; very peaty and growing much ling.	0.78 % CaCO <sub>3</sub>
Peaty alluvial soils (carr land)	Pasture field rapidly deteriorating; much sorrel, thick "mat," and large barren patches. Adjacent field ploughed out, 3 out of first 4 crops failed. Rather better carr land.	0.68 % CaCO 0.77–0.90 % CaCO <sub>3</sub> . 0.30 % CaCO <sub>3</sub>
Boulder clay soils in West Riding	Grass land on which woodrush and sorrel are prevalent.	0.1 %–0.22 % CaCO <sub>3</sub>
Chalky boulder clay soils in East Riding	Usually very little indication of "sourness."	Nil–0.05 % CaCO <sub>3</sub>
Warp soils .. ..	Ditto. Ditto.	Ditto.
Light sandy drift soils in Vale of York	Chiefly arable land. Spurrey a very common weed. Leguminous crops, barley, and turnips often unsatisfactory.	0.1–0.2 % CaCO <sub>3</sub>

The above results were all obtained from samples taken on land typical of fairly large areas, under ordinary methods of cultivation, and outside smoky districts.

The "sourness" is due to the neglect of liming soils either originally deficient in lime, *e.g.*, coal-measures or millstone grit, or from which the lime has been washed out by rain, *e.g.*, limestone soils and many of the drift soils.

The inevitable loss of lime by leaching is augmented

(1) By the continuous use of acid fertilisers, particularly sulphate of ammonia.

(2) By the presence of a smoky and acid atmosphere.

tions on soils affected by smoke are given in Table 2.

In the case of the Don Valley soil which was originally well supplied with calcium carbonate, the deterioration appears to have taken place very rapidly. The calcium carbonate has been leached out to a depth of about 15 inches; the bottom 3 or 4 inches of the second 9-inch sample contained plenty of calcium carbonate.

"Sourness" is corrected by dressings of calcium oxide, hydroxide, or carbonate (or in some cases the corresponding compounds of magnesium, see later).

It is obvious that, for agricultural purposes, the presence of small quantities of impurities, provided that they are completely oxidised or in an easily oxidisable form, and are otherwise non-

\* J. Agric. Sci., 7, 75.

TABLE 2.

*Time-requirements of soils affected by smoke.*

Type of soil and locality.	Characteristics of soil and crops.	"Lime-requirements." CaCO <sub>3</sub> required, expressed as percentage on air-dried soil.		
		1st 9 ins.	2nd 9 ins.	3rd 9 ins.
River alluvium from Don Valley near Mexboro'	Dorelict grass land which 10 years ago was good pasture.	0.53 %	0.20 %	Nil.
River alluvium from Calder Valley near Dewsbury	Dorelict grass land, full of sorrel, with patches of creeping Yorkshire fog. Covered with a mat of dead and charred vegetable matter.	0.48 %	0.19 %	0.17 %
River alluvium from Calder Valley near Wakefield	Rapidly failing grass: thick "mat": much sorrel and large patches of creeping Yorkshire fog: many barren patches.	0.34 %	0.09 %	—
Coal-measures soil at Halifax	Dorelict grass land. Barren arable land, corn crop a failure: grows nothing but stunted sorrel.	0.57 % 0.62 %	— —	— —

poisonous to plants, is of little consequence so long as the price of the lime is consistent with the percentage of CaO and the cost of transport and application of the required dressing of lime.

It is, however, essential that an agricultural lime should dissolve as quickly as possible in rain or soil water so that it may come into contact with every part of the surface soil. This can only be attained by the thorough incorporation of a finely divided material with the soil.

In the case of quicklime extreme fineness is obtained by slaking, and one of the great advantages of ground quicklime is that it allows of mechanical distribution, and by slaking in the soil allows of still more thorough distribution during subsequent cultivation.

Lump lime is usually slaked in heaps in the field and the slaked lime spread with shovels; this allows of effective distribution only if the dressing exceeds 2 tons per acre.

In the case of carbonate of lime all the disintegration must take place during grinding, but when this is effectively carried out so that about 80% will pass a sieve with 100 meshes per linear inch, it gives results equal to those obtained with quicklime. This applies also to the different forms of waste carbonate of lime obtainable from some works.

The following tables show the first year's results obtained from experiments at Selby and Garforth in 1917:—

TABLE 3.

*Effects of quicklime and carbonate of lime on a light soil at Selby (initial lime-requirement 0.20% CaCO<sub>3</sub>).*

Treatment.	Weight of turnips per acre.	Percentage reduction in lime-requirement after 1 year.
No lime	4 tons 18 cwt.	0
2 tons undried ground chalk, spread with shovels	10 tons 12 cwt.	39 %
5 tons 11 cwt. ditto, ditto	10 tons 4 cwt.	39 %
2 tons dried ground chalk, mechanically distributed	11 tons 6 cwt.	52 %
1 ton ground quicklime, mechanically distributed	11 tons 11 cwt.	26 %

The undried ground chalk used was waste material from whitening manufacture. It contained about 15% moisture and was somewhat lumpy; the lumps, however, broke down easily on drying.

The dried ground chalk passed through the distributor at the rate of 10 cwts. per acre, so that the whole of the plot was passed over 4 times to complete the dressing of 2 tons per acre. An extremely uniform distribution was thus obtained.

TABLE 4.

*Effects of different dressings of quicklime and carbonate of lime on a Garforth soil (initial lime-requirement 0.3% CaCO<sub>3</sub>).*

Treatment.	Percentage reduction in lime-requirement after 1 year.
No lime	0
Half lime-requirement of quicklime	17 %
Full ditto	34 %
Double ditto	37 %
Half lime-requirement of calcium carbonate	19 %
Full ditto	36 %
Double ditto	30 %

The land was not cropped; the corresponding dressings of quicklime and carbonate of lime reduced the "sourness" to the same extent in every case.

The effect of quicklime is, in one respect, different from that of calcium carbonate; dressings of quicklime in excess of the lime-requirement of the soil produce partial sterilisation effects.\*

Calcium hydroxide, sold as hydrated lime, is occasionally obtainable. Its main advantage is that it can be stored and applied at the farmer's convenience.

There are five varieties of limestone quarried and burnt in Yorkshire, viz., Carboniferous (Mountain), Lower Magnesian, Upper Magnesian, Oolitic, and Chalk. With the exception of the Lower Magnesian limestone, there is very little to choose for agricultural purposes between the best samples of lime from the different sources.

The magnesian limestone requires special consideration not only on account of the high magnesia content of the Lower Limestone, but also because of the important position of its outcrop. It occupies a belt extending from Nottinghamshire to the Durham coast, and passing through the middle of Yorkshire, where it adjoins some of the "sourest" soils in the county.

The Upper Magnesian Limestone is fairly pure,

\* Hutchinson and MacLennan, J. Agric. Sci., 6, 302.



TABLE 5.

*Analyses of ground quicklime from Upper and Lower Magnesian Limestone.*

Upper Magnesian Limestone (Yorkshire).					Lower Magnesian Limestone (Yorkshire).				Mag. Limestone (Durham).	
	Knotting- ley.	Knotting- ley.*	Womers- ley.	Bedale.	Kiveton Park.	Kiveton Park.	Mickle- field.	Ripon.	Sunder- land.	Coxhoe.*
CaO ..	83.32	82.04	88.00	79.77	46.49	55.59	49.47	54.21	87.81	56.78
MgO ..	1.12	3.99	1.19	1.96	32.88	37.32	32.35	37.94	4.60	35.46
Fe <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> O <sub>3</sub> ..	2.73	7.38	3.91	3.56	4.88	4.42	4.58	1.46	3.28	1.20
Insoluble ..	6.74		1.15	3.35	7.21	1.26	7.67	1.29	0.85	
CO <sub>2</sub> ..	1.44	4.06	3.94	8.75	4.77	0.61	3.09	3.43	2.55	6.35
Moisture, etc.	4.60	2.53	1.81	2.61	3.77	0.80	2.84	1.67	0.91	0.21

\* Samples of lump lime.

and usually contains less than 2% of magnesia, but the Lower Limestone, separated from the Upper by a bed of red marl, although variable in composition, usually approaches a true dolomite.

In Durham the middle red marl thins out, but the marked differences in the magnesia content of the different beds of Magnesian Limestone are maintained.

That the two types of limestone yield agricultural limes of different qualities was recognised in the eighteenth century, and Tennant† published in 1799 the results of an investigation into the causes of the harm done to certain land by applications of lime with a high magnesia content.

Magnesian lime has been, and still is, used with great success on some types of soils, and such cases which have been brought to the notice of the author have usually occurred on heavy, "sour," coal-measures soils, but one marked result was also obtained on a heavy alluvial soil with a small lime-requirement, where a dressing of 2½ tons per acre of Warmsworth lime made a great improvement in the crops and in the texture of the soil.

Where complaints of the bad effects of magnesian lime have been made the soil has usually been either a light sand or a soil overlying the magnesian limestone rock.

An excess of magnesia in the soil is toxic to most cultivated plants, and such toxicity can be corrected by dressings of a pure lime.

Magnesium oxide or carbonate will improve soils where "sourness" is the factor limiting production. Equivalent dressings of CaO, MgO, CaCO<sub>3</sub>, and MgCO<sub>3</sub> were given to a "sour" Garforth soil with the results shown in Table 6.

TABLE 6.

*Comparison of effects of compounds of lime and magnesia on a "sour" soil (Garforth).*

Dressing, equal to full lime-require- ment.	Crop (control=100).	% Reduction in "sourness" of soil.
Control .. ..	100	Nil.
CaO .. ..	124	22%
CaCO <sub>3</sub> .. ..	160	22%
MgO .. ..	166	28%
MgCO <sub>3</sub> .. ..	150	22%

Magnesian lime is never preferable to a pure lime for agricultural purposes, but in view of its effectiveness in correcting "sourness" in soils, and of the convenient position of its outcrop, it is desirable that full use should be made of it for initial dressings on some of the very "sour" coal-measures and millstone grit soils of the West Riding, particularly where short transport would reduce considerably the cost of such applications.

Magnesian lime should be used only in moderate dressings (not exceeding 3 tons per acre on heavy

land) and should not be used on very light soils, soils containing already a high proportion of magnesia, nor on soils not definitely "sour."

There are three forms of lower grade lime usually available at the limestone quarries:—

(1) "Seconds" or small lime—the screenings left after the selection of the best lump lime. This material is usually sold for agricultural purposes, and like ground lime can be applied direct and allowed to slake in the soil. The composition is variable, but analyses of four samples, all from the Mountain Limestone, are given in Table 7. The quality is obviously determined mainly by the quantity of coal ashes, etc., present.

TABLE 7.

*Analyses of "seconds" or small lime.*

	%	%	%	%
CaO .. ..	88.51	75.52	60.38	59.59
MgO .. ..	1.28	1.24	—	—
Fe <sub>2</sub> O <sub>3</sub> and Al <sub>2</sub> O <sub>3</sub> ..	1.98	11.18	11.03	18.66
Insoluble .. ..	5.64	8.18	17.18	11.45
CO <sub>2</sub> .. ..	1.12	1.13	2.65	2.07
Moisture, etc. ..	1.47	2.75	8.76	8.23

(2) *Lime refuse*—usually a mixture of hydroxide and carbonate. A sample from a Lower Magnesian Limestone quarry showed on analysis CaCO<sub>3</sub> 10.15, MgCO<sub>3</sub> 11.76, Ca(OH)<sub>2</sub> 31.23, Mg(OH)<sub>2</sub> 24.82, Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub> 3.38, insoluble 7.65, moisture, etc., 10.91%.

(3) *Limestone dust*—produced during the breaking up of the rock. Such material is usually too coarse to be of much use, but if finely ground would be extremely useful to agriculture.

The production of lime for agricultural purposes has dropped considerably during the war, whereas there has been an increased demand from other essential industries. A large amount of the lime used in industries is, when "spent," merely regarded as waste material and consigned to the tip, although it is often still in a state quite suitable for application to the land.

"Spent" lime from industries usually contains some calcium hydroxide, but consists chiefly of calcium carbonate, so that thorough distribution of a finely divided material is necessary if the most effective use is to be made of such lime. The wet and sticky condition of most of the spent limes renders effective distribution impossible, but when dried most of them pulverise easily. The farmer has no conveniences for drying, and is only too frequently short of space where such lime can be stored under cover; moreover "spent" lime with a high percentage of water will not bear the cost of any appreciable transport. A second objection to the use of some forms of "spent" lime is their liability to contain substances poisonous to plants. Such poisonous substances, e.g., salts of arsenic and copper, may retain permanently their poisonous nature, and lime containing these should on no

† S. Tennant, Phil. Trans., 1799, 305—314.

account be used; but others, e.g., organic compounds from tar distillation or inorganic compounds such as sulphides, sulphites, and ferrous salts, are either lost by volatilisation or are converted into harmless compounds on exposure to air, and lime containing these may be used after some months' exposure to weather. In such cases autumn application of the lime is always desirable.

Some of the waste limes examined include:—

*Waste ground chalk from whiting manufacture.* This is usually available in the East Riding. When dry it contains about 96%  $\text{CaCO}_3$ , but owing to its being stored in the open it is liable to contain a considerable percentage of water. It has been tried experimentally at Selby (see Table III.), and has also given good results in other parts of the county.

*"Spent" lime from re-causticising soda* is in a fine powdery condition when dry, and where the amount of soda contained in it is not excessive, it is quite suitable for application to the land.

*Carbonate of lime from water-softening plant* is also in a finely divided state and has been used successfully.

*Burnt sewage* may contain 60% of finely divided calcium carbonate and has been used successfully on lawns on "sour" soils near Leeds.

*Gas lime* has been used by farmers and gardeners for such a long time that the risks attending its use are well known. It contains a number of incompletely oxidised compounds which become harmless after a winter's weathering. A number of other "spent" limes such as carbide waste (which is at present being tried experimentally at Garforth and elsewhere) should be treated in the same way and never applied to land immediately before the sowing of a crop.

*"Spent" lime from tanneries and fellmongeries* may in some instances possess all the disadvantages to which waste limes are liable. It is wet and sticky when fresh, always contains sulphides, and occasionally, where arsenic sulphide is used for unhairing, arsenic as well. The sulphides are quickly converted into harmless sulphates when the lime is spread out, but tanners' lime containing arsenic should never be used.

In order that the most efficient use may be made of waste or "spent" lime it should, whenever possible, be supplied by firms in a condition suitable for application to the land, i.e., it should be dry and powdery. It should be cheap in comparison with lime of similar quality obtained directly from the quarries, and since the percentage of  $\text{CaO}$  would never be high it should be used chiefly in the neighbourhood of the works producing it so as to eliminate as much as possible the cost of transport.

Industrial districts in Yorkshire coincide for the most part with districts where the soil is naturally "sour" and badly in need of lime, and the crops in these districts would be greatly improved if more of the waste lime from industries were applied to the soils affected. There are probably many sources of waste lime not mentioned above, and any information about such lime would be of great assistance to agriculturists at the present time.

#### DISCUSSION.

The CHAIRMAN asked, in relation to the use of lime along with sulphate of ammonia, whether there was any escape of liberated ammonia from the soil. Another question would relate to tests applied to lime in the laboratory to find out how far it was suitable for application. The value of lime for water-softening was not strictly determined by its chemical composition, and it was worth while to find out how much of the lime became available as a softening agent within a

limited time. Was a test of that kind of any use in this case? Lime from water-softening was an obvious possible source for agricultural purposes, and he asked whether it was at all the practice in the county to utilise the solid product of such plants for application to the land. He thought not. With reference to carbide waste, the acetylene given off by direct treatment with water was impure, and one would be afraid of impregnating the soil with poisonous gases; he wondered how far that was so.

Mr. F. W. RICHARDSON asked what occurred with regard to the organic acids in the soil, and as to whether the additional lime influenced the fertility of the soil through some action on the bacteria. Did the lime destroy protozoa, and thus particularly affect the development of the necessary microbes?

Mr. B. A. BURRELL remarked that many farmers did not appreciate what an unstable substance lime was, and often stored it in open sheds or exposed to the weather. He had analysed a large number of samples of ground lime. Some contained over 75% of caustic lime; in other samples which had been badly stored the quicklime was as low as 4 or 5%, the remainder being carbonate or hydroxide. Some, however, had been imperfectly burnt. As a corrector of "sourness" this did not matter, but he thought as a rule farmers used it as a soil fumigant and as an agent for liberating reserve plant food—potash and nitrogen. Owing to the quantity of sulphuric acid which was present in the air of our large towns and in the immediate vicinity, and which was mainly deposited on the land by means of rain, most of such land was "sour." At the present time lime either in lumps or ground was difficult to procure, and he had been recommending the application of basic slag at the rate of 5 cwt. per acre; when applied in the late autumn the results had in many cases been excellent. This substance when freshly prepared contained from 2–10% caustic lime, and even when long prepared had an alkalinity equivalent to about 1/4 of its weight of lime. It thus acted as a soil fumigant, a corrector of "sourness," and at the same time yielded the fertilising phosphates.

Mr. H. G. BENNETT thought that arsenical tannery waste was a good deal less common than it used to be, because certain old methods were disappearing from the tanneries. Was there any way of getting over the stickiness of the waste?

Dr. L. G. PAUL asked if there would be likely to be ill effects from the use of waste carbonate of lime from the re-causticising of caustic soda used in the process of washing crude coal-tar naphtha and similar products. These products would contain phenol, cresol, naphthalene, etc.

Mr. H. W. WARSON remarked that he had tried the use of material containing phenols, etc., as a weed-killer. It acted temporarily, but after a while weeds grew as strongly as ever, so that it would appear as if the destructive elements were neutralised or washed out by the rain.

Mr. C. P. FINN asked if waste lime from ammonia stills was likely to be of use. Much of it left the stills unchanged, though the bulk was in the form of carbonate contaminated with varying amounts of sulphides, thiocyanates, etc. He had had a quantity of sludge removed from settling ponds which had accumulated over a number of years, and had found amongst it traces of nitrates such as one would not expect to find in the liquor as it left the stills. Was it possible for a nitrifying organism to work in such a sludge and convert the ammonia present into nitrates, and might the waste be of use on the land, either in the fresh state or after being kept for some years? With regard to the effects on the soil of acid in the atmosphere he had personal experience that the



complaints by farmers of damage caused by smoke were not always justified, the failure of crops being attributable to other causes. The rainfall samples taken in the neighbourhood of a particular colliery were almost uniformly alkaline. The colliery possessed a by-product coking plant, and the waste liquor from the stills was evaporated. Presumably the small amount of ammonia found in this liquor was sufficient to neutralise any acid in the atmosphere.

Mr. W. McD. MACKAY said he presumed Dr. Hanley would not be against the use of lime from water-softening plants, but a difficulty was that it was in a sludgy condition. At present the residual carbonate of lime was usually run into a pit, but if it could be dried it would be in perfect condition for distribution on the land.

Mr. W. Lowson asked how long an interval there should be between applying dressings of lime and manure. Would it be advantageous to apply manure in the early winter, and lime shortly before planting? How long did it take the ammonia in the manure to become converted into nitrites and nitrates?

Dr. HANLEY, in reply, said that the effects of liming the soil were chemical, physical, and biological, and recent work had shown that liming on some soils promoted the development and activity of bacteria, *e.g.*, azotobacter, to a very considerable extent. Further, Hutchinson and MacLennan had obtained results with quicklime exactly similar to those obtained when a soil was partially sterilised by means of heat or antiseptics. Gas lime usually contained a number of incompletely oxidised compounds and should be applied early. It should be spread out on the surface of the soil, before digging in, for at least three months. No harm would then result. It was a fact that arsenic sulphide was rapidly disappearing from tannery waste, because only a few old-fashioned firms now adhered to old methods. Sodium sulphide and calcium chloride were not harmful if sufficient time elapsed for the oxidation of the sulphide before a crop was sown. Sodium chloride was toxic if present in excessive quantities, but there would not be sufficient present in such waste lime for an ordinary dressing to produce toxic effects. With regard to the relative costs of grinding and burning limestone, he had no doubt that any comparison would be in favour of grinding, and he rather thought therefore with Prof. Cobb that ground limestone would be produced to a greater extent as the cost of power became less. It still remained, however, to make it generally realised by farmers that ground limestone was as good as quicklime. In mixing of lime and sulphate of ammonia there was certainly an escape of ammonia, but if they were applied separately and mixed in the soil he should say there was no loss of ammonia; if mixed before applying there would be a loss. As to laboratory tests for effectiveness of lime used for water-softening, so far as he knew such tests had not been applied to lime used for agricultural purposes. In regard to the use of carbide waste, he might say that from samples he had had there was some evolution of acetylene when dilute acid was used, but there could not be very much from the use of the waste product on the soil. Experiments were being made at Garforth now. The value of the grinding of lime depended of course on the better distribution obtained. The relative values for the first year were stated to be something like twelve to one. Five cwt. of ground lime could be applied mechanically—that was with thorough distribution—whereas good distribution could not be obtained with less than 2 or 3 tons if applied with a shovel; but there had to be taken into account the nitrate effects of the

heavier dressing of lump lime in subsequent years. The small amount of lime in basic slag was apparently quite useless for correcting "sourness" in bad cases. It was very often advised for certain soils, but he had known cases in which it had not acted at all until the soil had been limed. It required heavy liming to get over the "sourness"; then the phosphate would act, and until then no addition of essential plant foods would make any difference. Naphthalene or phenolic substances were used for partial sterilisation; substances effective for that purpose destroyed the fauna, *i.e.*, the protozoa, but the spores of bacteria were probably retained in the living state and ultimately became active. The one essential in relation to the use of any waste product containing naphthalene or phenolic bodies was that the injurious effects must disappear before the cultivated crop was put on; otherwise they had the same effect on the crop as on the bacteria and protozoa of the soil. Calcium sulphate was of course of no use to get rid of sourness: it had been used with benefit in the case of alkali soils in America. As to the use of waste lime from ammonia stills, if the incorporated compounds were easily oxidisable they would become harmless after a winter's exposure. He would hardly expect that nitrating organisms would be able to work in a lime sludge of the kind spoken of by Mr. Finn—the presence of cyanides, etc., would make it very doubtful. The recovery quantitatively of the fertilising elements put on to the land in various forms was not accomplished in practice, *e.g.*, in the case of nitrogen approximately 70% of the nitrogen applied in heavy annual dressings of dung may be unaccounted for in soil, crop, or drainage water, and it was assumed to be lost as gaseous nitrogen into the atmosphere.

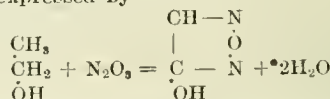
## Communication.

### FULMINATE OF MERCURY.

Prof. W. R. HODGKINSON has contributed the following remarks on the paper by G. S. Heaven on "Fulminate of Mercury" (this J., 1918, 143 r):—

In regard to the constitutional formula of mercury fulminate, a formula based on the mode of formation is just as likely to represent the true constitution as one based on the decomposition products. Mercury fulminate cannot be made without the action of nitrous acid, or the red oxides, on alcohol in the presence of the mercury salt. The interaction of pure mercury nitrate solution and alcohol does not give fulminate hot or cold, but a solution of mercury nitrate, to which alcohol has been added, treated with the red gases or a strong solution of nitrous acid, gives the fulminate, the reaction assuming the character of the usual method of making.

The action of nitrous acid, or  $\text{N}_2\text{O}_3$ , on alcohol may be expressed by



and this "fulminic" acid in presence of a mercury or silver salt forms an insoluble compound which separates from the sphere of action. Certainly nitric acid is re-formed during the precipitation of the fulminate. When fulminate is boiled with water for some time a moderate yield of mercury cyanate is obtained, especially if the mixture is well agitated during the boiling.

As to the poisonous properties of fulminate the writer of this note has certainly suffered from its effects, for although a heavy substance it "dusts" very readily when in a dry state.

## Newcastle Section.

Meeting held at Bolbee Hall, Newcastle, on  
April 24th 1918.

PROF. HENRY LOUIS IN THE CHAIR.

### THE CONSTITUTION AND INFLUENCE OF A CORED DENDRITIC STRUCTURE IN ALLOYS.

BY O. SMALLEY.

The data available on the constitution and influence of the cored dendritic structure in alloys was sparse, and had hitherto received but little attention from either the ferrous or non-ferrous metallurgist. To the former, its presence is most common in low-carbon-nickel steels, in all grades of nickel-chrome steels, and in common steels which have either been scorched during melting or incompletely de-oxidised. It may or may not be detrimental in its influence, various opinions prevailing as to its significance; it is seldom regarded with disfavour. This may be due to its almost innate connection with all commercial steels and to its persistency irrespective of subsequent treatment, mechanical or thermal, also to it being in no way associated with the carbide the physical condition and geometrical form of which are primarily responsible for the ultimate value of all steels.

To the non-ferrous metallurgist a dendritic structure indicates poor physical properties. Its presence can nearly always be accounted for and in general is due to non-homogeneity of the constituents. In such cases its removal is purely a question of subsequent heat-treatment to effect diffusion. Failing this, combined mechanical and thermal treatment is always effectual except in some special alloys where its origin is similar to that in steel; in these cases its presence persists after repeated rolling and annealing.

An example of this type is found in oxygenated nickel silver, and Thompson<sup>1</sup> states that "nickel silver exhibiting this structure is almost devoid of ductility, although the crystal grain be of normal size."

In view of these facts, before considering the actual influence of this structure on the commoner alloys, it is necessary first to consider the genesis of the structure, and to review briefly our present knowledge of the relation of composition and structure to the physical properties of metals and alloys.

#### *Genesis of micro-structure.*

In all alloys the micro-structure has its origin in the freezing-out from the molten state, hence for the purpose of this investigation metals and alloys will be subdivided into two classes:—I. Pure metals and those alloys which freeze at one specific temperature; II. Alloys which freeze through a range of temperatures. This classification enables a clear conception to be framed as to the nature of the physical changes that accompany alloys during solidification, and it also shows at a glance whether an alloy may, or may not, be subjected to the influence of a cored dendritic structure.

The origin of each crystal grain of alloys of class I. is its centre, from whence it develops in a regular fashion determined by the crystallographic system to which it belongs; the last particle of

metal to freeze is at the same temperature as the first particle, so that each crystal grain in such alloys is homogeneous throughout.

Where an alloy freezes through a range of temperatures, a different type of crystal growth results, and it is characteristic of solid solution crystal grains of an isomorphous mixture of metals to develop faster in one direction than in another, resulting in the formation of unbalanced crystallites. This is illustrated by photomicrograph No. 20, which shows the formation of the primary and secondary axes on incipient solidification and the subsequent emanation of other axes, which take a higher order of symmetry; these continually grow into, and are fed by the residual or mother liquor until solidification is complete. In the alloy cited, which crystallises in the cubic system, these skeletal arms are shown to have developed along the principal axes of the cube. Such a structure at once indicates that sufficient time has not been allowed during solidification for the adjustment of the homogeneity which theory demands, so the intensity of the heterogeneity or, as it is more commonly known, "coring" of each crystal grain of alloys in this class will be dependent upon the alloying properties of the metals concerned, the range of temperatures through which the alloy freezes, and the rate of cooling during solidification.

#### *The relation of composition and structure to the physical properties.*

Theoretically the physical properties of all metals and alloys are governed by their composition and structure. Unfortunately practice does not always conform to theory, and many external influences

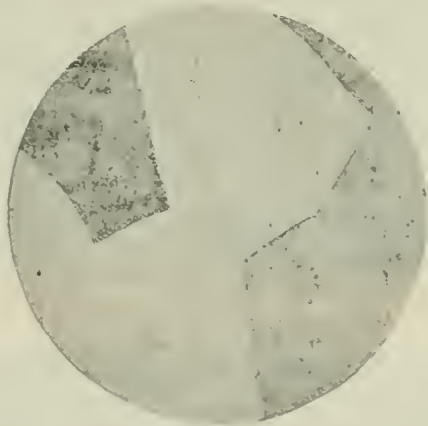


FIG. 1.

must be taken into consideration, viz., occluded gases, volume changes on solidification, temperature, impurities, and other external agencies, each modifying theory to a degree totally dependent upon the constitution and nature of the metal or alloy. This is augmented in those metals and alloys which undergo allotropic and isomeric changes respectively, and in many instances by physical changes which are not accompanied by any apparent structural alteration. An example of this type is found in the immediate softening and subsequent hardening of aluminium and aluminium alloys containing small quantities of magnesium, when quenched in water from a temperature between 400° C. and 500° C.

It is, then, clear at the outset this investigation is limited to those alloys of which we have a more precise knowledge.

<sup>1</sup> Chem. Soc. Trans. 1914, 105.



TABLE 1.

*Effect of forging on the properties of "β" brass, when the crystal grain is reduced and when it is unchanged.*

Mark	Treatment.	No. of crystal grains per sq. cm.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	Elongation, % on 2 in.	Reduction of area, %.	Brinell hardness no.	Alternating impact-no.
"A"	"As cast," 3 in. sq. ingot.	152	20.50	38.60	19.00	21.50	159	23
	Forged in one heating to 1½ in. sq. bar. ... ..	2535	18.90	43.00	29.00	33.50	159	27
"A" modified	"As cast," 3 in. sq. ingot.	2500	18.60	44.50	26.00	27.60	159	19
	Forged in one heating to 1½ in. sq. bar. ... ..	2565	19.80	44.90	26.00	29.00	159	26

## CLASS I. ALLOYS.

Of the alloys in this class are those of eutectic composition, intermetallic compounds, and some special alloys having an exceedingly small freezing range, *e.g.*, aluminium-bronzes and high-tenacity brasses.

Of these only high-tenacity brasses will be considered, and only for the purpose of bringing out clearly the influence of a cored dendritic structure in alloys of class II.; and to show that, given an isomorphic alloy of homogeneous texture, strength and ductility depend upon the size, orientation, and external form of the crystal grains, so that if suitably cast, neither mechanical nor thermal treatment is beneficial.

Full data concerning the treatment and test results obtained in practice of two such alloys are included in Table I. These figures call for no special comment, being self-explanatory. The microstructure of each alloy is similar, with the exception of the size of the crystal grains, and is illustrated by fig. 1.

The former was made to the following composition:—Cu 94, Sn 5, P 1%. Twelve bars were chill-cast in pairs 9 in. × 1½ in. × 1½ in. from the same melt at varying temperatures, ranging from 1112° C. to 1040° C. The temperature was measured by means of a platinum-platinum rhodium thermocouple protected by a fused silica sheath. The charge was well stirred before reaching each pre-designed successive temperature, obtained by allowing the metal to cool, after which each successive pair of bars was cast.

Tensile and Brinell hardness tests were made on each bar and are included, together with the casting temperatures, in Table 2.

Microstructures, representing casts 1, 3, and 6 respectively, are illustrated by figs. 2, 3, and 4, at a magnification of 50 times, showing a coarse complex dendritic structure to exist in cast no. 1; a similar structure in cast no. 3, with the exception of the absence of the dendritic pattern; and no. 6 to exhibit an adumbrant cellular structure.

The effect of the dendritic structure is thus

TABLE 2.

*Influence of varying casting temperature on the properties of phosphor-bronze castings poured from the same melt.*

Cast no.	Composition	Casting temperature.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	Elongation % on 2 in.	Reduction of area, %.	Fracture.	Brinell hardness no.
1	Cu 93.90 Sn 5.25 P 0.85	1112° C.	10.40	23.50	23.50	4.50	Earthy dendritic. Exhibiting slight oxide inclusions	73
2		1085° C.	10.90	24.60	28.00	6.20	As cast no. 1, but free from defects	75
3		1075° C.	10.90	25.30	30.00	6.60	Fine, earthy, almost free from crystallinity	78
4		1050° C.	10.40	24.20	22.50	5.00	Earthy with a small copper-coloured cavity in the centre	78
5		1044° C.	10.00	20.40	12.50	3.10	As cast no. 4, but more defective	74
6	Cu 93.48 Sn 5.72 P 0.80	1040° C.	10.00	18.80	8.00	2.70	Earthy, exhibiting copper-coloured patches in which cavities are contained	79

## CLASS II. ALLOYS.

Amongst the alloys examined of this class are tin-bronzes, brasses, light alloys, and steels.

*Tin-bronzes.* Of the innumerable range of alloys offered by this series of bronzes, two of the most important will be considered, namely, ordinary phosphor-bronze and Admiralty gun metal.

clearly shown. It is, nevertheless, palpable that although casting too hot is detrimental, through permitting the formation of a highly crystalline structure, it is equally detrimental to cast at any temperature below that which just suffices to suppress its formation; the poor physical properties that accompany the low casting temperature are



FIG. 2.  
Casting Temperature 1112° C.

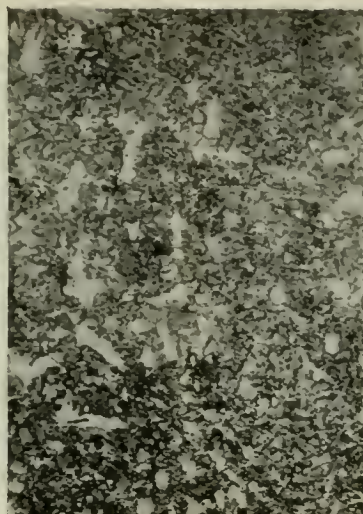


FIG. 3.  
Casting Temperature 1075° C.

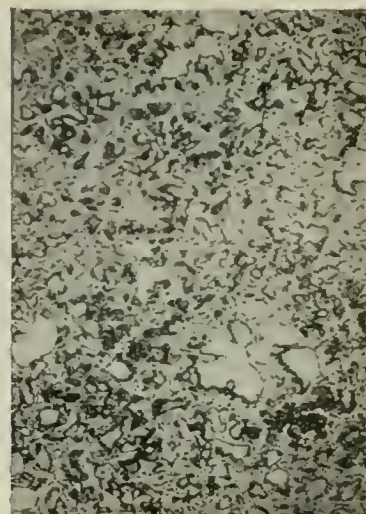


FIG. 4.  
Casting Temperature 1040° C.

CASTING TEMPERATURE EXPERIMENTS WITH PHOSPHOT-BRONZE—CHILL CASTING.

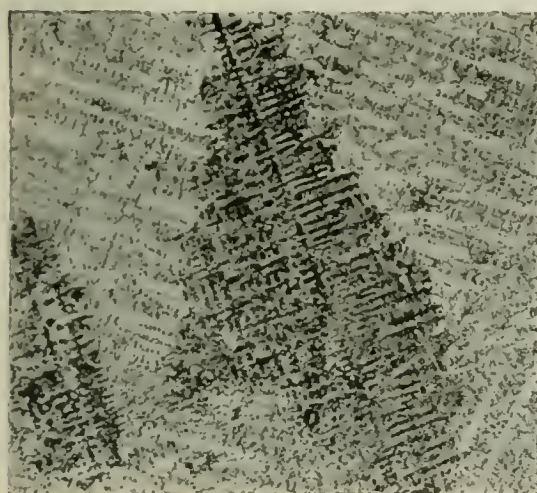


FIG. 5.  
Chill Casting.

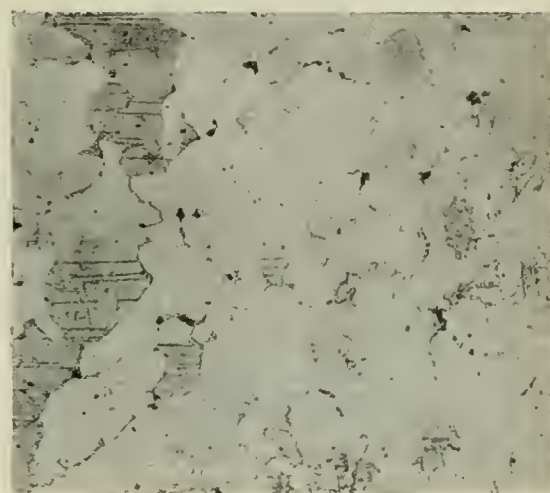


FIG. 6.  
Chill Casting after Annealing 725° C.

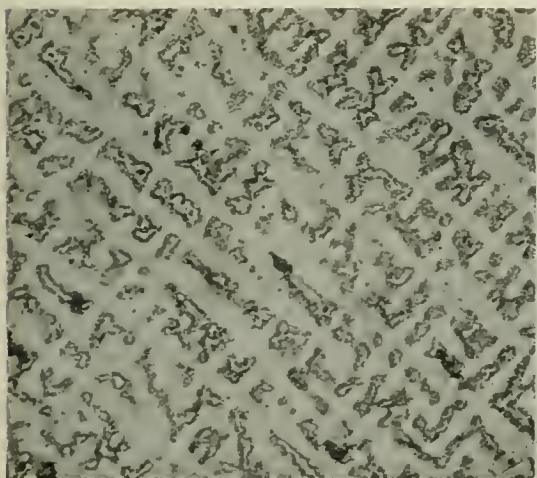


FIG. 7.  
Sand Casting.

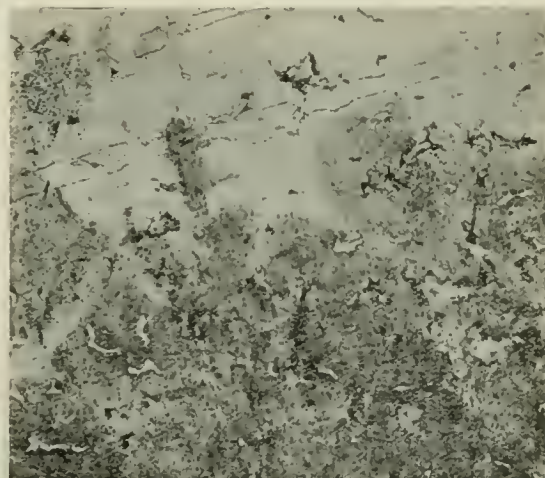


FIG. 8.  
Sand Casting after Annealing 725° C.

EFFECT OF HEAT TREATMENT ON ADMIRALTY GUN METAL.



not, however, attributable to structural so much as to mechanical defects, which are manifest in the form of entrapped oxide, liquation spots, and cold cracks due to the increased viscosity of the metal.

It may then be concluded that (1) the presence of a cored dendritic structure is embrittling in its influence on phosphor-bronze; (2) the correct casting temperature depends upon the size and shape of the casting to be made, and for general practice the best results will be obtained between 1100°—1075° C.

*Admiralty gun metal.* This alloy, while permitting a wider range of casting temperature, is just as susceptible to the formation and the objectionable influence of a dendritic structure.

C. P. Karr and H. S. Rawdon<sup>2</sup> in an investigation on this alloy found for ordinary sand and chill castings the range of temperature permissible lies between 1120° and 1270° C. This is to some extent supported by our own practice, provided the alloy is subsequently treated to remove the dendritic cores formed at the higher temperatures. Failing this, the best results in the case of chill castings are obtained from 1100° to 1170° C., and in sand castings from 1125° to 1200° C.

That the absence of a dendritic structure is of high importance and of far greater moment than a fine crystal grain may be gleaned from the following experiment:—

Two 25-lb. casts of this alloy were made from 50% virgin metal and 50% previously made alloy, under identical conditions. Two ingots were cast

matrix of a cored solid solution of copper-tin-zinc, in which are embedded in the interstices of the primary skeletal crystallites numerous granules of a hard light-coloured rich-tin compound.

Photomicrographs figs. 6 and 8 show annealing to have almost removed this composite dendritic matrix, there existing now a simple polyhedral crystalline structure—common to that of pure metals—except for the presence of some of the hard copper-tin compound, which is still present, but in reduced quantities.

It is clear from these that had the annealing been such as to effect complete solution of this hard, brittle compound into the now homogeneous  $\alpha$ -matrix, better results would have been obtained on test, its presence being conducive to both inter-crystalline and intergranular brittleness.

H. S. Primrose<sup>3</sup> states that "for general work, such homogeneity is obtained by annealing this alloy at a temperature of 700° C. for 30 minutes."

J. C. Longbottom and A. Campion<sup>4</sup> find this temperature to be injurious to Admiralty gun metal castings, and that homogeneity is best obtained by a long annealing at 250° C.

The present experiments, however, indicate that such general statements cannot be made, and experimental work conducted on this class of alloy at different times proves that the treatment necessary to obtain the best results is determined by the size and condition of the copper-tin compound, and in the case of large sand castings its removal is not technically practicable.

TABLE 3.

*Effect of heat treatment on the properties of Admiralty gun metal.*

Mode of casting.	Casting temperature, °C.	Subsequent treatment.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	Elongation, % on 2 in.	Reduction of area, %	Fracture.	Brinell hardness no.	Number of crystal grains per sq. cm.
Chill ..	1175	None	10.90	11.20	Nil	Nil	Uniform, exhibiting dendritic markings	85	2200
Chill ..	1175	Annealed 725° C. for 45 mins.	9.50	15.30	12.00	11.00	Uniform, granular	55	1760
Sand ..	1175	None.	10.00	14.30	6.00	7.00	Coarse, exhibiting a marked dendritic pattern	78	69
Sand ..	1175	Annealed 725° C. for 45 mins.	7.80	14.20	16.00	26.00	Coarse, presenting an adumbrant dendritic pattern.	55	70

into chill moulds, and two into dry sand moulds. Casting in each case was performed at a temperature of 1175° C., the metal reaching a maximum temperature of 1270° C. in the furnace. Tensile and Brinell hardness tests were made in duplicate on one of each of the chill and sand cast ingots before and after heat-treatment. The heat-treatment consisted of an annealing at 725° C., during 45 minutes, cooling off in air. The results of the tests are embodied in Table No. 3.

The nature of the structural changes responsible for the improvements in the physical properties wrought by heat-treatment in both the chill and sand cast specimens are shown by figs. 5, 6, 7, and 8, each specimen in the "cast" condition displaying a structure of a complicated nature, the chill cast specimen being somewhat similar to that of the phosphor-bronze previously considered.

Both the sand and the chill cast specimens of this mixture consist of a complex definite dendritic

*Brasses.* An examination of the constitutional diagram of the copper-zinc series of alloys shows all " $\alpha$ ," and to some extent " $\alpha\beta$ " brasses, to enter into the class of alloys under consideration. The principal value of  $\alpha$ -brasses being their malleability and ornamental appearance, the effect of a dendritic structure, although always manifest—due to the high casting temperature necessary—is of little moment. That its presence can be shown to exercise some influence, in spite of the native softness of such brasses, however, is of the greatest importance in its direct correlation to other alloys where combined tenacity and ductility are the essentials. Unfortunately, the investigation could not be conducted by simply modifying the casting temperature, as in the case of the tin-bronze series of alloys, for if cast too hot they are accompanied by numerous defects in the form of blowholes,

<sup>3</sup> The Metal Industry, Vol. 10, No. 24.

<sup>4</sup> Trans. Inst. Eng. & Shipbuilders in Scotland, 1914 (Newcastle meeting).

<sup>2</sup> U.S. Bureau of Standards, Bulletin No. 59.

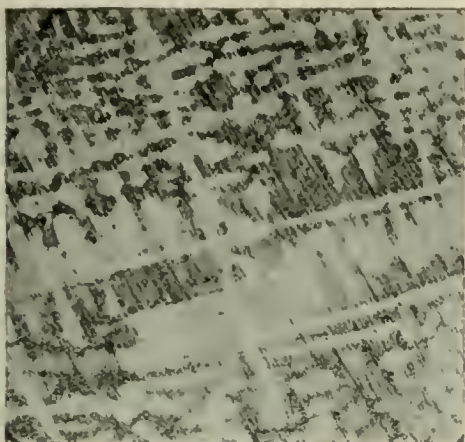


FIG. 9.

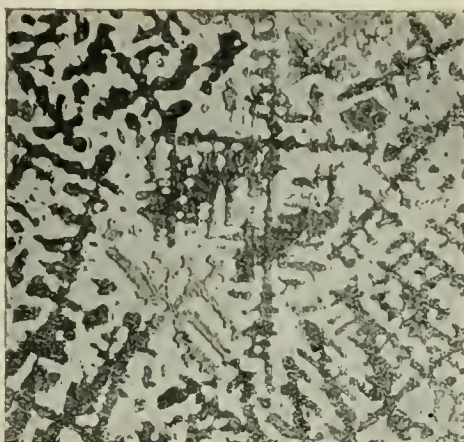


FIG. 10.

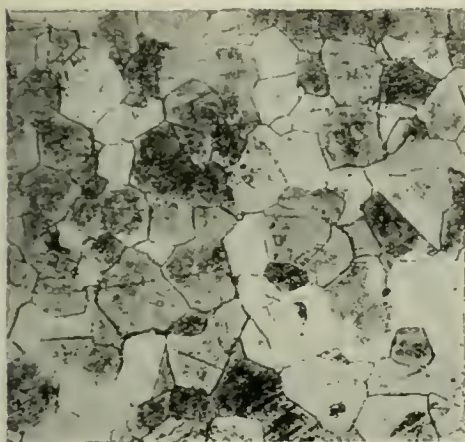


FIG. 11.

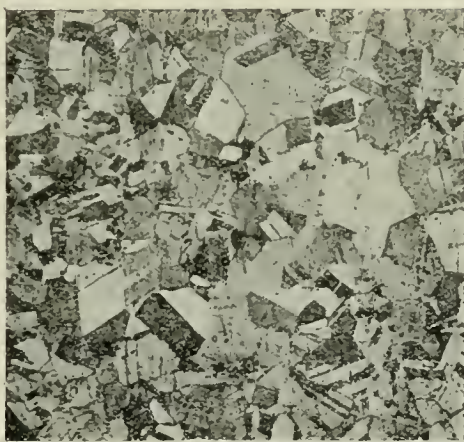


FIG. 12.

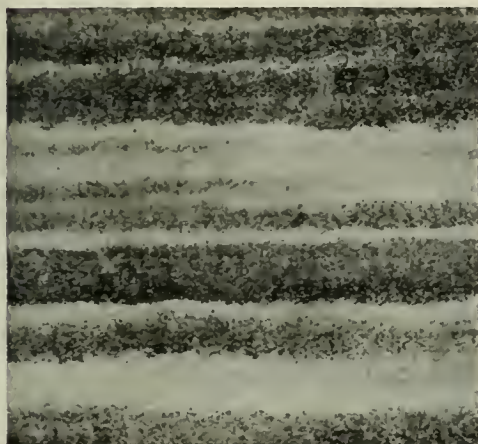


FIG. 13.

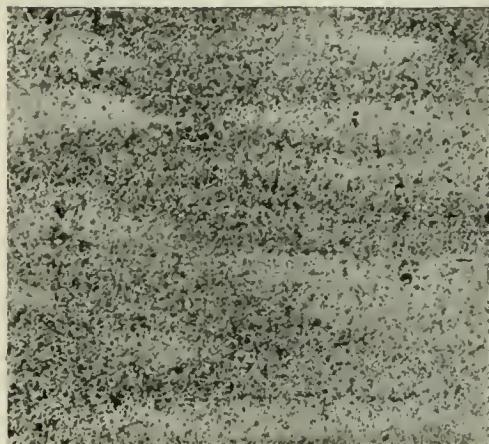


FIG. 14.



oxide contamination, and hot cracks; or if cast too cold, "spilliness," entangled oxide, and foreign matter.

The difficulty was eventually overcome by taking three casts of 70:30 brass made under identical conditions, cast from the same temperature into slabs 4 ft.  $\times$  1 ft.  $\times$  1 in., and having everything in common with the exception of the presence of 0.20% of arsenic added to one, and 1.00% of iron to another. The lower half of each slab was used for the requisite physical tests in the "cast" condition. Similar tests were made from the upper half of each, after the cast cored structure had

(2) Its formation is augmented by those impurities which are soluble in the molten alloy and prevented by those impurities which are insoluble.

*$\alpha\beta$ -Brasses.* These will only be considered briefly, the short range of temperatures through which they freeze being such as almost to prevent any difference of composition between the first and last portions to solidify. This is particularly interesting because this group of alloys forms the bedrock on which is built all high-tensile brass, and proves that their physical properties will be purely a question of their polymorphic nature and fineness of the crystal grain.

TABLE 4.

*Influence of impurities on the properties of 70:30 brass before and after the removal of the cast structure.*

Mark.	Composition.				Physical condition.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	Elongation, % on 2 in.	Reduction of area, %	Brinell hardness no.
	Cu	Zn	As	Fe						
1	70.00	30.00	—	—	"As cast" .. Cold-worked and annealed	6.50 8.00	16.70 21.50	58.00 68.00	48.20 62.00	55 55
2	69.57	30.00	0.20	—	"As cast" .. Cold-rolled and annealed	6.20 8.00	15.30 22.50	39.50 66.50	30.00 64.00	55 55
3	69.50	29.50	Nil	1.00	"As cast" .. Cold-rolled and annealed	10.70 11.00	24.00 26.50	50.00 54.00	59.30 67.00	72 76

been removed and the brass restored to its original hardness. This latter was achieved by cold-rolling to 0.45 inch in five passes, with one intervening anneal and a final anneal, the temperature of which was predetermined by experiment.

The test results are given in Table 4, and show that in each sample the improvement wrought by the removal of the cast structure is different, being a maximum in No. 2 and a minimum in No. 3. The explanation of this is not far to seek. In No. 1, a cored dendritic structure is replaced by a homogeneous one composed of polygonal twin-crystals (compare figs. 9 and 12). In No. 2, a similar change is evinced, but the dendritic structure is more marked in the cast sample (compare figs. 10 and 12). In No. 3, a minimum structural change has resulted (see figs. 11 and 12), the cast sample of this brass being shown to exhibit no trace of a dendritic pattern.

That the added impurities should have such diverse effects on 70:30 brass when cast from the same temperature—in the one case accentuating, and in the other preventing, the formation of a dendritic structure—was to be expected on consideration of their nature, arsenic being soluble in brass when molten, forming an arsenide of zinc and copper; and iron forming an insoluble copper-iron compound.

The influence of the former, then, is analogous to that of phosphorus in steel, which forms a phosphide of iron and is generally responsible for the well-known "ghost" marks or heterogeneity common to ordinary steels; while the latter, which is present in the form of finely disseminated, insoluble particles, forms the nucleus for the germination of the crystal grains, so that on solidification crystal growth now tends to proceed radially from each nucleus, resulting in the gross cored dendritic structure being replaced by a fine grained texture, each grain having an onion-like type of coring, i.e., it is heterogeneous in the form of concentric layers.

It may then be concluded,

(1) A dendritic structure, although of little moment in " $\alpha$ " brasses, is distinctly embrittling.

*Light alloys.* In the course of recent research on this series of alloys, it has been impossible to correlate structure with physical properties. Moreover, a metallographic study of aluminium and its alloys may be said to have only just begun. In one instance of the manufacture of some small articles from an aluminium-copper-zinc alloy, a great variation was found on tensile test. The microscope showed each sample to be equally sound, but on etching, the brittle castings presented a cored dendritic structure. The test results obtained are given in Table 5. In spite of the evidence of this structure being responsible for these results, they must be accepted with some reserve, there being many physical changes accompanying this class of alloys which are at present little understood.

TABLE 5.

Sample.	Maximum stress per sq. in.	Elongation, % on 2 in.
Without dendritic structure ..	12 tons	15.00
With dendritic structure ..	7 tons	2.00

*Steel.* The most noteworthy feature of the non-ferrous alloys just considered is that the harder the alloy, the more susceptible is it to the embrittling influence of a cored dendritic structure. It then follows that its absence in steel is of paramount importance, particularly with regard to special steels the principal qualities of which are combined strength and ductility. This is disconcerting, as at the present time the nature and constitution of heterogeneity in steel are conjectural. Its origin cannot be traced directly to either incomplete diffusion of added elements, or to the presence of the commoner impurities; although in every instance it is directly connected with the freezing out of the steel from the molten condition, and is the progenitor of the ghost-markings manifest in all "worked" steel.

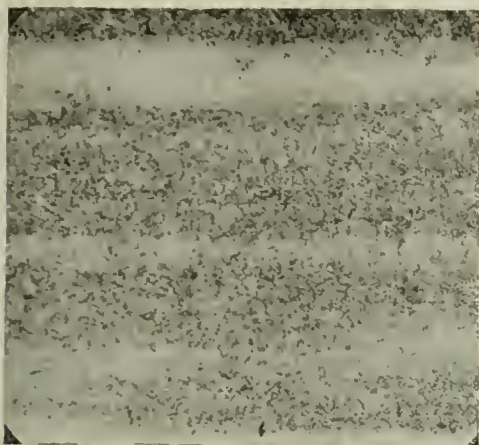


FIG. 15.

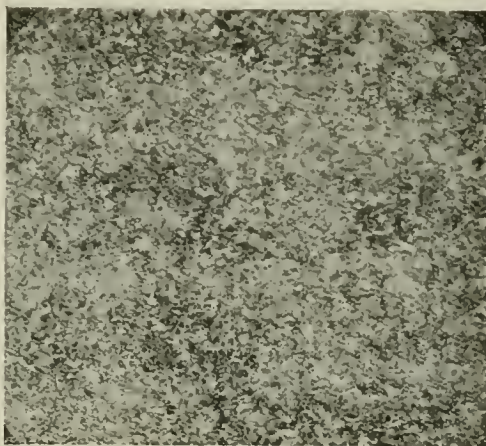


FIG. 16.

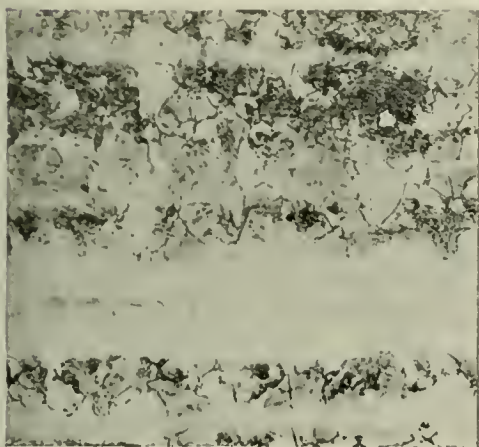


FIG. 17.

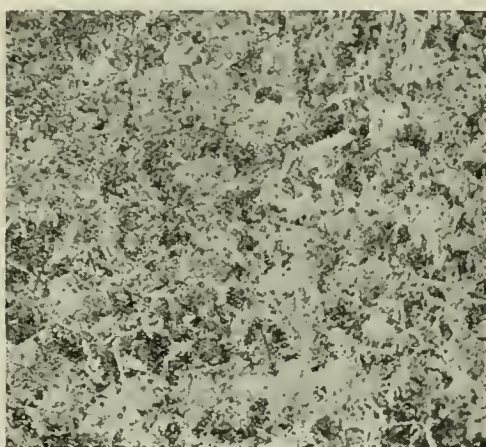


FIG. 18.

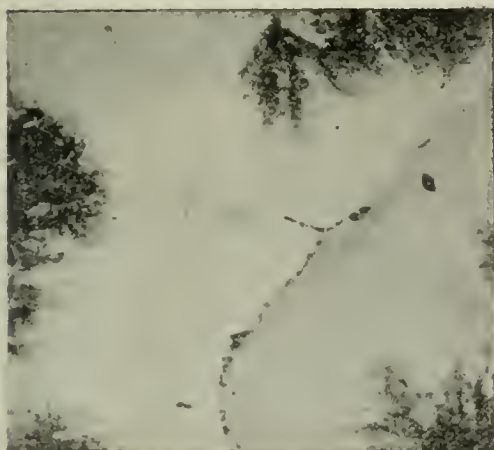


FIG. 19.



FIG. 20.



The conflicting views on ghosts that have been expressed by various authors, notably Arnold,<sup>6</sup> Stead,<sup>6</sup> le Chatelier and Lemoine,<sup>7</sup> Charpy and Bonnerot,<sup>8</sup> Hatfield,<sup>9</sup> and Rosenhain and Haughton,<sup>10</sup> show that ghosts in steel are not only little understood, but that they are only indirectly connected with the commoner foreign elements, such as phosphorus, silicon, sulphur, and manganese. The variations found by each of the forementioned investigators when working with the same reagent, or with reagents having similar characteristics, indicate their presence to be attributable to some inherent defect common to all steels in a greater or lesser degree when made by an oxidising process; this defect has not received due consideration, and in consequence each claims to have discovered something new. This is supported by the fact of copper-depositing etching reagents being found to be equally sensitive to oxides, silicates, sulphides, gas fissures, phosphide, and even mechanical defects, and to reveal heterogeneity equally well in high or low grade steels, although the carbide be homogeneous. This is exemplified by figs. 13 to 18. Figs. 13 and 14 were taken from the same section of nickel-chrome steel (C 0.45, P 0.02, S 0.021, Si 0.1, Mn 0.06%) cast from a "wild" heat, and etched with nitric acid and cupric chloride respectively.

Figs. 15 and 16 represent a special high grade nickel steel containing C 0.40, Si 0.06, Mn 0.35, S 0.022, P 0.016, Ni 3.50%, when etched with picric acid and cupric chloride respectively.

Figs. 17 and 18, representing a poor quality carbon steel containing C 0.65, Si 0.12, Mn 1.10, S 0.034, P 0.038%, etc., were etched with the same reagents as those shown in figs. 15 and 16 respectively.

These illustrations show the carbide in each sample to be uniformly distributed and the ghosts to have no relation to the heterogeneity of the essential constituents or to phosphorus, sulphur, manganese, or silicon, although, as fig. 18 indicates, their presence is intensified by such impurities.

#### Impurities.

In view of the changes which steel undergoes in the process of solidification, impurities will influence it to a degree depending upon their nature and quantity. As regards common steels the elements to be considered which give rise to these impurities are carbon, aluminium, silicon, manganese, sulphur, occluded gases, and phosphorus; and in special steels, similar elements, together with the additional elements which so classify it.

**Carbon**, as has been shown in the three steels cited as exhibiting ghosts, was homogeneously distributed, so does not call for consideration.

**Aluminium, silicon, and manganese** may be considered together, each being used as a deoxidant, although their actions are somewhat different. They are used singly and conjointly, but whether they remove the oxygen or render it soluble in the steel as gas or oxide is not known. There is, however, every reason to believe that both silicon and manganese perform the latter function, so are preferable to aluminium so far as the casting qualities of the steel and the production of a sound ingot are concerned, but, it is generally believed, to the detriment of the steel.

Incidentally, both silicon and manganese are also beneficial in removing the injurious influence of sulphur when present as iron sulphide, converting it into a comparatively inert sulphide.

As far as gaseous impurities are concerned, little

can be said. Oxygen has been mentioned, but there still remain to be considered hydrogen, carbon monoxide, and nitrogen. Their extreme solubility in the molten steel is well known, but to what extent they are soluble, in what form, and how they influence the finished product, is unknown.

In common steel, then, we may have

(1) Aluminium present in solid solution or as an insoluble oxide.

(2) Silicon and manganese, in solid solution, as manganese silicate, or as a complex insoluble sulphide.

(3) Phosphorus present in solid solution as Fe<sub>3</sub>P.

#### Relation of impurities to ghosts.

Impurities in steel then are of three types and may be classified as follows: (a) those insoluble in the molten metal; (b) those separating out as definite constituents during solidification; and (c) those entering into solid solution. Hence it is quite evident that the mode of crystallisation of steel from the liquid state cannot be followed so readily as with non-ferrous metals and must depend upon the type of impurity most manifest.

Of the insoluble impurities, sulphides and silicates, being in a viscous condition—their melting point being lower than that of steel—are entrapped in the meshes of the branches of the primary crystallites, where they freeze, taking a skeleton form of the primary dendrites. If present in abnormal amounts, together with entangled slag, free alumina, extraneous refractory material, etc., they will, of course, tend to act as suggested by Arnold.

Whether impurities of class (b)—which separate from solution during solidification—are present or not, will depend upon the condition of the metal in the ladle; if scorched or oxygenated, it is quite conceivable that oxide may be rejected on freezing and be present in the finished steel in mechanical admixture at the junctions of adjacent crystal grains. An example of this type is illustrated by photomicrograph fig. 19. In consequence there is every reason to believe that a steel correctly deoxidised, so far as the production of a sound ingot is concerned, may still contain a considerable amount of oxygen in solid solution as oxide, the solubility being increased by the presence of such elements as nickel and chromium. If this be so, the influence of this oxide will be similar to those impurities of class (c), which exist in solid solution, and intensify the production of a cored dendritic structure.

The influence of phosphorus in this direction has been shown by Stead (*loc. cit.*), and the fact that an oxygenated steel free from phosphorus, silicon, manganese, or the other common impurities, exhibits an exactly similar effect, points to the presence of such dissolved oxide which, due to the mode of crystallising of the steel from the liquid state, is concentrated in the interstices of the primary crystallites.

The relative positions occupied by impurities in steel must then depend upon numerous factors; if a common grade steel containing both soluble and insoluble impurities, cast from the correct temperature, they will be contained in and between contiguous crystal grains; if the insoluble impurities be in excess, then the soluble impurities will be more concentrated in the external zones of each crystal grain. If the soluble impurities be predominant, the insoluble impurities will be forced between the primary skeletal arms and subsequently solidify, *in situ*, together with the residual material containing the bulk of the soluble impurities.

In special steel containing soluble impurities, the extent of coring will depend upon the quantity of the impurities present, together with alloying properties of the added element or elements.

<sup>6</sup> J. Inst. Mech. Eng., 1915, Vol. I.

<sup>6</sup> J. Iron & Steel Inst., 1915, Vol. I.

<sup>7</sup> Comptes rend., 1915, p. 161.

<sup>8</sup> Comptes rend., 1917, p. 165.

<sup>9</sup> J. Iron & Steel Inst., 1915, Vol. II.

<sup>10</sup> J. Iron & Steel Inst., 1914, Vol. I.

*Influence of a cored dendritic structure on the physical properties of steel.*

An ingot of nickel chrome steel of the composition C 0.50, Si 0.10, Mn 0.07, S 0.025, P 0.03, Ni 2.00, Cr 2.00%, was used for this investigation; it was known to have been cast too hot, and found to have a marked dendritic structure.

Static and dynamic stress tests were taken from one half of the base of the ingot, in the "cast" condition, after annealing, and after hardening. Similar tests were made from the other half in the annealed and hardened conditions, after reducing by "forging" to one half the original cross sectional area and also after reducing to one-eleventh the original cross sectional area.

perature of 18° C., and subsequently tempered in oil at 165° C., for 1 hour. This latter treatment was performed as a safeguard against cracking on grinding. The test results are detailed in Table 6, and clearly show the improvement effected by "forging" on this steel, in both the annealed and hardened condition.

The structural changes responsible for this improvement are depicted by photomicrographs figs. 20, 21, and 22, representing samples nos. 1, 1F, and 1FF respectively. In either the annealed or hardened condition, delineating the progressive deformation of the primary dendrites by mechanical treatment.

That neither fineness of the crystal grain nor homogeneity of the carbides has any influence is

TABLE 6.

*Effect of forging on the physical properties of nickel chrome steel in the annealed and hardened condition.*

Mark.	Physical condition.	Treatment.	Yield point, tons per sq. in.	Maximum stress, tons per sq. in.	Elongation, % on 2 in.	Reduction of area, %	Fracture.	Brinell hardness no.	Calculated tenacity.	Alternating impact no.	Izod, impact ft. lb. absorbed.
	"As cast."	None.	28.80	30.50	1.50	1.00	Coarse crystalline, exhibiting brilliant crystal facets.	223	55	1	4
1.	"As cast."	Annealed 900° C. 1 hr. Cooled in furnace. Reannealed 790° C. 1 hr. Cooled in furnace.	31.50	50.00	13.50	28.00	Coarse crystalline, but of dark velvety appearance.	207	51	107	26
	"As cast."	Annealed as above and oil-hardened.	51.20	54.20	Nil.	Nil.	Fine, amorphous, exhibiting an abundant dendritic pattern.	578	140	1	1½
	"As forged," reduced to ½ the original cross sectional area.	Annealed 900° C. 1 hr. Cooled in furnace. Reannealed 790° C. 1 hr. Cooled in furnace.	31.80	50.00	23.00	55.00	Grey velvety. Cup and cone.	207	51	173	53
1F.	"As forged," reduced to ½ the original cross sectional area.	Annealed as above and oil-hardened.	80.00	80.00	0.30	Nil.	Fine, amorphous.	578	140	5	3½
	"As forged," reduced to ¼th the original cross sectional area.	Annealed 900° C. 1 hr. Cooled in furnace. Reannealed 790° C. 1 hr. Cooled in furnace.	30.00	49.60	26.00	63.00	Grey velvety. Cup and cone.	207	51	201	72
1FF.	"As forged," reduced to ¼th the original cross sectional area.	Annealed as above and oil-hardened.	124.00	124.00	1.00	Nil.	Fine, amorphous.	578	140	7	2½

Forging was performed at 1050° C., one heating sufficing for the former reduction, and two heatings for the latter. Each sample was given a double anneal, performed at 900° C. and at 790° C., under identical conditions. The efficacy of this treatment is made apparent by the similarity of the tenacity and hardness figures obtained from each sample, the former being 50 tons per sq. in., the latter 207.

The remaining portions from each of the annealed samples were machined to the same dimensions for hardening purposes, 0.01 in. being allowed for removal by grinding after quenching.

Each sample was heated, in an electric tube furnace, to a temperature of 80° C. ± 2° C., above the calescence point, quenched in oil having a tem-

peratures of 18° C., and subsequently tempered in oil at 165° C., for 1 hour. This latter treatment was performed as a safeguard against cracking on grinding. The test results are detailed in Table 6, and clearly show the improvement effected by "forging" on this steel, in both the annealed and hardened condition.

It may then conclusively be stated that "cored" dendrites present in nickel chrome steel give rise to cleavage brittleness, and that "forging," although not affecting diffusion, causes an interpenetration of the branches, and if carried far enough removes its embrittling propensities, tending to give to the steel a fibre, thereby strengthening it against impact shock applied at right-angles to the direction taken by it, but weakening it if applied in a parallel direction.



This latter is substantiated by single blow impact tests which were made on longitudinal and transverse test-pieces cut from each annealed sample, the results of which are given below:—

*Impact figures in ft.-lb.*

		Longitudinal.	Transverse.
1A	..	26	24
1FA ..	..	53	37
1FFA ..	..	72	26

Possibly the most striking feature of these results is the considerable improvement effected by forging in the reduction of area and the shock-resisting properties when in the annealed condition. The greatest influence of the removal of the dendritic structure, however, seems to be when the steel is hardened, and although samples 1 and 1FF have everything in common with the exception of the crystalline formation of the primary cores, these show a difference in the tensile tests of 70 tons per sq. in.

*Conclusions.*

(1) Analogies exist between alloys with regard to the origin, constitution, and objectionable influence of a cored dendritic structure.

(2) Soluble impurities give rise in all alloys to a cored structure which is peculiarly persistent and detrimental to their physical strength, despite subsequent treatment.

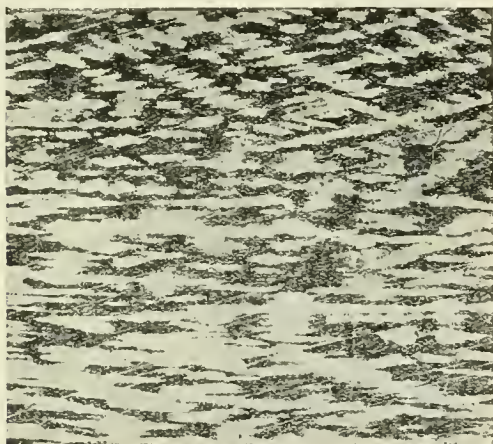


FIG. 21.

(3) The geometrical relationship between a cored structure and the crystallographic system to which the alloy belongs is determined by the nature of the alloy, the casting temperature and rate of cooling, and the impurities present.

(4) Ghosts are universal in all steels made by an "oxidising process." They may exist in the absence of solid foreign matter, but the converse does not hold.

(5) Ghosts are an effect of soluble impurities, and the laminated form usually exhibited by "worked" steel is a result of the attenuation of the cored primary crystallites. They are unaffected by any kind of heat-treatment, normal or abnormal; in consequence, if the original dimension of the primary crystallites be known, the degree of mechanical work applied to steel is readily revealed by the aid of a copper-depositing etching reagent.

(6) Many structural anomalies exist in steel, and the copper-depositing reagent is a valuable adjunct to the metallographist if skilfully used.

In closing, I wish to thank Colonel Hadcock for permission to draw upon the data accumulated in the laboratory of Messrs. Sir W. G. Armstrong, Whitworth & Co., Ltd., and to recognise the valu-

able assistance rendered by Mr. R. L. G. Johnston and Mr. R. N. Richardson in the preparation of this paper.

*DISCUSSION.*

Prof. LOUIS stated that Mr. Smalley had tackled one of the most difficult and most complicated subjects in the range of metallurgy and one on which very many and very widely-divergent opinions were held. It would be difficult to select a better subject for discussion.

Mr. H. DUNFORD SMITH said that some of the figures for maximum stresses given for Admiralty gun metal and for phosphor bronze castings were so very much below what one usually obtained in practice that it made one think there might be some cause for such low figures. He wondered whether some oxide had been occluded in the Admiralty gun metal.

Mr. G. DUNCAN asked how the samples were taken for the estimation of phosphorus and manganese in steel. In making a eutectic mixture of antimony and lead, he had found trouble due to segregation. When a small bar was cast into a small thin mould, there were very marked differences when the sample was sawn through the centre or diagonally.

Professor HENRY LOUIS remarked that dendritic crystallisation was not confined to metals. For a

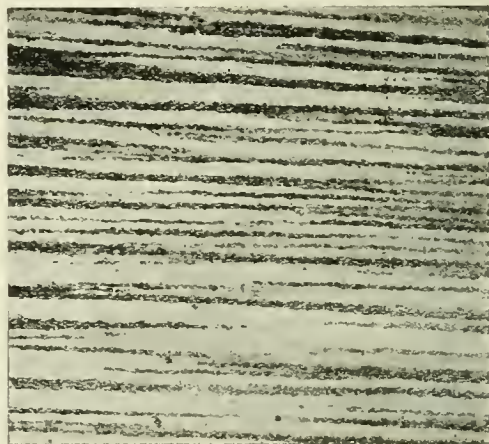


FIG. 22.

long time he had looked upon dendritic structure in an alloy as an indication of crystallisation through a rapidly-passing phase. He meant that, if one took a homogeneous substance, there was no reason why the crystals should not be perfectly complete. There was sufficient matter to fill out the skeleton, so to speak, of the crystal. In crystallising material like brass, as any one of the particular brasses fell out of solution, the remainder necessarily changed position rapidly and, therefore, it was necessary to suppose that there would not be sufficient material to fill out the skeleton. Up to a point, that appeared to be a feasible explanation, but there was the consideration that the best dendrite that he knew was, probably, the crystallisation on the surface of star antimony, which was usually looked upon as an indication of a very high degree of purity. Obviously, therefore, there must be more than one reason for the formation of a dendritic crystal.

Mr. A. SMOKE said that commercially pure antimony was not absolutely pure; it was very seldom over 99½% of purity, so that there might be enough impurity to help the crystallisation in dendritic form. A cast block of lead when etched gave an appearance very similar to some of the structures



which had been shown in the photomicrographs

Mr. GRATTON said that most of the ghosts the author had shown were etched with copper reagent. There were other kinds of ghosts that did not require such treatment to show them up, in the cast condition at any rate—ghosts that had got occlusions in the metal. It was evident that there were two kinds of ghosts. Mr. Smalley, with the copper reagent, had shown ghosts on several kinds of steel but, in the ordinary course of events, the speaker did not think it would be fair to refer to these steels as being ghost-marked. It seemed necessary to distinguish between ghost-marks that did harm and ghost-marks that did not. Ghost-marks with occlusions certainly did harm and they were absolutely attributable to the steel-maker. Mr. Smalley had referred to scorching and oxidation but did not exactly indicate whether these were the same. It was the speaker's experience that the hotter the steel was made, within reason, suitable after-treatment, teeming, checking down, etc., was much more likely to get rid of what were known as ghost-marks with occlusions.

Mr. DUNFORD SMITH asked how the "A" sample was modified in Table 1.

Mr. SMALLEY replied that, as to the low figures for the phosphor bronze, the tests he had included were quite normal. They were good, sound works' practice results. The bronze was not entirely free from occluded slag; no metal was. As far as the Admiralty gun metal was concerned, his low results were obtained purposely. The bars were cast in vertical chills  $1\frac{1}{2}$  in. square. They were doing some experiments at the time on extruded metal, and bars cast in that way gave information with regard to that point besides showing exactly the influence of the dendritic structure. Dendritic structure was an unbalanced structure and crystals emanating from the plane of the cooling surface which were responsible for the low results in the Admiralty gun metal clearly explained the results to which Mr. Smith had referred and were not due to any untrapped oxide or deleterious slag. As to Table 1 "A" modified, the analysis of cast "A" was copper 59, zinc 38, aluminium 3%. Cast "A" modified was the same alloy except that 1% of zinc was replaced by 1% of iron, the latter being introduced to create a nucleus action for the growth of the crystal grain. In sampling phosphor bronze and the Admiralty gun metal, drillings were taken near the top and bottom of the ingot. In steel, duplicate drillings were taken in different portions of the ingot. The most difficult point with which they had to contend was what the strength of the metal was due to. That was a question to which he could not attempt to reply at present. As to Mr. Gratton's remarks, what he inferred was that true ghosts in steel resulted from soluble impurities, and not from insoluble impurities. Such ghosts were equally detrimental to those produced by insoluble impurities. Insoluble impurities were always present in ghosts in virtue of the mode of solidification of the steel. The insoluble impurity, or the slag to which Mr. Gratton referred, always accompanied ghosts. As regarded the soluble impurity or true ghosts not being detrimental, he thought the tests he had shown completely settled the point that they were actually detrimental, sufficient to cause serious trouble to steel in the absence of any occluded slag. Tests made with the grain and against the grain of the ghosts gave figures, respectively, of 72 tons and 26 tons. He had not really been testing whether an insoluble ghost or an insoluble slag was detrimental or more detrimental than the other ghost. He was trying to demonstrate that a dendritic structure gave rise to ghosts, which were exceedingly detrimental to the quality of the steel.

## New York Section.

*Meeting held at the Chemists' Club on April 12th, 1918.*

### PLANNING A RESEARCH LABORATORY FOR AN INDUSTRY.

BY DR. O. E. K. MEES.

(Abstract.)

For the internal organisation of an industrial research laboratory there are two forms of organisation possible. For brevity these may be spoken of as the "departmental" system and the "cell" system. In the departmental system the organisation is that familiar to most businesses. The work of the laboratory is classified into several departments—physics, chemistry, engineering, and so on, according to the number necessary to cover the field, and each of these departments is in charge of a man of suitable scientific attainments. In a large department each of these men will in turn have assistants responsible for sections of the department, all the heads of departments finally being responsible to the director of the laboratory. Under the alternative or cell system the laboratory consists of a number of investigators of approximately equal standing in the laboratory, each of them responsible only to the director, and each of them engaged upon some specific research. Each such investigator, of course, may be provided with assistants as may be necessary.

In practice, some combination of these two systems of organisation is essential and will develop in any laboratory. The form of organisation which is the easiest in administration is undoubtedly some modification of the departmental system, since only by this means can young students, fresh from college, acquire adequate training and at the same time keep in touch with different branches of their subject and avoid the danger of over-specialisation too early. A laboratory should therefore be organised in departments with an intra-departmental arrangement under which a young man who develops the ability to carry out his own work may be able to take up work on his own initiative, still retaining his position in the department and carrying on his work under the general supervision of the chief of his department.

The laboratory itself should be housed in a convenient, special building. It is very advisable that all research work under the same general direction should be conducted under the same roof, although in technical research, where it is often necessary to instal model plants on a small scale, this cannot always be carried out. The annual cost of research work is very high in comparison with the cost of the building itself. The greater part of that expenditure is on salaries, and any inconveniences or disadvantages which may be caused by the working conditions and surroundings can easily depress the production to an extent which renders economics in building very unprofitable. The cost of the research man, in fact, is so high that it is worth while to provide him with the very best facilities for carrying out his work.

Research laboratories are almost always too small, and it is desirable that, in designing such a laboratory, some system of construction should be chosen in which expansion can be obtained by the duplication of units. It is important, also, in designing a laboratory to arrange, if possible, that expansion may take place without any considerable rearrangement.

Everything that has been said as to the necessity for the provision of a satisfactory building applies also to the question of equipment, but with even



greater force. It is an economic error to allow expensive men to be short of the apparatus which they require for their work. The total cost of equipment for a physical laboratory represents about two months' cost of operation, and, if economies are to be made, it is clear they should be made in limiting the amount of work undertaken and the consequent cost of operation, rather than in depriving the employed workers of the necessary tools for their work.

From various sources of published information, as well as from personal experience, it is possible to form an estimate of the cost of a research laboratory per scientific worker employed, taking the term "scientific worker" to cover all graduate men working in the laboratory. It might seem that there would be very great variation in the cost, but in laboratories of the physical and chemical type there is a surprising agreement between the different figures, which show that cost of building and equipment for a laboratory will be between \$3000 and \$4000 per man; it may be taken, therefore, that the first cost of a laboratory will be about \$3500 per scientific worker employed. The annual cost of maintenance of such a research laboratory appears to be slightly lower than the first cost. Probably \$3300 per man would be a fair estimate of the cost of maintenance, and of this about 60% represents salaries and wages and the other 40% all other expenses.

The work of an industrial research laboratory may be classified in three divisions:—

A. Work undertaken on the initiative of manufacturing divisions for the improvement of operations, for the lowering of cost, or in order to locate manufacturing difficulties.

B. Work undertaken with a view to the development of new materials or of entire new processes. This may be initiated by the management, by manufacturing sections, by sales divisions, or by the director of the laboratory or his assistants.

C. Work which deals with the fundamental theory of the subject the results of which, if successful, will lay a foundation for the expansion of the industry as a whole, along lines which usually cannot be foreseen when the research work is commenced.

The work classified under Division A is, of course, common to all industrial laboratories, and many research laboratories in connection with manufacturing plants confine themselves almost entirely to problems arising from the manufacturing division.

Class B includes a large portion of the work of industrial research laboratories, and the best known successes of such laboratories are included in this division. A typical example is the development of the drawn wire tungsten filament by the research laboratory of the General Electric Company, a research which, although originating from a general research on the properties of rare metals such as would be classified under Division C, developed into a study of tungsten with the direct purpose of obtaining a satisfactory lamp filament from the metal. Another example is the manufacture of indigo by the Badische Company. Such researches usually have their basis in some fundamental work.

More rarely do research laboratories work on subjects classified under Division C, that is, on the fundamental theory of their subject. Yet those who do, achieve the most conspicuous successes. The work of Prof. Abbe on the theory of the microscope, and, indeed, all the work on applied optics at Jena, come under this heading. The great success of the Zeiss works is directly due to the attention paid by Abbe to the development of the fundamental theories of optics. At the General Electric laboratory at present much attention is

being paid to the emission of electrons from hot bodies, and from this work there have already developed the Coolidge X-ray tube and the Kenotron high-frequency transformer, while the possibilities of application are as yet only just beginning to be realised.

If proper co-ordination exists between the laboratory and the management, work classified under A and B will certainly be reasonably remunerative, although not necessarily so completely so as to pay the dividends on an investment in the research laboratory, which is commonly expected from such an investment. The same may not appear true in the case of Division C, the fundamental work, which in the hypothetical case discussed would represent nearly a third of the total expenditure of the laboratory; nevertheless, it is probable that this section of the work would be likely to prove the most remunerative of all, as exemplified by such work as that done by Prof. Abbe on the geometrical laws which govern the formation of images by lenses; by the various bureaus of standards in research on analytical methods; the study of the relation between inductance and capacity in alternating electrical circuits; work on the photo-electric effect, the diffraction of the X-rays by crystals, or the emission of electrons by hot bodies, etc.

It is often felt that small industries cannot afford to support scientific research, but this argument is exactly as if it were suggested that small industries cannot afford to support advertising. The object of spending money on research, for a small industry at any rate, is not to support the research but to be supported by it, and it is scarcely an exaggeration to say that the smaller a business is, the more important is it that it should make use of scientific research to the greatest extent possible.

A small business is at a disadvantage in comparison with a large one in regard to all its cost charges. In the purchase of raw materials, in manufacturing, and in selling, its cost per unit of output tends to be larger than in the case of big businesses, but, on the other hand, it is at a real advantage in regard to flexibility and enterprise. Any large business must necessarily be cautious and conservative. The amount at stake is so large that the penalty of error is heavy.

Small businesses can therefore make far more use of a research laboratory and get a much bigger percentage return for the expenditure than any big company can hope to do. In the small business, in fact, a research laboratory closely associated with one of the high executive officers should begin to return a profit within a few months of its establishment, whereas in the case of a large company it may be years before a research laboratory can be considered to be financially successful.

The greatest difficulty in the establishment of a research laboratory in a small business is that any research laboratory will depend for its value upon the quality of the men at the head, or, if the laboratory is really small, of the man at the head, and a small business often feels that it cannot afford to pay even one good scientific man. The solution of this in a technical business might be that the research man should also be an officer of the company, so that his cost is borne not only by the scientific work but also by the value of the executive position which he holds. It may be objected that an investigator would not as a rule prove a capable business man, but there seems to be no particular evidence for this common belief, and there are many examples of men trained in science who have proved extremely good administrators. In many cases it will be found that the technical industries are directed by technical men who were themselves directly concerned with development and manufacture rather than with financial or business direction.

## PROCEEDINGS OF THE THIRTY-SEVENTH ANNUAL GENERAL MEETING,

*Bristol, July 17th to 19th, 1918.*

The thirty-seventh annual general meeting of the Society was held at Bristol University on July 17th, Prof. Henry Louis, the President, in the chair.

The Rt. Hon. the LORD MAYOR OF BRISTOL (Alderman Frank Sheppard), in offering the Society a hearty welcome to Bristol, said that the chemical industry was commanding more attention at the present time than it had done during the whole of its history. He remarked that one of the subjects to be discussed was the question of copartnership, and in that connection he believed that if we were to have peace in industry in the future there must be a far closer co-operation between employers and labour generally than there had been in the past. Many efforts had been made recently to establish this kind of understanding. The average working man now had little interest in the particular industry in which he was engaged. Thirty or more years ago the men who were engaged in a trade were craftsmen and had some interest in their trade, but during the years that had passed since then the boys that had grown into men in the industrial world were simply tiny eggs in connection with the very important industries of our country. Some complaint had been made of the lack of knowledge and of the ignorance displayed by working people as a whole, but they must not complain of that ignorance. It was due to somebody who had been seriously at fault not to have noticed the tremendous change that had gone on during the last 20 or 30 years and to have provided something to make up for what the young fellows lost compared with what formerly was the case. A lad left school at 14 years or so and was put straight into a factory where he was engaged in the most minute part of a particular industry; when he became a man he was no better than what used, in the old days, to be considered an unskilled labourer. Those in industry must see that something should be done to give the boys and men more interest in their industry. At the present time there was a great desire on the part of employees for reduction in the number of hours of labour, and the reason was that their work was so monotonous and uninteresting that they had no real love for it. He believed this would be remedied if they could be given some other interest in life than being a mere cog in a machine.

Complaint was sometimes made in regard to all industries that the right men were not put in the right place and that people in industry did not hold high office in the affairs of the country. They must try to find out whether they themselves were not to blame in that respect. If they wished to occupy the positions which many of those in industry ought to, they must adopt democratic methods. Another complaint was that the Government would not use the services of men in industry to the extent they could. It seemed to him that it was necessary to establish an ideal of honesty with regard to trade which would make the country feel it could appeal to representatives of trade and use their services and knowledge in high positions without any of the suspicion that had existed for so long, when a man in a trade who occupied these high positions was suspect.

Sir ISAMBAARD OWEN, Vice-Chancellor of the University of Bristol, said he had great pleasure in bidding the Society a very cordial welcome to the

University of Bristol. They had had but four years in which to endeavour to develop the University before the war overtook them. They had started with the largest conception of what a university should be and what it ought to aim at. They wanted to see a real seat of learning in the West, an assembly of men and women who were exponents of the various branches of learning in their highest and most up-to-date form, branches of learning not merely literary and scientific, not merely theoretical, but practical; not a piece of machinery for coaching young men and women to pass examinations or an apparatus for turning out a specified number of graduates per annum. The universities of the middle ages, far from concerning themselves merely with theoretical and impractical branches of learning, had their eyes constantly fixed upon the practical occupations which were open to learned men, and whereas learned professions in the universities had once numbered three, at the present day they were numbered by the dozen and their tendency was constantly to increase. It was difficult to see what occupations in the future would not be carried on by the help of systematised and exact knowledge, and thereby the scope of our future universities had become enormously enlarged. The practical aims of a university had been constantly kept before them at Bristol since its foundation, and with a university that had been founded and which was largely maintained by local benefactions, they had esteemed it their primary duty to endeavour to assist the leading industries of the district. The members of the Society of Chemical Industry, who were engaged in turning one of the most abstruse and one of the most rapidly advancing of modern sciences to practical purposes, were very heartily welcome at Bristol.

The PRESIDENT, in expressing the thanks of the Society for the very kind welcome that had been extended, said it was one which peculiarly fitted the aims and objects of the Society. The Lord Mayor had given them a civic welcome on behalf of the citizens engaged in the industries of Bristol and the Vice-Chancellor had given them a welcome on behalf of the seat of learning which was disseminating education throughout the same ranks. It was that combination, the application of science to industry, which was the main object of the Society. The fact that a new Section of the Society had been inaugurated recently at Bristol and was already in such a flourishing condition showed very clearly the way in which the efforts of the whole country were tending. Bristol had long been famous as one of the oldest and most enterprising centres of commerce of the United Kingdom. Its entry into the big industrial ranks had been comparatively recent and that movement was also typical of what was taking place throughout the country. Napoleon was not far wrong when he jeered at us as a nation of shopkeepers. We had always had the unhappy habit of putting commerce before industry as though industry could be founded upon commerce instead of commerce upon industry. Commerce could only be flourishing when there was a powerful and energetic industry supplying the goods. In modern times industry without science was unthinkable. It would be difficult to name any industry which



in some sense or other was not based upon some part of the chemical industry. In that respect the Society represented modern industrialism in the widest sense. The Lord Mayor had pointed out very truly how closely industry and education must be connected in the future if we were to maintain our place among the industrial nations of the world. It was one of the objects of the Society to act as the bond of union between scientific education and the industrial applications of science. The double welcome that had been extended to the Society was therefore entirely gratifying to them and they heartily thanked the Lord Mayor and the Vice-Chancellor.

The Lord Mayor then withdrew and the President took the chair.

The minutes of the last Annual General Meeting and of the Special General Meeting on 15th August, 1917, were read and confirmed.

Messrs. C. J. Waterfall and B. D. W. Luff were appointed scrutineers for the ballot for the election of Council and officers for the ensuing year.

### REPORT OF COUNCIL.

During the year the Council has held 10 meetings, Accounts Committee 10, Full Publication Committee 3, Executive Sub-Committee 21, Review Sub-Committee 18, Emergency Committee 3, Annual Reports Committee 2, Finance Committee 4, Premises Committee 4, Reconstruction Committee 1, Library Catalogue Committee 1, Joint Abstracts Committee 3, International Catalogue Committee 2, Chemical Bibliographies Committee 2, Patent Bill Committee 3.

The number of Members on the Register is now 4823, as compared with 4429 last year. Since the last Annual Meeting 605 Members have been elected, and the losses have been 208.

The Council regrets to record the deaths of 56 Members (of whom 15 were Original Members), viz.:—A. Alhusen, W. Raymond Baird, Dennis H. Barraclough, Clayton Beadle, D. Bendix, Sir Samuel Boulton, Bart., Harold R. Braid, Caesar R. Brown, Jas. Buchanan, Jas. Burbridge, Alexander M. Chance, Spencer Chapman, Geo. Christison, T. C. Clond, C. G. Cresswell, Wm. A. Dixon, Sylvain Dreyfus, J. J. Eastick, J. H. Elson, Prof. G. A. Ferguson, H. Forth, I. Frankenburg, R. Le Neve Foster, R. B. Gibbins, Walter H. Giles, Chas. W. Graham, Jos. Hartley, Norman M. Henderson, G. T. Holloway, T. Kenyon, F. W. Lee, T. W. Lovibond, S. Lupton, W. T. Lye, G. E. McCleak, Peter MacEwan, J. G. McIntosh, W. Marsh, John Meikle, John Milne, Percy S. Nelson, Mulgrave D. Penney, Rufus D. Pullar, Prof. Jos. P. Remington, Chas. M. Reubens, A. Gordon Salamon, Dr. Hugo Schweitzer, R. W. Skipp, J. L. Spoor, Walter S. Taft, Edward C. Thompson, Thos. Tyrer, Walter A. Voss, Jas. L. Wade, Wm. J. Ward, Reginald C. Woodcock, G. S. Woolley, John Young.

Mr. Thos. Tyrer was President of the Society from 1895 to 1896, and was Hon. Treasurer from 1908 to the time of his death; Mr. C. G. Cresswell was General Secretary from 1883 to 1916.

The following members of the Society have died on active service:—Dennis H. Barraclough, Roland B. Gibbins, Walter H. Giles, Percy S. Nelson, and Ralph W. Skipp.

Mr. D. Lloyd Howard was appointed Honorary Treasurer in succession to the late Mr. Thomas Tyrer; and Mr. Thomas Fairley, Honorary Secretary of the Yorkshire Section, was elected a Vice-President in room of Mr. Howard.

To the vacancies caused by the retirement of four Vice-Presidents, Dr. E. F. Armstrong, Professor W. R. Hodgkinson, Mr. Robert L. Mond, and Mr. Walter F. Reid have been nominated.

Mr. W. J. A. Butterfield and Mr. E. F. Hooper were elected Ordinary Members of Council in place of Professor H. E. Armstrong resigned, and Mr. R. D. Pullar deceased, respectively.

Four Ordinary Members retire from the Council, and to fill the vacancies thus created 6 nominations have been received; a ballot will therefore be taken.

The following Chairmen of Local Sections retire:—Mr. T. H. Wardleworth (Canada), Mr. James Macleod (Glasgow), Mr. H. Peile (Newcastle), Dr. R. M. Caven (Nottingham), and Professor J. W. Cobb (Yorkshire). The following have been elected to succeed them:—Dr. W. L. Goodwin, Mr. Quintin Moore, Professor P. F. Bedson, Mr. F. H. Carr, and Mr. W. McD. Mackey.

The following changes have taken place among the Honorary Local Secretaries:—Mr. T. D. Morson (London) and Mr. E. F. Hooper (Newcastle) resigned, have been succeeded by Dr. S. Miall and Mr. H. Dunford Smith respectively. The Council desires to express its thanks to the retiring officers for their services to the Society.

The Council has appointed a Finance Committee, of which Dr. Carpenter is the Chairman, to supersede the Accounts Committee now discharged, having executive powers in all financial matters not involving questions of principle, the decisions of the Committee to be reported to the Council.

Two new Sections have been formed, and are now in full working order:—the Bristol and West of England Section, and the Canadian-Pacific Section (originally called the British Columbia Section). The Council has had much pleasure in accepting the invitation of the Committee of the former Section to hold the Annual General Meeting of the Society this year in Bristol, and it heartily wishes for each of the new Sections a future of prosperity and increasing usefulness.

Owing to difficulties experienced in the way of successfully carrying on its work, the New England Section was dissolved by the unanimous vote of its members. A proposal from the Committee of the New York Section to take over the members of the Section was approved by the Council.

The suggested formation of an Indian Section of the Society was discussed at the Conference of Chemists held at Lahore in January. It was agreed that at present the proposal is impracticable, and that until the number of chemists in India increases or a Government Department is organised the Chemical Science Congress appears to meet all requirements.

There have been two Conferences of Local Chairmen and Secretaries during the year, the first being held in Birmingham at the time of the Annual Meeting and the second in London. These meetings have proved helpful to the officials attending them, enabling them to discuss matters relating to the organisation and development of sectional activities.

The Balance Sheet and Annual Statement of Accounts, which have already appeared in the Journal for 30th June, will be laid before the Meeting.

The Journal for 1917 contained 1290 pages of text, compared with 1274 pages in 1916.

The resolutions unanimously adopted at the last Annual Meeting that (1) the Annual Subscription for Membership of the Society should be raised from 25s. to 30s., commencing on 1st January, 1918, and (2) the life composition fees should be increased by £5 each, were confirmed at a Special General Meeting called for the purpose and held on 15th August, 1917, in accordance with the Society's by-laws.

A Committee has been appointed by the Council to take steps to arrange for a memorial to the late

Mr. Thomas Tyrer, and it has been suggested that such memorial should take the form of a Technological Library to be established in the Society's rooms.

The Council has awarded the Society's Medal this year to Sir James Dewar, F.R.S., in recognition of the conspicuous services which by his research work in both pure and applied science he has rendered to Chemical Industry.

The Council has appointed a Committee to consider the whole subject of the Patent Law and to advise it as to any action that should be taken by it either individually or jointly with other Societies.

In January the Government took over Broadway Chambers, Westminster, and it became necessary for the Council to secure new offices for the Society. Suitable premises were acquired at Central House, Finsbury Square, where ample accommodation is now provided for both the Secretarial and Editorial Staffs.

Subsequently, the Society's printers, Messrs. Vacher & Sons, Ltd., and their total output, were commandeered on behalf of H.M. Stationery Office. The Council has, however, now entered into a contract with Messrs. Spottiswoode, Ballantyne & Co., Ltd., for the printing of the Journal, and the issues from 15th June inclusive have been sent out from their press.

The Council has decided to take into its own hands the publishing and advertising arrangements of the Journal.

In last year's report it was stated that the Council had decided to take advantage of the marked development of interest in the Journal that during the past 2 or 3 years had become manifest, to increase its efficiency and to make it as useful as possible to all those engaged in the various departments of Chemical Industry. The Council is gratified to learn from many sources that its efforts in this direction have not been in vain, and that the new features embodied in the "Review" introduced into the Journal at the beginning of the present year are highly appreciated by the members generally.

At the request of the Association of British Chemical Manufacturers the Council has agreed to allow the "Review" part of the Journal to be the official organ of the Association.

Dr. Charles Carpenter and Mr. W. G. Wagner have been elected members of the Publication Committee.

The Council is gratified with the success that has attended the publication last year of the first volume of its Reports of the Progress of Applied Chemistry. Volume 2 is in the press and will, it is hoped, be published immediately.

In the competition for the Cross and Bevan Prize (with T. P. Latham's Studentship: see Report of Council 1917), four essays were received and examined by Mr. C. F. Cross and Mr. S. S. Napper. These gentlemen reported that two of the essays had sufficient merit to be entitled to a recognition, and they suggested an award from the Prize Fund of 5 guineas to Mr. Edward Wheeler of Bellevue Crescent, Ayr, and of 3 guineas to Dr. H. A. Tempamy, Department of Agriculture, Reduit, Mauritius. As there was not sufficient ground for the award of the Studentship, this matter will be re-opened for competition, of which due notice will be given in the Journal.

The Monograph on the "Constitution of Coal" by Dr. Marle C. Stopes and Dr. R. V. Wheeler, based on a joint memoir read in January 1917 before the London Section of the Society, has been published by H.M. Stationery Office on behalf of the Department of Scientific and Industrial Research with the consent of the Council of this Society. The authors were invited by the Chairman of the London

Section to undertake in co-operation the production of a Monograph on the whole subject, which is of considerable importance from both the economic and scientific standpoints and forms a record of work of much value. The Council has purchased a supply of copies of the Monograph, and a copy was sent to each member of the Society with the issue of the Journal for 15th July.

With regard to the Research on Cellulose carried out at the College of Technology, Manchester, at the instance of the Society, Mr. Julius Huebner, under whose direction the work is being done, reports that the results so far obtained are promising, and will form a valuable foundation for more extensive research which he hopes to undertake after the conclusion of the War. The Department of Scientific and Industrial Research has agreed to make a further grant at the rate of £100 per annum from 1st April, 1918, to the end of the academic year towards the expenses of the research.

The Joint Committee of Representatives of the Chemical Society and the Society of Chemical Industry (see Report of Council 1917) have recommended that in order to prevent overlapping in publishing abstracts of scientific papers, the Chemical Society should deal only with papers on pure Chemistry, while the Society of Chemical Industry should restrict its abstracts to those of papers on Applied Chemistry; also that if a sufficient number of members of the one Society who are not members of the other Society desire it, the latter Society should supply them with copies of its abstracts printed in a separate volume. These recommendations have been approved by the Council of each of the Societies; but owing to the small number of applications that have been received for the proposed volume of "Journal" abstracts, the Council has decided not to proceed further with the proposal at present. The matter will be brought up again next year for further consideration.

The Empire Sugar Supply (Technical) Committee which was formed (see Report of Council 1917) in connection with the London Section has now received voluminous and informing replies from the Governors and Secretaries of practically all the Dominions, Colonies, Dependencies, and Protectorates of the Empire, setting forth their production and requirements of sugar for different purposes. A large amount of information has also been obtained regarding the cultivation of sugar beet in this country, and the manufacture of sugar therefrom. The information collected is most valuable, and the work of arranging and collating the statistics is now proceeding.

The following Representatives of the Society have been appointed:—

Sir Boverton Redwood, Bart., and Sir George Beilby, F.R.S., on the Board of Scientific Societies.

Dr. Alfred Holt on the Sub-Committee of the Board of Scientific Societies for considering the future of the International Catalogue of Scientific Literature.

Mr. A. R. Ling and Mr. J. W. Macdonald on the Empire Sugar Research Association.

Mr. E. V. Evans on the Committee of the British Science Guild considering the position of the British Dye Industry.

Prof. F. G. Donnan, F.R.S., and Dr. E. F. Armstrong on the General Board of the National Physical Laboratory; and Prof. Donnan on the Executive Committee of the Board.

Mr. W. F. Reid on the Sectional Committee dealing with chemical products in connection with the British Scientific Products Exhibition to be held in July in King's College, London.

Dr. Carpenter and Dr. Keane on the General Committee of the Chemical and Allied Societies.



The General Committee of Chemical and Allied Societies having had under consideration the publication of Chemical Bibliographies in the English language has resolved (1) that it is expedient to publish an English work of reference completely covering scientific and industrial chemistry on lines similar to those of Beilstein, and (2) that this resolution be communicated to the corresponding American Chemical Societies and their co-operation invited. The Council has approved of these resolutions, and is informed that the General Committee is at present considering a scheme for giving effect to them.

The Council notes with pleasure the formation of the Société de Chimie Industrielle, and is gratified to learn that it is to be organised on the same lines as the Society of Chemical Industry. The Council expresses its hope that the formation of the Société will lead to closer co-operation between the chemists of the Allied countries.

Dr. E. F. ARMSTRONG moved the adoption of the report. As a member of Council he said he felt very proud of the large amount of work that the Council had been able to do. Chemistry and the chemical industry, like all other branches of industry and commerce, were passing through a period of flux and change and the Council had had to consider many difficult problems during the year, and he felt they had reason to be proud of the success which had attended their efforts. A great deal of the success of the Council's work this year had been due to the work which Dr. Longstaff, the Secretary, had done. The formation of the Bristol Section was referred to in the report; he hoped this was a good augury for the future and that the Bristol Section would flourish.

Dr. STEPHEN MYALL seconded the adoption of the report and associated himself with all that Dr. Armstrong had said with regard to the formation of the Bristol Section, to which they wished great success and prosperity.

The report was unanimously adopted.

#### HON. TREASURER'S STATEMENT.

The Hon. TREASURER, in submitting the balance sheet (printed in the Journal of June 30, 1918), said the accounts did not call for special comment except that the Society could congratulate itself upon having a fairly normal balance sheet. There was a gratifying increase in the amount of the annual subscriptions, and as the figures applied to last year, the increase was entirely due to increased membership, because the increased subscription had not then begun to bear fruit. The excess of income over expenditure was £556 4s. 7d. Many items of expenditure had increased and in the current year there would be still further increases.

Prof. SCOTT RUDOLF moved the adoption of the accounts and congratulated the Society on being in such a comfortable financial position. They were all very much indebted to the Treasurer and those associated with him for the manner in which they had safely piloted the financial ship during the past year.

Mr. E. WALLS seconded the adoption of the Treasurer's report. He referred to the loss of the Society's late Treasurer, Mr. Tyrer, and expressed the opinion that they had secured in his successor one who would carry on the high ideals which had always been associated with that office. Having regard to the extreme difficulties of the present period, the great progress that had been made by the Society indicated an admirable state of affairs.

The PRESIDENT, in putting the motion to the meeting, called attention to the fact that this was the first appearance of the new Treasurer, who he thought had every reason to be proud of the statement he had been able to put forward.

The balance sheet was then adopted.

#### AWARD OF THE MEDAL.

The PRESIDENT said it was his high privilege to announce the award of the Medal of the Society to Sir James Dewar, Fullerian Professor of Chemistry at the Royal Institution since 1877 and President of this Society exactly 30 years ago. It was unnecessary to attempt to catalogue the long series of important discoveries of Sir James Dewar in the numerous and varied domains of physical chemistry that he had long made his own. It was sufficient to say that his labours had most worthily carried forward the brilliant record of epoch-making scientific discoveries that formed the main glory of the Royal Institution. Whilst his discoveries had been confined to pure science, the importance of their practical application would not be denied by the veriest utilitarian who had ever drunk his hot coffee on a winter's morning out of a Thermos flask. It could not be too often or too insistently repeated that it was to the scientific discoverer, to the man who worked out scientific truths for their own sake, that industry was indebted for the solution of its most complex problems. True research had been and was perhaps still too little considered in this country; it was true that attempts had been made recently to repair our past neglect by fostering what our authorities were pleased to call "industrial research," but he gravely doubted whether there really was such a thing as industrial research; what did exist and what we as a nation emphatically required was scientific research, applicable of course to the industries, but research conducted by scientific men in a true scientific spirit. It was to work of this kind that Sir James Dewar had dedicated his life and it was inevitable that discoveries such as those which we owed to him should have proved of value in many different industries, and in not a few cases in industries with which their connection at first sight might seem remote. To take but one example:—The scientific work of Sir Humphry Davy, Sir James Dewar's great predecessor, had enabled him to teach the coal miner how to combat the dangers of an atmosphere of fire-damp; the scientific work of Sir James Dewar had led to the production of one of the most effective forms of rescue appliances that enabled the coal miner to penetrate an atmosphere of after-damp, and the practical value of the one and the other of these inventions could hardly be over-estimated. Yet no one would dream of applying either to Davy or to Dewar the title of practical man. Great as had been the value of Sir James Dewar's work in the advancement of pure science, and in its applications to industry, he ventured to think that it was at least equally valuable to us for the lesson that it enforced that the men capable of work of such high scientific order constituted one of the most important of our national assets. Sir James Dewar had expressed his regret at being prevented from coming to Bristol to receive the Medal in person, and at his request he had much pleasure in handing it for transmission to him to Dr. Armstrong, the son who is following so ably in the footsteps of his eminent father, to whom we owed amongst many other things a most profound and illuminating appreciation of the work of Sir James Dewar. He asked Dr. Armstrong to hand the Medal to Sir James Dewar and to express to him the cordial wishes of the Society for his health and welfare, and the sincere hope that he might long continue to give them the benefit of his scientific investigations.

Dr. E. F. ARMSTRONG said that Prof. Dewar had asked him, as the youngest representative of the old school, to receive the Medal on his behalf. The younger men must find in the example of such men as Sir James Dewar a great stimulus to do their best to advance the science and the



cause of the chemical industry. He was sure that the very happy words the President had said were just those which Sir James would appreciate, because he held the view that there was no research worthy of the name which was not scientific research and that it was by pure scientific research alone that advances would be made in the future. He would convey to Sir James Dewar what the President had said.

#### PRESIDENTIAL ADDRESS.

Prof. HENRY LOUIS then delivered his Presidential Address as follows:—

My first duty is to thank you for the great honour that you have done me in electing me to the Presidential Chair of this important Society. If I confine myself to this simple expression of thanks, believe me that it is not because I am in any measure wanting in gratitude for the honour that this evidence of your confidence has conferred upon me, but merely because other and more important matters claim our attention to-day.

As the report of the Council has shown, the past year has been one of the most eventful in the history of our Society, and one that has caused all your officials an unusual amount of care and anxiety. In the first place we had decided to alter the form and widen the scope of our Journal, in itself no light task. Whilst we were fully engaged on this work we received peremptory orders from the authorities to vacate our old offices and to find for ourselves a new home elsewhere. I willingly admit that this summary ejection was in reality a blessing in disguise; the activities of the Society had long outgrown our old quarters, which were far too small to allow of our work being carried on satisfactorily, and we have been very fortunate in securing offices sufficiently commodious to provide not only for present needs, but for such expansion of the Society's work as we may fairly look forward to for a considerable time to come. Even before we had fairly begun to settle down in our new abode, the Paper Controller threatened to cut down drastically the supplies of paper for our Journal, and almost simultaneously the Government commandeered at short notice the works of the printers who had published it for so many years. I am only too well aware that many of our members have suffered considerable inconvenience through issues of the Journal being delayed, in some cases for many days beyond their due date, and I can only thank the members as a whole for the forbearance that they have shown under the circumstances; but I think that the facts that I have just stated will make it clear that the fault did not lie with the permanent officials of the Society, and that it is rather to be scored to their credit that the Journal has been kept going as well as it has been, and that the delays that have occurred are quite excusable. I will not go so far as to say that all our difficulties have been entirely surmounted, but we are certainly beginning to get things running rather more smoothly, and I have every hope that a few months more will see everything going as steadily as is possible in the critical times through which we are passing. It is gratifying to be able to add that neither these difficulties nor the world's crisis in which we are involved have retarded the steady growth and development of the Society, which we can fairly claim is to-day in a highly flourishing condition.

Great though the honour is of occupying the Presidential Chair of an influential and old-established Society such as this, I must admit that it entails one serious corresponding drawback. I have before me a long line of illustrious predecessors, and I must endeavour to live up to the high standard set by them as far as lies within my

power. Whilst this difficulty is continuously with me, I am necessarily more sensible of my shortcomings at a moment such as this, when it falls to me to submit to you an address on some subject of general interest; not that it is difficult to find such a topic at the present moment, but rather because all the problems of the chemical industry have already been so well and so exhaustively treated from every conceivable standpoint.

It so happens that the supreme crisis in which the nation now finds itself involved has caused us to revise our views on many subjects, and on none perhaps more completely than on the value of chemistry as a national asset. The nation has gone on for generation after generation, cheerfully committing its government to men whose training necessarily resulted in a complete ignorance of and almost a contempt for scientific methods. Fortunately we have always possessed a sufficient number of quiet unobtrusive workers, whose devotion to science for its own sake has succeeded in maintaining the high standard of British science in spite of official neglect, and it is such men as these who have come to their country's assistance in its hour of need; had it been otherwise, our position would indeed have been hopeless. Under the spur of rude necessity has come a sudden awakening, and even the most case-hardened parliamentarian has come to realise how entirely the nation is indebted for its very existence to the applications of science, and above all of chemistry; and many are at last beginning to recognise that this dependence is as complete in the industries of peace as in those of war. With this recognition has come a desperate rush in the attempt to make up for lost time, as though methods of scientific thought could be learnt in a day, and a century's neglect of scientific principles could be atoned for by the creation of miscellaneous committees. The realisation of the vital need to the nation of a large body of thoroughly trained industrial chemists is at any rate all to the good, and it is a hopeful sign that the best methods of equipping such men for their life's work are attracting increasing attention. Fortunately the public interest thus aroused has enabled chemists themselves to attack those problems in a bolder manner than heretofore, under conditions that would have been impossible had they not felt that they are at last sure of a reasonable measure of public support.

One of the questions that have been most prominently before British chemists recently is that of the best means of securing an adequate supply of chemical engineers to maintain and develop the great chemical industries of the country. Many of us realise that our chemical industry may in some respects be said to have been practically stationary during recent years, and in no respect is this fact more noticeable than in the failure to take advantage of modern mechanical methods of handling large bodies of material. In other words, not sufficient has been made of the application of modern engineering methods to chemical industries. The difficulty undoubtedly lies in finding men who are equally conversant with the chemical problems to be solved and the engineering facilities available for their solution, whilst the problem is further complicated by the fact that the constructional materials employed are frequently required to withstand corrosive influences which lie outside the experience of the ordinary engineer. As has been repeatedly pointed out of late, a man is required with special training, who is for convenience sake usually spoken of as a chemical engineer, though I am free to admit that the term is not in all respects a happy one. No doubt the difficulties in meeting this modern demand for a thoroughly trained chemical engineer are considerable. No



chemist would admit that a man can be considered to be a qualified technical chemist unless he had spent a reasonable amount of time in working in an industrial chemical laboratory; on the other hand no engineer would consider that a man had received a true engineering training unless he had put in a reasonable time in working in the shops. It is therefore evident that unless a young man is willing and able to devote an exceptional length of time to his preliminary training he cannot be thoroughly equipped as a chemical engineer, and the result is almost necessarily a compromise. Hitherto work of this kind has perforce been carried out by men who were either only engineers or else only chemists, and I should have considerable difficulty in saying which I have found to be the worse—the average chemist's engineering or the average engineer's chemistry. There have, of course, been numerous and brilliant exceptions, and most of the important problems of chemical engineering have been tackled by men who had naturally special aptitude in both directions, so that there has been really a large amount of first-class chemical engineering work done, though much of it is scattered through various branches of industry not generally included in what we understand by chemical industry in the ordinary acceptance of the term.

It may perhaps be helpful if we devote a short time to a consideration of the nature of the principal operations that a chemical engineer is called upon to conduct. I propose to limit the field by omitting on the one hand such work as falls generally to the ordinary mechanical engineer, such as the consideration of prime movers, the transmission of force, etc., and on the other hand such processes as can scarcely be classed as mechanical operations at all. This leaves for our consideration a series of such operations as comminution, transport of materials, handling of solutions, filtration, thickening or clarifying, etc. I am omitting from this discussion such operations as involve furnace work, evaporation or distillation, because these can hardly be classed as mechanical operations.

Nevertheless it must not be forgotten that mechanical methods have been applied to most of these; anyone who will take the trouble to read through the list of patents taken out, for example, in connection with mechanically worked roasting furnaces cannot but be struck by the immense amount of ingenuity that has been devoted to this one comparatively simple operation. Such furnaces have, it is true, been devised essentially for metallurgical operations, but many of the principles and methods could be applied readily enough to purely chemical practice; in any case a roasting furnace which generates sulphurous gases forms the starting-point of so important a section of chemical industry that the chemical engineer cannot afford to disregard it. In the same way the steel-maker has evolved a number of highly ingenious tilting furnaces and furnace-charging appliances that are well worthy of consideration from the same point of view. By far the largest-scale distilling operation in the world is that of coke-making; the coke oven is the largest retort used for any purpose, and here again it is interesting to note that mechanical methods both of charging and discharging the retorts have been evolved and are in successful daily use. In the same way many interesting mechanical devices for similar purposes are being employed in gas-making. Again, engineers are applying some of the scientific principles of physics to large-scale evaporations, and it is well known that remarkable economies have of late years been achieved by these means.

The operations of comminution cover a very wide range, varying with the hardness of the materials

and the size to which it is required to reduce them. At one end of the scale we have the so-called rock-breakers, reciprocating or gyrating, capable of dealing with huge masses of the hardest rocks. Some of these have been built large enough to take blocks over  $1\frac{1}{2}$  yards cube and to break them down at one operation to about 9 inches; such an immense machine requires about 300 h.p. to drive it, and can break no less than 250 tons per hour. Crushing rolls are extensively used for a large number of different products; like the last-named machines they were originally devised for breaking down ores, but their use has been extended to a vast number of different materials. The modern high-speed roll is a very admirable machine, especially advantageous when it is desired to obtain a granular product suitable for percolation. Rolls have been built of very large size, but the general practice now is rarely to exceed 4 ft. in diameter by 2 ft. face, running with a peripheral speed of about 1000 ft. per minute. Such rolls may be taken as crushing on an average about 5 tons per hour, and absorbing some 10 h.p. I need hardly say that figures varying widely either way from the above averages are to be met with in practice. Californian stamp mills, which are simply mechanically worked pestles weighing up to 10 cwt. apiece, have rarely been applied to anything except the crushing of ores of gold and silver, but can be adapted to a wide range of purposes. They are usually arranged to crush to meshes ranging from  $1/50$ th to  $1/20$ th of an inch, and their capacities may be averaged as from 2 to 10 tons per stamp head per 24 hours. For still coarser crushing the Lake Superior steam stamp, which is in effect a steam hammer arranged for crushing, can render valuable service; it is generally set to stamp to about  $\frac{1}{2}$  inch mesh, and one stamp will crush up to about 1000 tons per day. Both these forms of stamping machinery deserve careful consideration whenever it is required to crush any hard material wet, and when an excessively fine product is not demanded; they are strong, simple, efficient, and thoroughly reliable. For still finer crushing, ball mills are extensively used; these have already been employed in various branches of the chemical industry, but, as far as I know, the improved Hardinge conical ball mill, which has a considerably greater capacity than the original cylindrical ball mill, has so far rarely been employed except for crushing ores. For the finest grinding, tube mills, originally devised for grinding cement, are being extensively used, having largely replaced such appliances as edge runners and burr stones on account of their greater capacity, their higher efficiency, and lesser need for attention. For crushing softer materials, disintegrators of various types are used. I believe that these were first devised for crushing coal, but they have found an extensive application in numerous branches of chemical industry on account of their large capacity and general convenience.

In connection with modern crushing appliances, attention should be directed to the development of various automatic feeders, which are capable of very precise regulation so as to deliver to the machine the exact amount of material that it can treat with the maximum of efficiency. It is quite obvious that either over-feeding or under-feeding will bring about a falling off in this respect, more particularly in the case of such machines as stamp mills and ball mills, which consume practically the same amount of power when they are running empty as when they are running at their full capacity. Out of this extensive range of comminuting appliances with their appropriate feeders it is possible to select machines for doing practically any kind of coarse-crushing or fine-grinding, and on almost any desired scale. Many of these



have not yet made their way into ordinary chemical industries, but they are extensively used in the dressing of minerals and allied metallurgical operations, and their use is obviously indicated wherever large quantities of material have to be broken down.

I need say very little about the appliances available for the transport of materials either to or within the works. Aerial ropeways are coming into general use, and their flexibility and wide range of application are universally recognised; telfer lines of various kinds are well known, belt conveyors, scraper conveyors, screw conveyors, shaking conveyors, have all been thoroughly studied and brought to a high state of efficiency. There are few cases in which one or other of these forms cannot be employed with advantage, so that they have in the majority of important works to a great extent supplanted the labourer with his wheelbarrow, although this time-honoured relic of the days of cheap labour is still too much in evidence in many of our chemical works.

I will add nothing more under this head, but may turn to the equally important and perhaps more difficult problem of dealing with liquids in very large quantities. The economical solution of this problem was indispensable for the full development of an important group of processes—namely, wet metallurgical processes or the extraction of metals from their ores in the wet way. Such processes are by no means new; the extraction of copper from mine waters containing it in solution by the so-called cementation process dates back to the Middle Ages, and the well-known Longmaid process, patented in 1849 and applied successfully to Spanish cuprififerous pyrites in 1860, is still in extensive use. For many years silver was extracted from its ores by various wet processes, beginning with the Augustine process devised in 1849 up to the highly efficient Russel process, based upon the solubility of compounds of silver in sodium-copper thiosulphate; these processes have only been abandoned owing to changes in the nature of the ores in the deeper parts of the mines, which have rendered them more suitable to treatment by smelting. The first wet process for the extraction of gold was the chlorination process, which was proposed simultaneously in 1849 by Plattner in Germany and by Percy in this country, and which was greatly developed in the sixties in California. Quite recently wet processes for the extraction of zinc from its ores by solution and electrolytic deposition have been used to some extent in this country, and have apparently been brought into successful operation in the United States and in British Columbia. Whilst wet metallurgical processes have then been employed for a variety of metals, they have until recent times been conducted on a relatively moderate scale, and the plant employed was of quite ordinary dimensions and presented no especial engineering problems. I prefer, therefore, to direct your attention to some of the very large-scale plants that have gradually been developed within recent years. I might cite for example the huge plant for leaching and electrically depositing the copper from copper ores containing only some 2 per cent. of metal, now being put into operation in Chile, and designed to treat 10,000 tons of ore daily. The best examples, however, for my purpose are to be found in the modern forms of the cyanide process of gold extraction, a process which has been in use for about a quarter of a century, and which has developed slowly from quite modest beginnings and has now reached a very high pitch of perfection. The details of this process are not perhaps very familiar to chemical manufacturers in this country, and it may be helpful to consider it briefly as illustrative of this portion of the subject that I am discussing.

The process has mainly been worked out in the Transvaal, though Australasia, the United States, and Mexico have all contributed to the elaboration of certain of the details, and have worked out methods particularly adapted to local ores or local conditions. The magnitude to which it has attained may be gauged from the fact that the Witwatersrand district alone cyanides over 2½ million tons of ore every month, whilst single units, like that of the City Deep, treat over 60,000 tons per month. The process, of course, varies considerably in its details, but the general Witwatersrand process may be taken as a typical example. The ore is crushed, generally in stamp mills, to about 30-mesh; in many cases the coarser particles in the pulp issuing from the stamp mill are separated in some form of classifier, depending upon the principle that in such a pulp the coarser particles will settle more rapidly than the finer ones, the coarser particles being then ground finer in tube mills; in other cases the whole of the pulp from the stamp mill passes through the tube mills. The pulp thus produced is streamed over amalgamated copper plates to take out all the free, readily amalgamable gold that it contains, and it is at this stage that the cyanide process proper commences. The pulp is led into some form of settler, a large rectangular or circular tank, or perhaps more often a conical tank about 8 ft. in diameter by 10 ft. deep, in which the coarser sands settle to the bottom, whilst the finer slimes overflow and are led to other appliances in which they are collected. These may be large circular tanks up to 35 ft. diameter by 12 ft. deep, in which there revolves slowly a vertical shaft carrying arms to which inclined blades are attached, which force the thickened pulp through a central aperture. Rectangular tanks are also used, one of the best known of which is the Dorr classifier, which is provided with scrapers moving over an inclined bottom, which steadily push up and discharge the settled material. One such machine will handle up to 200 tons (dry weight) per day, the appliance being quite automatic. The sands and the slimes are thus collected separately, and are each cyanided separately, the same treatment not being applicable to both, owing to the fact that liquids will percolate readily through the sands whilst slimes settle down in a practically impervious mass.

The sands are generally run into a collecting vat by means of some arrangement which distributes them evenly and uniformly, a set of rotating spouts known as the Butters distributor being very generally employed. Whilst the tank is filling with sands the surplus water is drawn off, any slimes in it being collected, and when the tank is full the sands are discharged into the leaching vats, which usually stand at a lower level than the collecting tanks. There are various means of discharging the sands, but it is usually done through bottom discharge doors. The percolating vats are practically always circular in plan, and are built either of steel plates or of wooden staves held together by iron hoops. They range up to 60 ft. in diameter by 15 ft. high, capable of holding 600 tons of sands, but as a rule their diameter rarely exceeds 40 ft. The lower part of the tank contains a frame-work of wood over which is secured a filter made of coconut matting covered with canvas, the filter being wedged tight against the sides of the vat. A series of solutions of cyanide are pumped on to the sands and allowed to percolate through them, and after sufficient leaching has taken place the sands are washed in the same way. I need not trouble you with the chemistry of the process, which is quite well known, as I am now only concerned with its mechanical aspect. It may be added that the quantities of solutions dealt with are quite considerable, being rarely less than 1½ tons per ton of sand treated, whilst they



may reach three times this amount. The exhausted sands are discharged in various ways; if the tanks are low down near the ground level, cranes and grabs may be used, but the general plan is to support the tanks on piers high enough to admit of trucks running beneath the former, when discharging is performed by means of doors in the bottom of the vats, connected, of course, to suitable openings through the filters. It need hardly be said that in view of the enormous quantities of material to be handled, mechanical appliances are used as far as possible, and the discharging is often performed by means of the Blaisdell excavator, a form of plough revolving on a vertical spindle that forces all the sands towards a central discharge door. The remaining portion of the process consists in precipitating the gold from the auriferous solution, which is usually done by running it through a series of boxes containing zinc shavings, upon which the gold is deposited, to be subsequently collected.

The percolation process for sands thus presents but little novelty in its mode of execution, the most interesting feature being undoubtedly the huge scale upon which the operations are conducted, far in excess of anything that has ever been attempted in any other chemical process. Witwatersrand alone treats 1,300,000 tons of sands every month. The case is different when we come to the slimes, which demand very different treatment, seeing that they settle so tight as to be practically impervious to water, and are not, therefore, amenable to simple leaching. This fact was for a long time an obstacle to their being treated at all, but gradually the methods now in vogue were evolved. The first successful process was one of agitation with the cyanide solution, followed by settlement of the slimes and decantation of the supernatant liquid, and this method is still in use where very low-grade material has to be treated. The slimes having been suitably thickened in one of the appliances already mentioned, or by being allowed to settle in large tanks 60 ft. in diameter by 20 ft. deep, are transferred into somewhat smaller treatment tanks and a weak cyanide solution is pumped in. The pulp thus formed is agitated either mechanically or by the injection of compressed air. A usual method consists in agitation by means of revolving stirrers and circulation by a centrifugal pump; in some cases circulation without agitation is found to be sufficient. A vat 24 ft. in diameter by 8 ft. deep holds about 25 tons of slimes (dry weight) which require about 100 tons of cyanide solution. After agitation has been continued for a sufficient length of time, the pulp is run off into a settling tank, where the slimes are allowed to settle as completely as possible, and the supernatant solution is drawn off. One or two washes are given in the same way, about 4 tons of wash-water being required to each ton of dry slimes. It can thus readily be seen that the quantities of solutions dealt with attain to an enormous bulk. The method of agitating by means of compressed air appears to have been evolved in Mexico, the so-called Pachuca tank being employed; this is a tall tank 10 to 15 ft. in diameter and 45 to 60 ft. in height, with a conical bottom; it holds up to 100 tons of slimes, and when the cyanide solution has been run in the pulp is agitated by forcing compressed air through a vertical tube in the centre of the tank; several modifications of this appliance are used, and Dorr has devised a tank in which agitation by revolving arms is combined with the use of compressed air.

The defects of this decantation process are sufficiently obvious, as the settled slimes must under any conditions retain a considerable amount of auriferous solution, so that attention was soon

turned to the possibility of employing filtration. The ordinary filter-press presented numerous objections for use on so vast a scale, prominent amongst these being its small capacity, the expense of working it, and the need for employing much manual labour. One form, known as the Dehne press, has been used to some extent; it contains about 50 chambers with plates 40 inches square, and is operated under a pressure of 3 to 7 atmospheres; such a press holds about  $4\frac{1}{2}$  tons of slimes, and can be filled and discharged in two hours, giving a capacity of about 54 tons per 24 hours; its main advantage is that it requires only one-quarter as much solution as does the decantation method. The Merrill filter-press presents the improvement that it can be discharged by sluicing out the contents without its being necessary to take the plates apart. Such a press with 90 plates 3 ft. 8 in. by 5 ft. 8 in. can treat 160 tons per 24 hours; it necessitates, however, an ample water supply, 4 or 5 tons of water being required for sluicing out one ton of slimes.

The special conditions attending the cyanidation of slimes have brought about the introduction of entirely new types of filters, some of which have reached a high stage of efficiency. One of the earliest of these was the Moore filter, which is still used a good deal. The principle of this filter consists in employing a number of "leaves," each of which is formed of a rectangular frame about 2 inches deep over either side of which is stretched a filter cloth; the hollow interspace between the two cloth surfaces is connected by pipes to a suction pump. When such a leaf is immersed in the pulp the solution is drawn through the cloth into the interior of the frame and thence into a receiver, whilst a coating of dry slime gradually builds up on either side until a sufficiently thick cake is formed. The leaf with the adhering cakes of slime is then lifted out and lowered into a tank containing wash-water, which is in turn drawn through the cakes of slime until this is sufficiently washed. The leaf is then lifted out and the adhering cakes of slime are thrown off by forcing air or water into the hollow space inside the frame. In practice the leaves are, of course, not operated singly, but in groups of 30 to 45, suspended from an iron frame, which is lifted or lowered by means of a crane. A group of 35 frames, 4 ft. 6 in. by 6 ft. 2 in., having a total filtering surface of 1836 sq. ft., worked with a vacuum of 18 inches of mercury, will deal with 4 tons of dry slime at a time, or about 40 tons per day. The Butters leaf filter is a modification of the same principle, the leaves being made of canvas stretched over a frame-work of iron tubing. These leaves are fixed in a tank which is filled alternately with pulp and with wash-water; when the washing is complete the cakes of slimes are discharged into the tank by water pressure and are generally sluiced out. Such a filter with 80 to 90 leaves, 10 ft. long by 5 ft. deep, takes a charge of 20 to 25 tons at a time, or treats 160 to 200 tons per 24 hours. This is one of the most satisfactory filters yet devised, and is largely used in the Transvaal. Out of one million tons of slime treated monthly on the Witwatersrand, Butters filter installations are now treating over half a million tons, so that this may be looked upon as a thoroughly well-proved appliance. In the Kelly filter, long, relatively narrow leaves are employed, which slide into a slightly inclined cylinder, the latter being about 5 ft. in diameter by 14 ft. long, and carrying 13 leaves, having a total filtering surface of 1200 sq. ft. Filtration is performed under a pressure of about 2 atmospheres; the capacity of such a filter may go up to 100 tons per 24 hours, and the final press-cake may be obtained very dry, retaining in some cases only 10 per cent. of moisture.



A filter used to some extent in Mexico is the Burt filter, which consists also of a horizontal cylinder either revolving or stationary, the cylindrical shell being lined with suitably arranged filter cloths. Slime and wash-water are forced in under a pressure of 2 to 3 atmospheres. Air is then forced in the opposite direction through the filters, thus stripping off the press cake. It is claimed that these pressure appliances work with a very small power consumption, less than required for the more usual suction presses. There is, however, no doubt but that one or other of the various types of leaf filter is by far the most generally employed. I may add that there are a good many other forms in addition to those that I have mentioned.

All the filters so far described share with the ordinary filter-press the drawback of being discontinuous in their operation; there have, however, been several continuously acting filters devised and used successfully. A good example is the Ridge-way filter. This consists of horizontal filtering leaves carried on arms attached to a revolving vertical hollow shaft; they are thus caused to traverse an annular trough divided into three compartments; one contains the slimes to be filtered, another the wash-water, whilst the third receives the exhausted press cake. The filter leaves, which carry a filter cloth on their lower surface only, run on an annular track; the interior of each is connected by pipes to a suction pump whilst it traverses the first two compartments. The track is so shaped that each leaf is in turn immersed in the slime tank until a sufficient thickness of slime cake has been formed, then rises out of it and dips into the next section, where wash-water is drawn through it, rises out of this again, and finally passes over the third compartment, where the slime cake is forced off by compressed air, after which it again enters the slime compartment, the operation thus being continuous. There are usually 12 arms, each carrying a leaf with a filtering area of 4 sq. ft.; the total power required is about 5 h.p., and the capacity is up to about 65 tons per 24 hours. This filter is quite extensively employed in West Australia.

The Oliver continuous filter is largely used in the United States of America. It consists of a hollow drum up to 15 ft. in diameter by 20 ft. long, mounted on a horizontal axis. The cylindrical surface forms the filter proper, and it is immersed down to the axis in a tank of slimes, in which it revolves at the rate of 6 to 12 revolutions per hour. As it traverses the slime the solution is sucked through, and a cake of slime forms upon the filter; on emerging from the slime tank the cake is washed by means of a spray, the wash-water being also drawn through the filter. Finally the adhering cake is loosened by forcing compressed air through the filter from the inside, and it is removed by means of a scraper. This machine is capable of treating up to 150 tons of dry slime per 24 hours.

There is no difficulty in dealing even with the enormous volumes of solutions in cyanide plants as long as these are tolerably clear, any ordinary type of pump being quite suitable for the purpose. Special methods have, however, been devised for elevating the thick slime and sand pulp. One such device is the "tailings wheel," built like a water-wheel with the buckets on the inner side of the rim; instead of the falling water rotating the wheel as in a water-wheel, the rotation of the wheel elevates the pulp; these wheels have been built up to 70 ft. in diameter, have large capacities, and are but little liable to get out of order. Special ram pumps are also used in which the ram is protected from the action of the sand by a film of clear water; another successful type for relatively low lifts is the Frenier spiral sand pump. Of the

ordinary forms of centrifugal and other pumps and air lifts there is no need to say anything, as these are of standard types.

My main object in putting before you this synopsis of the appliances that have been evolved in working out the cyanide process of gold extraction is to point out a field in which chemical engineering in the strictest sense of the term has been given free play, and has attained results of quite exceptional importance. I have thought it advisable to draw attention to the mechanical ingenuity that has been displayed in devising methods of dealing with such enormous quantities of both solids and liquids, these being methods and appliances which necessarily lie somewhat outside the experience of the ordinary chemical manufacturer, whilst the scale upon which the operations are conducted is one of altogether unprecedented magnitude. I venture to think that the chemical industry will not only have to evolve new engineering methods in the future, but will have to utilise to the fullest extent all the methods and appliances that have been devised in cognate industries. There can be no doubt that the future of the chemical industry, particularly of the heavy chemical industry, in this country depends essentially upon our being able to compete with our neighbours by producing at least as cheaply as they can, and I venture to suggest that this implies that chemical works will have in the future to be constructed on a scale of magnitude far greater than has been the case heretofore. Furthermore the processes will have to be conducted with a minimum of labour, because labour will in the future not only be scarce, but will be exceedingly dear. I for my part see no reason to expect that wages will ever come down to anything approaching their old level, nor have I any great desire to see them down again to that point, my ideal being rather a properly paid workman, better educated and more intelligent than his predecessor, and above all anxious to give an honest day's work in return for a good day's wage.

I have dwelt upon appliances requiring the minimum of labour because I feel convinced that such appliances must be employed in the chemical industry far more extensively in the future than they have in the past. The substitution of mechanical for manual power necessarily involves an increase in capital outlay, and although capital has gone up in value as well as labour, the latter has risen much more in proportion, so that there is still an important economic advantage to be gained by the introduction of labour-saving appliances. I feel convinced that the chemical industry, along perhaps with all the other industries of the country, can only survive in the fierce economic struggle which will undoubtedly succeed to the present conflict if we are prepared to organise our industries in such a way as to conduct all our operations systematically by means of processes and appliances involving the minimum of unskilled labour, though they may require no inconsiderable amount of intelligent supervision. Such methods no doubt imply a yet further consideration, perhaps to us in this country the most important of all—namely, the supreme necessity for having at our command sources of extremely cheap power. In the chemical industry, as in every other industry in Great Britain, this is the dominant factor; our national prosperity has been built up essentially on a basis of cheap power, that is to say, on an abundant and low-priced coal supply, and unless we can retain this advantage after the war our outlook for the future will be black indeed. Not only must we take care that we are able to produce coal at least as cheaply as any of our competitors, but also that we utilise it to at least as good advantage. Provided only that we are able to produce power at a reasonable cost, I have no



fear but that British ingenuity, with the powerful aid of the late-born recognition of the important part that scientific education plays in fostering and in applying it, will prove equal to any of the demands that the highly complex requirements of modern advanced industrialism may make upon it.

Dr. T. HOWARD BUTLER, in proposing a hearty vote of thanks to the President for his address, said that it was a great benefit and pleasure to hear such an able survey of a subject such as Prof. Louis had given. He assured the President of the hearty support of the Society. As a member of Council he could say that the large amount of business which the Society had got through during the past year had been not a little due to the admirable way the President had managed affairs.

Dr. F. W. RIXON, in seconding, especially thanked the President for his remarks on the question of the value of scientific research.

The vote was accorded with acclamation.

#### ELECTION OF COUNCIL.

The report of the scrutineers of the ballot for the election of officers and members of Council showed that the following were elected:—President, Prof. Henry Louis; Vice-Presidents: Dr. E. F. Armstrong, Prof. W. R. Hodgkinson, C.B.E., Robert L. Mond, and Walter F. Reid. Ordinary members of Council: Sir Robert Hadfield, F.R.S., Dr. F. Mollwo Perkin, W. J. A. Butterfield, and C. S. Garland.

#### ELECTION OF AUDITORS.

On the motion of Dr. J. C. Cain, seconded by Mr. F. W. Attack, Messrs. Price, Waterhouse and Co. were elected auditors to the Society.

#### VOTE OF THANKS TO UNIVERSITY.

The PRESIDENT expressed the thanks of the Society to Sir Isambard Owen and the authorities of the University for placing the University rooms at their disposal during the period of the meeting.

Sir ISAMBARD OWEN briefly thanked the President.

After the meeting the members were entertained at luncheon at the Grand Hotel by the Local Section, Dr. T. H. Butler being in the chair.

Wednesday afternoon, Thursday, and Friday morning were devoted to the reading and discussion of papers. The discussions on "Copartnership" and on "Industrial Efficiency" will be recorded in a later issue. The remainder of the papers are printed in this issue.

### ASPECTS OF THE LOW TEMPERATURE CARBONISATION OF COAL.

BY EDGAR C. EVANS, B.S.C., F.I.C.

The question of the low temperature carbonisation of coal is not a new one; the disadvantages of bituminous coal as a fuel have been recognised from very early times, and as far back as 1656 Evelyn described in his diary a "project by Sir John Winter of charring sea-coale, to burne out the sulphure and render it sweeter." The resulting "cinders" made a "cleare pleasant chamber fire, depriv'd of their sulphure and arsenic malignity."

In 1681 a patent was taken out by Becker and Serle for the production of pitch, tar and smokeless fuel from coal, while the Earl of Dundonald took out in 1781 a patent for making pitch, tar, oils, cinders, etc., from coal.

The early literature of the gas industry, from 1792 onwards, teems with references to low temperature carbonisation, but the gas engineer's desire for the utmost yield of gas from his coal

led to the adoption of higher and still higher temperatures, until to-day the highest possible temperatures compatible with the nature of the retort are universally used in this industry.

From the point of view of the gas industry, with gas as the primary product, this choice has been thoroughly justified by the results obtained, and the same thing can be said of the coking industry, where a hard metallurgical coke is the main desideratum.

Neither gas coke nor metallurgical coke, however, is an ideal substance for use as a domestic fuel, and in the modern sense of the term, the idea of low temperature carbonisation is the outcome of attempts that have been made to produce a smokeless fuel from bituminous coal.

In this sense, low temperature carbonisation can be said to date from 1890, when the late Col. Scott Moncrieff suggested the withdrawal of the charge from gas retorts when half the usual quantity of gas had been evolved, with the idea of using it as a domestic fuel. It was found, however, that the only result was the production of a partially coked layer on the outside of the charge with an inner core of practically raw coal.

A more feasible suggestion was made in the same year by Parker, who proposed (Eng. Pat. 67, 1890) to produce a smokeless fuel by passing steam, water gas, or coal gas, superheated to a temperature of 600° to 650° C., through a mass of coal in a retort. The idea of using an inert gas as a heating medium is an excellent one, but it failed in this as in subsequent attempts owing to the peculiar nature of the phenomena connected with the carbonisation of coal at these comparatively low temperatures.

In 1906, Parker took out his famous master patent (Eng. Pat. 14,365, 1906) for the production of "Coalite" by heating coal in the presence of steam to a temperature not exceeding 800° F. A number of retorts were tried, details of which are given in a series of patents taken out between 1906 and 1911. The first proposal was to use  $\Delta$ -shaped retorts about 5 ft. wide, 7 ft. long, and 16 in. high, the coal being placed in a layer not more than 6 in. thick.

Coal which melted under heat was treated in tapering cylinders about 10–12 in. in diameter, one end being perforated to allow the escape of the gases produced, or it was heated in completely filled tubes of not more than 6 in. in diameter.

These experiments failed, however, and the next step was to use a narrow vertical retort of oblong cross section, which again was replaced by bunches of vertical tubes, each tube being from 4 to 6 in. in diameter, and each separate battery of tubes being connected by means of a separate pipe with the hydraulic main. In a later patent, these tubes were provided with slots by means of which communication was established between adjacent tubes, so that if one of the tubes became temporarily choked, the gas evolved could pass up the other.

This gradual alteration in the design furnishes an interesting commentary upon the difficulties experienced by the Coalite company, and these proved so great, that despite the assistance of some of the best scientific brains of the country, and the expenditure of large sums of money, the process failed to satisfy commercial requirements.

The Coalite failure, however, served one good purpose—it created a popular interest in coal carbonisation, it showed the need that existed for a free-burning smokeless fuel, and cleared up many obscure points in coal carbonisation.

A considerable amount of work has since been performed on low temperature carbonisation both from a scientific and a technical standpoint, and, whilst commercial success has yet to be realised, a number of the problems involved have proved capable of solution on a semi-commercial scale.

The question of commercial success by any system capable of general application is not an easy one. The process of coal carbonisation itself is a most complicated one, and it is rendered still more complex by the wide differences that exist between different coals.

#### *Constitution of coal.*

The whole phenomena of coal carbonisation at low temperatures are intimately bound up with the constitution of the coal that is treated, and, in the solution of the problem, the most important requirement is a clear understanding of the nature of coal itself. Unfortunately this is extremely difficult to obtain by purely chemical methods. The brilliant researches of Wheeler and his collaborators have established the fact that coal contains two classes of substances, viz.:—Resinous substances derived from the resins and gums of the vegetation from which the coal was originally formed, and cellulosic or "humic" compounds derived from the cellulose of the original vegetation. Some of the conclusions of Wheeler and his co-workers are still the subject of debate, but the following distinctive properties of these two classes of substances are agreed to by most of the workers in this field.

*Cellulosic or "humic" compounds*:—(1) Are infusible. (2) Yield very small quantities of liquid distillates on being heated, these consisting chiefly of phenolic compounds. (3) Are insoluble in chloroform.

*Resinous substances*:—(1) Have a low melting point. (2) Yield a large proportion of liquid products on distillation, these consisting below 500° C. chiefly of paraffins, naphthenes, and members of the olefine series. (3) Are soluble in chloroform, phenol, pyridine, and certain other solvents. (4) Leave on heating to 500°, a pitch which acts as a binding constituent in the formation of coke.

At first sight, it might seem possible to consider all coals as being made up of varying proportions of resinous and cellulosic or humic material, and that the properties of any coal would depend essentially on the relative proportions of these two substances that it contains. Such a constitution would, however, by no means account for the differences found in different coals. In the first place, neither resins nor cellulose contain sulphur and nitrogen, and the organic sulphur and nitrogen compounds of coal would still require a place in the essential constituents of the coal. Secondly the properties of non-coking cannel and spore coals could not be reconciled very easily with a constitution based only on resinous and humic substances.

It is therefore essential to widen our ideas of the constitution of coal to include substances other than these two, or else to broaden the terms to include substances widely differing both in constitution and in properties. Unfortunately, purely chemical methods have exhibited very serious limitations in the examination of the exceedingly complex mixture of substances contained in coal, and a clearer conception of the rational constitution of coal can probably be gained much more easily by a combination of chemical and microscopical methods.

The pioneer work of Lomax, followed by the researches of Stopes, Hickling, and others in this country, of Renault and Bertrand in France, and of White, Jeffreys and Thlessen in America have thrown considerable light on the constitution of coal. It is impossible to go into their researches in detail, but essentially, in the light of the microscopic examination of coal sections, coal can be regarded as being made up of varying proportions of humic matter, resinous substances, and cuticular substances, the last-named embracing spores, spore cases, etc. (including probably the so-called algae noted by Bertrand and Renault in cannel), and the

resistant cuticles of the original vegetation. To these can probably be added the nitrogenous and organo-sulphur compounds in the coal which presumably would have been derived from the cell contents of the parent vegetation.

It is impossible in this paper to discuss the latter substances in detail, but, from the point of view of carbonisation it is of considerable importance to gain an idea of the nature of the cuticular material in coal. At first sight, it would seem probable that this would be resinous in character: under the microscope it appears as a bright yellow substance resembling very closely the resinous particles in the coal, but so far as the author is aware, the small amount of work that has been performed in this direction seems to indicate that cuticular matter in general is non-resinous in character, or at any rate, if it is resinous, it differs in many important respects from the customary resinous constituents.

In an unpublished paper read by Lomax before the Manchester Geological Society in 1914, it was shown that spore coals were non-coking in character, and this was confirmed by Lomax and the author in an examination of the Lantern Seam of the South Rhondda Colliery. This seam is highly resinous in its general character; when distilled at temperatures of 500° to 550° C., it swelled, fused, and gave a considerable proportion of liquid distillates that were not phenolic in character. When the seam was examined in detail, however, it was found that those portions which were made up of spore coals were non-expanding, non-fusible, and except in places where resins were clearly evident, non-coking in the customary sense of the term. It appears likely therefore that the cuticular ingredients of coal are non-resinous. On the other hand, they have properties that are different from the purely humic constituents. Spore coals, even though they are non-coking, yield a large proportion of paraffin compounds on distillation at low temperatures, and this fact, together with their resistance to decay, seems to indicate a non-cellulosic origin.

Unfortunately, very little detailed work has been done as far as the investigation of the cuticular portions of living plants is concerned, but apparently they consist of cellulose combined with fatty and waxy compounds of very high molecular weight. Such a composition would account for the properties possessed by spore coals, but the exact nature of the residual product as it exists in coal has yet to be determined.

#### *Theory of the coking process.*

As far as the coking property of coal is concerned, the predominant factor is undoubtedly the proportion and character of the resinous constituents that it contains. Parr and Olin (Bulletin 60, University of Illinois) as the result of a study of the low temperature distillation of Illinois coals, put forward the following hypothetical conditions as being necessary for the formation of coke:—

(1) There must be present certain substances which have a definite melting point; (2) the temperature of decomposition of these substances must be above the melting point; (3) when the compounds that satisfy the first and second conditions are unsaturated, it is possible by subjecting them to oxidation so to lower the temperature of decomposition that the second condition is no longer fulfilled, in which case coking will not occur. These hypotheses seem to agree very well with the known data concerning the coking process, and, while the phenomena involved are exceedingly complicated, a fair idea can be obtained with their aid of the reactions that take place within the coking chamber.

It is necessary at the outset to distinguish between high and low temperature carbonisation. In the



former case, not only is the reaction complicated by secondary reactions taking place between the liquid and gaseous products at temperatures above 700° C., but there is a considerable physical difference in the structure of the resulting coke product. The secondary reactions of the liquid and gaseous products have already been very ably dealt with, and it is therefore advisable in these notes to consider only those factors that are involved in the production of coke.

As far as coke formation is concerned, the use of a high temperature simplifies the process considerably and actually reduces the number of factors that have to be considered in large scale operations.

When a charge of bituminous small coal is introduced into a coking chamber the walls of which are maintained at a temperature of say 1000° C., the outer layers become rapidly heated until, at a temperature of 300° to 400° C., they soften owing to the fusion of the resinous substances. The temperature rises too rapidly for this condition to last very long, however, and gas begins to be evolved, the evolution increasing rapidly as the temperature rises, until finally the portions nearest the wall solidify. Gas still continues to be evolved from the solidified portion, and continues in fact until the coke reaches the temperature of the walls, and even then, at 1000° C., there is still left a small quantity of volatile matter which could only be expelled by raising the temperature considerably.

Meanwhile, the adjacent layers towards the centre of the oven have attained the pasty stage, and gas begins to be evolved which passes principally through the porous, solid, outer layers to the wall of the oven. (Cf. Lewes, "Carbonisation of coal," Young, J. Gas Lighting, 1912, 119, and Evans, J. Gas Lighting, 1913, 587.) A small amount possibly given off from the inner portion of the pasty mass, may pass upwards through the central core of raw coal, but this quantity would not be very great. As the gases pass through the outer solid coke, the hydrocarbons are decomposed to a certain extent with the formation of carbon which is deposited on the outer layers, thus forming a stronger, harder coke.

Essentially the coking process resolves itself into the formation of a pasty zone, which, fairly rapidly at first, but afterwards with continually decreasing speed, travels towards the centre of the oven. The rate of advance of the zone depends on the temperature of the oven walls and the thickness of the charge. The higher the temperature, the more rapidly does the fused zone pass towards the centre of the oven. In the later stages of the carbonisation the rate of progress becomes very slow. It has been shown by Simmersbach, that in a coke-oven 20 in. wide, the centre of the charge remained at a temperature of 10° C. for 2 hours after charging, then rose to 100° and remained there for 13 hours, while even after 20 hours the temperature was only 410° C. (cf. Ramsburg and Sperr, J. Franklin Inst., Apr., 1917).

As the coking process continues, the coke becomes fissured along lines perpendicular to the walls of the oven, and finally when the fused zones reach the centre of the oven, the resistance of the outer layers becomes greater than the resistance towards the centre of the charge, and a considerable proportion of the gas evolved passes up the centre of the charge, the coke dividing into two distinct masses.

#### *Low temperature carbonisation.*

In its main essentials, the process of low temperature carbonisation proceeds along similar lines to the above, but several of the factors involved are so intensified that a radical alteration in the design of the oven is necessary for the process to be carried out on anything like a commercial scale. The following notes summarise the main differences.

(1) The low temperature (450°–550° C.) of the walls of the oven reduces enormously the rate of transmission of heat through the charge, or in other words, it reduces the velocity of the zone of fusion. For this reason, a thickness of four to five inches is the maximum that can be treated in stationary charges within economical limits of time. This factor brings in its train the following results:—(a) The capital outlay is increased owing to the increase in the number of units, (b) labour charges are necessarily increased, (c) the space taken up by the plant is increased, and (d) the maintenance cost is increased.

(2) The resistance of the fused zone to the passage of gas is enormously increased as the temperature diminishes. It has been estimated by O. B. Evans (J. Gas Lighting, 1913, 587) that the resistance offered to the passage of gas at 540° C. is about 7 times greater than at 700° C. Owing to this extremely high resistance, if for any reason the outer zone becomes choked, the gas accumulates in the charge to such an extent that serious gas pressures are developed. In several cases the author has found that when dealing with resinous coals, the gas escapes from the central portion of the charge not in a direction at right angles to the containing walls, but parallel to them, so that the resulting coke appears as if it were built up of extremely thin layers. This seems to indicate that in low temperature carbonisation, the outer layers are much more resistant to the passage of gas than is the case in high temperature carbonisation.

(3) The time during which the coal is in a state of semi-fusion is considerably prolonged.

In the case of high temperature carbonisation the time-temperature gradient is rather steep, and the interval of time during which the coal is in a state of fusion is comparatively short. In the case of low temperature carbonisation, however, this period is considerably prolonged and owing to this prolongation of the pasty stage, with the high pressures that are induced in the coal mass, the cell cavities in low temperature coke are considerably larger than is the case with high temperature coke. This produces ultimately a considerable expansion in the coke, an expansion often great enough in the case of some resinous coals and with well filled retorts to choke up the gas outlet completely. With such coals a considerable space must be left in the retorts to allow room for expansion, and the economic efficiency of the process is thus seriously affected. Further, the coke becomes porous and friable.

(4) Any free space left at the top of the coal charge increases the amount of air that is left in contact with the coal. This exerts a most deleterious action when the coal is carbonised at low temperatures and results in the formation of a friable, powdery coke.

It is evident that the low temperature carbonisation of bituminous (coking) coal involves the solution of a number of problems many of which are of a most conflicting character. The records of the Patent Office contain a number of attempts to solve these problems and of these, three main classes can be distinguished, *viz.*:—

(1) The use of externally heated, intermittently charged retorts.

(2) The use of intermittently charged retorts, internally heated.

(3) Continuous processes, in which the coal charge is carried forward by automatic means through a retort which may be heated either externally or internally, or by a combination of both methods.

*Externally heated retorts.* The various types of "Coalite" retorts are on the whole typical examples of low temperature retorts. Their failure was due to the lack of detailed knowledge regarding the constitution of coal, and also to the difficulty



of adapting the system for carbonisation on a commercial scale. The Coalite trials proved one thing very clearly, however, and that was the necessity of carbonising the coal in layers as thin as was compatible with commercial requirements. This result is achieved in a very simple manner by the Tozer retort of the Tarless Fuel Company. In this retort, the coal is charged in concentric layers, so arranged that no layer is more than 4–5 in. thick. It is obvious that much larger charges can be got into the same space than in the coalite process, the coal can be charged much more readily, labour charges for handling are reduced, and the heating of the retorts can be made very uniform.

The retorts are used in conjunction with Simpson's process for heating coal under a vacuum of from 20 to 26 in. of mercury. The use of such a high degree of exhaustion has certain obvious advantages. The oxygen left in the retort after charging is reduced to a minimum, the liquid and gaseous products would be removed very rapidly from the retort and possibly distillation would be effected more readily. The influence of the vacuum on the quality of the coke produced is not clear. Porter and Taylor state (Tech. Paper 140, U.S. Bureau of Mines) that Pittsburgh bituminous coal yielded a light, inferior, porous coke when slowly heated at atmospheric pressure, but at a pressure of less than 30 mm. it produced a dense coke. The reason for this is not very clear, but apparently the use of a vacuum produced a decrease in the tenacity of the tar film.

On the whole a vacuum process offers certain advantages over those carried on at atmospheric pressure, but on the other hand, from a commercial standpoint it has certain disadvantages which are obvious to workers familiar with coal carbonisation on a large scale. These can be summarised thus:—(1) Increased capital outlay; (2) Increased power consumption; (3) difficulty of avoiding leakage (this would be a difficult matter under works conditions, especially when working on a big scale with unskilled labour in a colliery district liable to subsidence).

Taking the Tarless Fuel process as a whole, it is attractive in many respects, but it has yet to prove its capacity for satisfying commercial requirements on a big scale.

A process that has attracted considerable attention is that carried on by the Barnsley Smokeless Fuel Company. This differs in many essential aspects from customary low temperature practice, and it might perhaps be briefly dealt with. In the first place the Barnsley retorts are made of fireclay instead of cast iron, the usual material used in low temperature retorts. Cast iron is certainly not an ideal material; its disadvantages were early recognised by the gas industry and led to its substitution by clay retorts. In the Barnsley plant vertical retorts of rectangular cross section are used which are somewhat wider than those used in most low temperature processes. In these (Eng. Pat. 108,200) four varying zones of heat were maintained, the lowest being at a temperature of about 450° C., the next 500°, the next 550°, etc., whilst finally, in the free space at the top of the charge, a temperature of 900° to 1200° C. was maintained. In this space was suspended a grid made of some suitable material (metal, metal oxide, fireclay, or carbon) so that the gaseous compounds of distillation were subjected over as great an area of contact as possible to the temperature necessary to convert the paraffinoid tars to aromatic hydrocarbons.

The author has had no experience of this plant, but from purely theoretical considerations the chances of success would be small if the above temperatures were adhered to. In the first place, the retorts are wider at the bottom, so that the

zones maintained at the lowest temperatures are actually wider than those at higher temperatures. Thus the top portions would be carbonised much sooner than the lower portions, so that assuming that the lower layers could be carbonised in economic limits of time (which is doubtful) the result would be the formation of a mass of coke of very uneven quality, the lower layers spongy and porous, whilst the top layers would probably be difficult to burn. It is also difficult to see how the dangers of a serious accumulation of gas could be avoided in the lower portions, and the conditions in this respect would probably be worse even than in the "Coalite" retorts. However, the author has had no experience of the process and it would be interesting to learn something of the results that have been obtained.

A number of other examples of low temperature retorts could be given, but, taking them as a type, they possess the following disadvantages, arising mainly from the necessity of having to work with charges of coal that are neither too wide nor too high. The number of units must be considerably greater than is the case in high temperature practice, and this involves increased capital outlay, increased labour charges, increased repair costs, and a lowering of the general economic efficiency of the plant.

*Internally heated retorts.* In this type, the coal charge is heated by the actual passage through it of inert gas preheated to a temperature sufficiently high to carbonise the charge. On purely theoretical grounds, this proposal is extremely attractive. The bulk of the time occupied in present systems of carbonisation is taken up in heating the innermost layers of the coal, and if these could be heated from the outset considerable economies in time could be effected. A considerable number of attempts have been made to carbonise coal by passing through it a current of inert gas heated to a temperature of from 400° to 600° C., and Parr and Olin (Bull. 60, Univ. of Illinois) had some excellent results on a small scale by this method.

As far back as 1890, Parker (Eng. Pat. 67, 1890) proposed to pass steam, water-gas or some other suitable gas superheated to 500°–600° C., with a view to making smokeless fuel, and a number of other inventors have followed along similar lines. In the case of bituminous (resinous) coals, the old difficulty arises that when the coal reaches the pasty stage, it becomes impervious to the passage of the gas, but there seems to be no reason why the method should not be used for shales, cannel, or for coals that are not fusible.

The results obtained by McLaurin with a process of this type (this J., 1917, 620) are extremely interesting. As would be expected, cannel coal proved to be quite easy to work when carbonised by means of a stream of hot producer gas, but it was also found that Cadder coking coal if screened came out of the retort in the same shape and same size as it was put in. McLaurin suggests that this is due to the slow heating to which the coal was subjected and that under those conditions it did not intumescence. If this condition is applicable to all coals, it opens up possibilities of an extremely interesting character. The author, however, has not found it possible to repeat this result with the highly resinous coals of South Wales, except under conditions in which oxygen was present in the heating gaseous medium. The effect of oxygen when coal is carbonised at low temperatures has already been discussed, and the author is inclined to believe that the small quantity of oxygen which would be present in the hot producer gas in McLaurin's experiments played as important a part as the slow heating. A typical analysis of the producer gas gave 0.9%



of oxygen, so that there seems to be every reason for believing that the coal was carbonised in an atmosphere containing a small proportion of this gas, an idea which is confirmed by a study of the properties of the tars obtained.

Another proposal of this type is that given by Lamplough (Eng. Pat. 108,343, 1917), the heating medium in this case being steam.

On the whole, internal heating seems to offer considerable possibilities in the treatment of shales, cannel, and possibly certain types of coal if carefully screened, but as McLaurin and others have found, difficulties would arise in the carbonisation of fine coal in this way. There is perhaps one point that would need consideration, and that is, if a small proportion of oxygen were systematically introduced into the heating gas, what would be the effect upon the tars produced? These apparently would have a similar composition to the old Jameson tars, which were produced under conditions theoretically analogous to those existing in McLaurin's process.

*Continuous processes.* The third class of retort which has been proposed for the low temperature carbonisation of coal is that in which the coal is carried forward through the furnace by means of a conveying system.

The Archimedean screw has been suggested as a suitable carrier for quite a long time. A most interesting anticipation of one of the most modern of these systems is given in Patison's Eng. Pat. 569, 1873, in which coal, shale, or other fuel is carried through a heated retort by means of a worm conveyor. For low temperatures—with a high oil yield—the inventor proposed to use a cast iron retort, but for high temperatures he suggested the use of a fireclay furnace.

The well-known Del Monte system differs only slightly from this system, but it had one innovation, and that was in the use of a screw mounted on a hollow shaft, which was heated internally by a row of jets from a central gas pipe. Further, the coal was subjected to a fractional distillation, one end of the retort being maintained at a considerably lower temperature than the other. This retort again could be used for wood, shale, lignite, or peat, but with coking coals, the swollen pasty mass which was produced completely prevented the working of the screw.

It seems difficult to believe that any system which involves the stirring up of the coal in any way can be used for bituminous coals, and for that reason, it is doubtful whether a continuous system on the lines of the Woodhall-Duckham or the Glover-West retorts can be used for the low temperature carbonisation of these coals, except perhaps under conditions already touched upon.

On the other hand, there seems to be no reason—apart from those connected with engineering difficulties—why the coal should not be conveyed through a furnace, by some system in which the coal itself is not stirred.

A system of this type that has been worked with some degree of success, is that introduced by Pringle and Richards. In this the coal is carried by means of an endless conveyor through a retort the temperature of which is maintained at about 500° C. The conveyor itself is fitted with compartments so as to subdivide the coal into a number of separate small charges each of which is rapidly heated to the desired temperature. The coal itself is undisturbed in its passage through the retort, and leaves as a porous semi-coke which is an excellent domestic fuel. This process as yet has only been worked on an experimental scale; the obvious difficulties are of an engineering character and whether or not they can be overcome on a large scale plant remains to be proved.

A system of this type seems to offer several advantages for low temperature carbonisation: temperature control could be readily effected, labour charges would be low, and by suitable engineering methods the rather heavy capital outlay and high maintenance and repair costs which would probably be inherent in this particular system could be reduced to a limit which would make it a commercial proposition.

A number of other proposals could be given, but as yet, not one of them has been tested on a commercial scale. As far as the author can see, there does not seem to be any overwhelming difficulties in the way of the low temperature carbonisation of shales, lignites, cannel, etc., whilst for resinous coals, success will probably be attained only by an individual study of the coals it is desired to treat, accompanied possibly by a suitable preliminary treatment which will give a final product of sufficient solidity and strength to withstand customary conditions of handling and transport. In this connection the briquetting of the coal either before or after carbonisation will have to receive very serious consideration. The main considerations in this direction are economic in character and in the present artificial conditions prevailing in the coal market it would be extremely difficult to estimate the commercial possibilities of such a combination.

*Economics of low temperature carbonisation.* An attempt might, however, be made to review from a general standpoint the financial possibilities of low temperature carbonisation, and this perhaps is all the more necessary because of the extravagant estimates that are periodically issued from certain sections of the financial press. In this connection it must not be overlooked that there are many coals which are almost unusable in the raw state, and in some cases even there are whole coalfields in which the coal is of such quality that under the economic and geographical conditions prevailing in their immediate vicinity they are commercially unworkable. Several such cases have been brought to the author's notice in which a well designed system of low temperature carbonisation would result in the economic salvation of the coalfield. Disregarding for the present these exceptional cases, there remain two or perhaps three main directions in which the adoption of low temperature carbonisation has been strenuously advocated.

The first is in the treatment of cannel coal. From a technical standpoint this does not appear to present any very serious difficulties, and the question practically resolves itself into a commercial one. The matter has already been fully treated recently ("A New British Oil Industry," Craig, Perkin, Berry, and Dunstan, J. Inst. Petrol. Tech., Apr., 1918) and need not be discussed here. In this case, however, the utilisation of the residue is the factor upon which the success or failure commercially depends. The method usually suggested is to gasify the residue in producers of the Mond type and recover the ammonia produced (see Paton, Iron and Coal Trades Review, Mar. 22, 1918, and "Fuel Economy," paper read before Birmingham Section, Inst. Elect. Eng., 1917). Technically such a process would be ideal, especially if the carbonisation and gasification could be performed in one plant as proposed by McLaurin (this J., 1917, 620) or Wollaston (Eng. Pat. 111,964, 1918); there is one point which needs to be emphasised, the necessity for considering the Haber process as a factor in the reduction of price of ammonium sulphate after the war. It is estimated by the Barrett Company of New York, that the production of ammonia from this process alone amounted in 1917 in Germany to a quantity

equivalent to over 1,000,000 tons per annum of ammonium sulphate. The world demand for this fertiliser is increasing rapidly, and there is every prospect of a still further increase during the next few years, but with a competitor of this magnitude in the field, there is every need for caution in considering the claims of the recovery producer against the cheaper and technically more efficient non-recovery producer.

There is one other point that can be only briefly dealt with, and that is the relation of the gas industry to low temperature carbonisation. In the author's opinion, the gas engineer, to whom gas is the main and primary product, is quite justified in adhering to high temperatures in general practice. The yield of gas in low temperature carbonisation is far too low to make it attractive to the gas engineer, and with humic coals this small quantity is seriously contaminated with carbon dioxide. Further, to attain a total yield comparable with present practice, the capital outlay would be considerably increased, the space required for the plant would be very much greater than in present practice, and the labour costs (per 1000 cub. ft. of gas) would be enormously increased.

There are of course advantages in favour of low temperature practice, but in view of the standardised conditions prevailing in the gas industry, with works already cramped for space in many cases, and with heavy capital charges on plant already installed, it is extremely difficult to see where its advantages come in. Gas coke is certainly not an ideal product either from a domestic or an industrial standpoint, but by suitable preliminary treatment of the coal used, even this can be made an excellent free-burning product; and if the market conditions allow, the gas engineer can always erect additional plant for this purpose. Apart entirely from any preliminary treatment, however, the uses of gas coke are rapidly extending and before long it should cease to be the drug on the market that it has been in the past.

Taking everything into consideration, the gas engineer is well advised in rejecting low temperature carbonisation as being unsuitable for the purpose of producing gas in as great a quantity and of as high a quality as possible.

There remains the last and greatest aspect of the question—the low temperature carbonisation of bituminous small coal. The total quantity of coal raised in the United Kingdom in 1916 was approximately 256,000,000 tons. Of this, 37,600,000 tons was carbonised yielding something over 8,000,000 tons of gas coke and 13,200,000 tons of metallurgical coke. Of the remaining 218,400,000 tons, allowing  $4\frac{1}{2}$  million tons of anthracite and say 30 million tons of low volatile coals, there remains nearly 184 million tons of bituminous coal, of which over 60,000,000 tons would be small coal.

The use of raw coal was characterised over thirty years ago as a barbarous procedure; the statement is true to-day, but it has gained an enormously greater force as the result of our experience during the past four years. Disregarding for the time being the idea of carbonising large coal as being too great a feat of mental gymnastics for the average commercial mind to attempt, there remains the fact that there is available in this country approximately 60 million tons per annum of bituminous small coal, all of which, under certain well-defined conditions, can be carbonised to leave a coherent, solid, free-burning smokeless residual fuel, which in many respects is superior to the large coal from which it is produced.

With this as a basis, and with the further consideration of the necessity for this country to be self-supporting in the ever increasingly important

matter of liquid fuel, it is evident that there is an enormous scope for the introduction of a successful system of low temperature carbonisation. The main question unfortunately is, will it pay?

Under present conditions of scarcity of coal, of steel, of raw materials and of labour, it is an extremely difficult matter to draw up an estimate which would hold good for possibly more than a few months, but a provisional estimate can be attempted (using pre-war figures) which might serve as a basis for future guidance.

#### *Cost of plant.*

This would depend on the type of installation that would be found serviceable. At first sight a low temperature plant working at only 500° C. would be less costly than a coke-oven plant working at 1000°. If the stationary type of retort were erected, this factor would be counterbalanced by the greater number of units found necessary, whilst the price of a continuous retort would be increased owing to the engineering devices found necessary. The cost of a 300 tons a day coking plant before the war would be approximately £50,000 to £60,000, and this figure has been taken as representing the capital outlay.

*Coal.* The value of the coal and the resulting fuel would be interdependent; for that reason, the price of the coal has been arbitrarily fixed at 10s. per ton of unwashed small.

*Labour* is figured at 2s. per ton of coal carbonised.

*Power, steam, etc.* This figure would again vary with the type of plant erected. In a vacuum plant the power consumption would exceed that of a coke-oven plant, and the same thing would apply in the case of certain types of continuous plants. For purposes of estimation the figure of 4d. per ton has been taken as the cost of steam and power.

*Repairs and maintenance.* The advantages of working at low temperatures would in this case again be counterbalanced by the increased number of units. In the case of continuous plants this figure would probably be high, but it would be counterbalanced by reduced labour charges.

*Depreciation and interest* is assumed to be 15%.

*Stores, etc.* A figure of 2d. per ton has been assumed.

The following is an estimate of the expenditure in a plant carbonising 300 tons of coal per day. Assuming 300 working days a year, 90,000 tons of coal per annum would be treated.

*Coal washing.* Assume unwashed coal at 10s. per ton. Cost of washing 6d. per ton. Assume 25% loss in washing. Then cost of coal to ovens=14s.

<i>Expenditure.</i>	<i>£</i>
Coal, 90,000 tons at 14s. ... ..	63,000
Labour ... ..	9,000
Repairs and maintenance ... ..	1,500
Steam and power ... ..	1,500
Interest and depreciation .. ..	9,000
Stores ... ..	750
	<hr/>
	£84,750

*Receipts* would be made up of the values of the coke, tar, sulphate of ammonia, and light spirit produced.

*Coke.* The character of the resulting smokeless fuel would depend on a number of factors, the most important being the nature of the coal used, temperature of carbonisation, time of carbonisation, thickness of coal charge, type of retort used, and gas pressure under which carbonisation is effected. Of these the first is easily the most important. As a general rule the coke from highly



oxygenated coals (humic coals) has a higher calorific value than the coals themselves, while with other coals the calorific value is lower. Further, humic coals yield a friable powdery coke, whilst resinous coals yield a porous coke which is, however, fairly strong if carbonised under suitable conditions.

By judiciously mixing the coals used an excellent product can be obtained which has qualities which make it superior even to the large coal. In most low temperature schemes it is assumed that the resulting fuel has a value (on a pre-war basis) of from 25s. to 35s., but if low temperature carbonisation were adopted on a large scale it is extremely doubtful whether the price would be much greater than that of the large coal.

As a general rule the difference in price between large and small coal varies from 7s. to 10s. per ton. With the small coal in this case at 10s. per ton, and allowing for the fact that it has been washed, it is perfectly justifiable to take the value of the resulting fuel at 20s. per ton.

For purposes of this estimate it is assumed that the yield of large fuel is 70%, together with 10% of breeze. The breeze could probably be reduced by the preliminary preparation, but even then it would be higher than is the case in coking practice.

The value of the breeze is taken as being equal to that of the initial unwashed small coal.

*Tar.* The value of this substance is difficult to ascertain under present conditions. Further, the cost of purification is difficult to calculate. The process of purification will have to be worked out very carefully, otherwise the percentage losses would be very high. Taking its constituents in detail, however, the probability is that the substance will prove to be a valuable product. Tar acids to-day are, of course, at a high figure, but even after the war there will be a heavy demand for creosoting materials, and this constituent of the tar will therefore probably prove valuable. The light oils and middle oils of the tar would probably attain a fair price owing to the growing demand for petroleum products of all kinds. The value of the pitch is at present an unknown quantity. With its freedom from so-called free carbon and a constitution more nearly approaching oil pitch than coal-tar pitch, there seems to be every prospect of its finding a ready market. Taken altogether, the value of low-temperature tar would probably prove to be at least as high as that of high-temperature tar; it may easily be higher, but to be on the safe side a value of 2d. per gallon has been assumed, with a yield of 15 gallons per ton of coal carbonised.

*Light spirit* should prove an excellent motor spirit. A yield of 2 gallons a ton is assumed, and a value of 1s. per gallon.

*Sulphate of ammonia.* The yield of this substance varies considerably with different coals, and rapidly rises after a temperature of 500° C. is passed. As far as the author's experience goes, the yield of ammonia at 500° C. is considerably lower than the 20 to 30 lb. per ton of coal usually quoted, and a figure of only 10 lb. per ton of coal has therefore been taken for purposes of this estimate.

*Gas.* The yield of gas at 500° C. is usually given as 5000 cubic feet per ton. This again is considerably higher than the quantity found by the author with most coals. Humic coals sometimes yield large quantities of gas at low temperatures, but this is often contaminated with high percentages of carbon dioxide. On the whole it is better to assume a gas yield sufficient to heat the oven, but insufficient to give any surplus.

On the basis of these assumptions the revenue side of the accounts gives the following figures:—

#### Receipts.

	£
Coke, 63,000 tons at £1 ... ..	63,000
Breeze, 9000 tons at 10s. ... ..	4,500
Tar, 1,350,000 gallons at 2d. ... ..	11,250
Light spirit, 180,000 galls. at 1s. ... ..	9,000
Sulphate of ammonia, 400 tons at £10 per ton ... ..	4,000
	<hr/>
	£91,750

Profit per annum, £7,000.

The resulting profit can scarcely be described as startling, and it certainly does not attain the glowing dimensions generally promised in the initial prospectuses of companies interested in low temperature carbonisation.

On the other hand the figures are distinctly conservative, and err probably on the low side. There is also ample room for improvement on both the debit and the credit sides of the account. Reduced labour and capital charges would make a great difference to the cost sheet. On the other side also there is ample scope for considerably improved figures. As far as the carbonisation of bituminous coal is concerned, the author is not inclined to fix too high a value for the tar. The profits will be made primarily on the solid fuel; the Coalite process certainly showed that there was a demand for smokeless fuel, and if the country wants this fuel it will certainly pay for it. There is no reason also why the temperature should be limited to the 500° C. assumed in the above estimate. This figure has been selected because the solid residue at that temperature is a free-burning, easily ignitable product. But, under certain conditions, the carbonisation temperature could be increased considerably beyond 500° and still leave an excellent fuel. The finest domestic fuel ever tested by the author had been carbonised at temperatures up to 800° C., and contained only 4% of volatile matter. The author is of opinion that the logical line of development in coal carbonisation lies in the direction of a fractional distillation of the coal, the tars being extracted at a temperature sufficiently low to avoid the possibility of secondary reactions, and the temperature afterwards raised to drive off the ammonia and a large proportion of the gas. Such a procedure would not only increase the yield of by-products, but would result in a firmer and denser coke with a consequent considerably reduced proportion of breeze.

Lastly there is the question of the coal used. The estimate has been based on the use of high-grade small coal (an unwashed small coal fetching a pre-war price of 10s. per ton can certainly be considered a fairly good product). There are coals (like Yorkshire unwashed smudge, for example) which sold in pre-war days for considerably less than this figure, and in such cases low temperature carbonisation offers a prospect of an excellent financial return.

Coals from coalfields of the type represented by the South Crop of the S. Wales and Monmouthshire coalfield are excellent in many respects; they are highly resinous, they possess excellent coking properties, they can be cheaply mined, and are situated in an area offering exceptional facilities for export. But unfortunately the coals of this area suffer from certain serious disadvantages. The higher seams contain a high percentage of sulphur, the lower ones are high in phosphorus—two factors which militate seriously against the use of the coal for the production of metallurgical coke. Further, the coal as mined contains an excessive proportion of small coal, a fairly average figure being 30% of large and 70% of small, the latter containing a large



percentage of duff coal. In a case of this kind the advantages of low temperature carbonisation for the primary purpose of yielding a smokeless fuel are obvious; the perfection of a commercially successful system would immediately render feasible the commercial development of this section of the coalfield.

Taking everything into consideration, the subject is one that is worthy of the serious attention of the industrial chemist. A system capable of dealing on a commercial scale with all kinds of coal may perhaps not yet have been perfected, the problems involved—and they are serious problems—may not have been completely solved, but, with a detailed preliminary study of the coal which it is proposed to treat, with an accurate knowledge of the conditions involved in its carbonisation, and with the whole-hearted co-operation of the chemist who investigates those conditions with the engineer who has to meet them, there is no reason why this last resting place of so many lost hopes should not attain an ultimate success which will give it a position of predominant importance in the mining and chemical industries.

#### DISCUSSION.

Dr. E. F. ARMSTRONG said that he feared that progress in this subject had perhaps been retarded by prejudice. The whole question of low temperature carbonisation had been the subject of such fierce controversy that it might have had some effect on its progress. Insufficient attention had been paid to the scientific basis of the subject. The question was an economic one. The monetary prize for success was small and not very attractive, and it was highly satisfactory that one of the first steps of the new Council of Industrial Research should have been to start a laboratory for the investigation of these problems.

Mr. W. J. A. BUTTERFIELD said that, as the author pointed out, it was almost impossible to say that any one type of retort or process would deal with the many varying types of coal and cannel that had to be dealt with. That applied particularly to the solid residue. The caking coals undoubtedly needed from the standpoint of fuel a different type of retort to a non-caking coal. The continuously fed retort, such as the Del Monte, could only be applied with some modification to caking materials. The disadvantage of the residue from caking coals was its bulkiness; it shared that with gas coke. All the apparatus used with it needed to be almost double the size. When using it for hot water pipes, for instance, an ordinary anthracite stove of half the dimensions would do the same work as one fed with coke. The same applied to transport, wagons, storage space, and handling. He did not think sufficient attention had been paid to the possibilities of utilising the low-ash non-caking coals of the Midlands where there was a powdery residue which it was quite possible to briquette into a material as dense as coal forming a very good domestic fuel. Moreover these were easier to handle in the carbonising plant. The temperature zones that were adopted in the Barnsley retort were very near to gasworks practice, and it was a curious commentary on the previous history of low temperature carbonisation processes that it should have been necessary to use high temperature zones in order to convert the paraffinoid hydrocarbons into aromatic hydrocarbons because of the greater stability of the latter. Some of the spirits that had been produced by low temperature carbonisation were entirely unstable, and became gelatinous after a short time. There were undoubtedly many cannels which it would pay to carbonise for the sake of the oil yield, disregarding the residue altogether. From the fuel standpoint, the prospects

of utilising residues in such cases in ammonia recovery plants after the war were not very great, but there were other cases where the coal was high in nitrogen, such as the very large amount of small coal at the pit's mouth, which it might pay very well to use in agricultural countries.

Capt. C. J. GOONWIN said he had no doubt that pulverised coal would play a very important part in the future. The semi-carbonised fuel produced in low temperature carbonisation plants was extremely bulky and somewhat friable, and he believed that in the transport of this material there would be a considerable amount of dust which would be valueless as far as its use for domestic purposes was concerned. The possibility of collecting this dust in central places and using it industrially as powdered fuel seemed to present a good future if low temperature carbonisation were more generally adopted. He suggested that some experimental work should be done on the use of powdered coal in the Bonecourt boiler.

Dr. F. MOLLWO PERKIN said that low temperature carbonisation work was rather more forward than the paper indicated. For example, the Government was putting up near Nottingham a battery of retorts on the Del Monte system. That process, as the author had said, could only be used on non-caking coal. One great objection to the screw system was that in order to carry the coal forward the screw had to be put very close against the edge of the retort, consequently a very fine residue came through at the other end and the only way to work that satisfactorily would be to briquette it. The Tozer retort, in which a vacuum was used, would deal with any class of coal, either caking or non-caking, and it was rarely that he had had any trouble in getting coal out. It swelled up quickly and he thought that the action of the vacuum caused the heat to pass through the coal more rapidly and it gave a really good fuel. With regard to the friability of smokeless fuel, he had recently had a consignment from the Tarless Fuel Co. which had been three weeks on the railway in frosty weather, and it only contained about 10% of breeze, and even this was not wasted because it could readily be briquetted. The Pringle-Richards retort had been considerably improved lately, and it would probably turn out to be a commercial success. It really was a grate conveyor. The coal was put into a V-shaped arrangement about 8 inches across at the top and 4 inches across at the bottom. At one time the difficulty had been that the coal held up and would not discharge. Now a slit was made in the V so that it opened out and allowed the coal to discharge. One of these retorts which he had seen recently had a capacity of 12 tons in 24 hours. It was 30 feet long and one yard across. Another retort which had not been mentioned was Lamplough's, in which, instead of using an inert gas for heating the coal, superheated steam was used; but he was not certain that, as no external heat was used, this system would be a commercial success. In this country we required large quantities of benzenoid hydrocarbons, which could not be obtained from low temperature carbonisation, and therefore we must depend on gasworks for them. At the present moment the navy needed fuel oil and that was why vertical retorts were being used for carbonising cannel coal. He thought that Mr. Evans was very conservative in putting the yield of tar at 15 gallons, because coal containing about 26% of volatile matter would give at least 20 galls. of tar provided the plant was working properly, whilst in certain cannels he had obtained as much as 80 gallons of tar although only a portion of it could be used for fuel oil. About 30 of the 80 gallons would be suitable for fuel oil, and there would be up to 10% spirit and



small quantities of oil which could be used for lubricating purposes. Reference had been made to the Barnsley plant, which was called a low temperature plant, but if benzenoid hydrocarbons were wanted, why not start at a high temperature? He thought that the process of working in stages would give an infinite amount of trouble and quickly block up the tops of the retorts with carbon. He had made some experiments in cracking oils in this way, and although it was possible to obtain benzenoid hydrocarbons, the yield was very poor and the apparatus was soon blocked up with carbon.

Mr. W. THOMSON, referring to the problem of smoke abatement in large towns, said that it would pay to charge very high prices for a smokeless domestic fuel. Was it possible to take, say, 500 cub. ft. of gas out in the ordinary way from the coal and leave a coke which could be used for domestic purposes?

The PRESIDENT said it was true, as Dr. Armstrong had said, that low temperature carbonisation was approached by most people with considerable prejudice. As an old coke maker in the days of the Beehive oven, he had more than once sacked a workman for producing too large a quantity of black ends in his oven. These old prejudices were really difficult to overcome. The question of the smaller non-eaking coal had been referred to. In addition to the small that was brought out of the mine, there was an immense amount of small coal left behind in the mine. He had seen mines in which they loaded the coal with  $1\frac{1}{2}$ -inch forks, and left everything less than  $1\frac{1}{2}$ -inch in the mine. In this way an enormous quantity of exceedingly valuable fuel was wasted. The authorities were afraid of the powerful coal miners' union and dared not tell the coal miner to load up everything that was underground. The result was that millions of tons of valuable fuel were left underground and wasted to the nation. This material would be exceedingly well adapted to low temperature processes. With regard to subsidence affecting plant, whenever a coal mine was laid out a large block of coal, which the miner called the shaft pillar, was left unwrought, and all the plant and buildings that were required for working the colliery were erected on that pillar, which was large enough to prevent any risk of surface damage. He could hardly conceive any rational engineer putting his coke ovens anywhere else than within the area protected in this way, and any structure of that kind supported on that shaft pillar was absolutely safe.

Mr. EVANS, in reply, said that the bulk and friable character of the low temperature coke were the crux of the whole question. What was being attempted now was to standardise the product that entered the retort. If coal were carbonised in a tube at a low temperature a structure was obtained which showed a central hole in a series of rings fairly closely compressed at the outside and gradually decreasing in density towards the centre. By a suitable preliminary treatment it was possible to get a solid block right through the mass without any hollow centre. With regard to the more recent developments mentioned by Dr. Perkin, he agreed that the Pringle-Richards and the Tarless Fuel Company's processes gave good results. In many respects the fuel was non-friable, and the only point was whether these processes would work on a large scale. With regard to the President's remarks concerning the shaft pillar, he was thinking of existing conditions at his own collieries where practically the whole shaft pillar was occupied by buildings. There was very little space left on the pillar for a low temperature plant, and he had no doubt that some of the older collieries in the country were in the same position.

## NOTES ON THE COMMERCIAL FRACTIONAL SEPARATION OF BENZENE, TOLUENE, AND XYLENES.

BY T. HOWARD BUTLER, PH.D., M.Sc.,  
AND F. J. W. POPHAM.

It is beyond the scope of this paper to describe the various plants available for the separation of coal-tar hydrocarbons; it is sufficient to say that efficient plant of British manufacture is now obtainable. The following notes on some of the factors that make for efficiency in working such apparatus may be of interest.

1. *Efficient column* is essential and may be attained by any means so long as intimate contact with vapour and refluxing liquid is obtained.

2. *Efficient dephlegmator*. Dephlegmation plays a most important part in good fractionation. S. Young in his book on "Fractional Distillation" describes dephlegmation as a means for separation brought about by the vapour bubbling through the condensed higher boiling liquid. On the other hand he calls the apparatus on top of a column through which the vapour passes a "constant temperature still head." It is usual now to discard these terms in practice and refer to the former as the fractionating column, and to the latter as the dephlegmator.

By careful regulation of the temperature of the water in the dephlegmator it is possible to increase very materially the efficiency of any plant. This is due largely no doubt to the fact that by this means a larger quantity of condensed liquid is returning down the column, thus affording better contact between vapour and liquid. In regulating any plant it is advisable to work as much as possible by altering the flow of water in the dephlegmator means of open steam, but even then high pressure to the coil in the still.

3. *Constant steam pressure* must be maintained, otherwise regulation of the temperature of still contents is almost impossible. A valve to reduce the pressure of the steam to the minimum usually obtained at any time is a material help in this direction.

4. *A high steam pressure*. A steam pressure sufficiently high to enable the toluene to be distilled off should be available; otherwise a partial vacuum must be put on the plant, which is never so effective. The xylenes may well be distilled over by means of open steam, but even then high pressure steam is preferable.

5. *Tight steam-heating coils*. Should the coils in the still leak even to a very small extent the separation will be entirely upset, and under these conditions it is often impossible to obtain pure products.

6. *Efficient water supply to dephlegmator* requires careful supervision. A works supplied with hard water will soon lose efficiency owing to the fact that the dephlegmator becomes badly scaled; in these cases a water-softening plant is necessary.

7. *Protection of column from external changes of temperature*. In plants put up in the open the columns must be protected against wind and rain, otherwise irregularity in speed of running will result.

The quality of the crude benzol available to the refiner varies very largely. The average, taken from a great number of consignments of "60/65 benzol" from producers, all of which have come to the knowledge of the authors, may be taken as:—

	Average.	Maximum.	Minimum.
	%	%	%
Benzene ... ..	45.0	61.1	34.7
Toluene ... ..	14.2	19.1	7.7
Paraffins ... ..	3.3	9.4	Nil
Xylenes ... ..	10.3	20.4	6.7
Heavy naphtha ...	7.3	16.0	4.0
Creosote ... ..	13.2	18.0	7.8
Loss on washing ...	6.7	10.0	5.2

The crude benzol is first washed with a dilute solution of sulphuric acid (sp. gr. 1.3), then with about 2% by volume of stronger acid (sp. gr. 1.8), and after washing with water it is agitated with caustic soda solution of sp. gr. about 1.1. For the fractions to be distilled after the crude benzol it is not necessary to wash with dilute acid, but greater care must be taken to wash more efficiently with strong acid and enough be used to free the material from nearly all the impurities.

The loss on washing is a very serious item in the cost of manufacture, and may rise in some cases to 10%. A crude benzol obtained by stripping gas shows a very much higher loss on washing than one from coal tar.

The acid tar obtained from the concentrated acid wash has been a great source of trouble to refiners. The free acid may be effectively extracted by washing with hot water and used in the manufacture of ammonium sulphate. The pitchy residue so obtained must then be disposed of in some manner that will satisfy the requirements of a factory inspector.

The practice followed in distilling for pure benzene and toluene will necessarily vary with different distillers and with different types of plant. Generally speaking, however, the crude benzol is run and preliminary fractions separated. Although with the present forms of column it is possible to separate pure products direct from the crude material, the time occupied in eliminating intermediates is so large and the washing difficulties are so important, that greater economy is obtained by running stills in pairs, one working crude and the other working intermediates for pure products.

The preliminary fractions from the crude are:—(a) Standard benzol, (b) light toluol, (c) crude toluene, (d) crude xylene. The distillation is represented in fig. 1, which shows the main features.

Fractionation of Crude Benzol. Preliminary Separation.

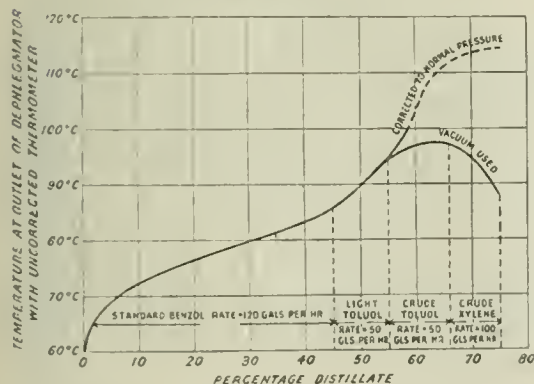


FIG. 1.

The variation in the rate of working will be noted; this rate is increased so long as the distillate is uniform in quality, and is decreased during the passage from one fraction to another. This will

be more apparent in the subsequent figures. The dotted part of the curve represents the distillation converted to normal pressure, the actual distillation under reduced pressure being shown by the solid line.

Fraction (a) consists of benzene and toluene and contains 90–95% benzene. It is fractionated for pure benzene and a yield of 65% is obtained on the first run. Results are indicated in fig. 2, the

Fractionation of Standard Benzol.

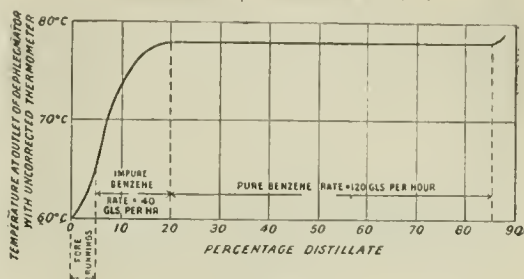


FIG. 2.

noticeable feature being the slow rate of distillation until the temperature at the outlet of the dephlegmator reaches a constant value; at this stage the rate can be increased and maintained until the temperature rises, when it is advisable to stop the distillation and recharge on the residue. The temperature of the dephlegmator water during this period is about 42° C., which ensures a good return to the column.

Fraction (b) consists of about 50% of benzene and 50% of toluene, no xylene being present. The working of this is shown in fig. 3. The

Fractionation of Light Toluol.

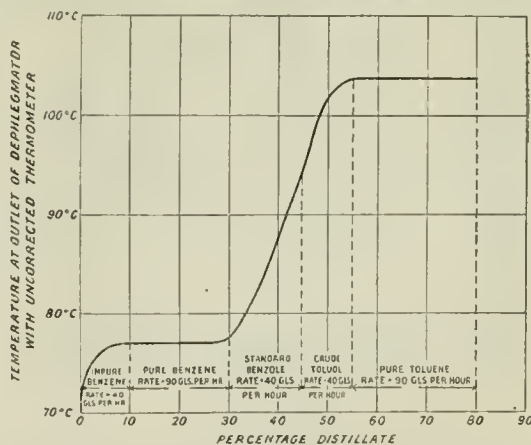


FIG. 3.

treatment of this fraction indicates perhaps more than any other the efficiency of the modern column. Both pure benzene and pure toluene are obtained in one distillation from the mixture. As the temperature rises after the separation of the pure benzene the rate is decreased, and is increased again when the temperature becomes constant on the pure toluene fraction. The dephlegmator water temperature is maintained at about 42° C. on the benzene fraction and allowed to rise slowly during the distillation of the intermediate until it reaches about 100° C. during the working of the toluene.



Fraction (c) consists of benzene, toluene, and xylene, the toluene content being 75%. The fractionation of this is indicated in fig. 4.

Fractionation of Crude Toluene.

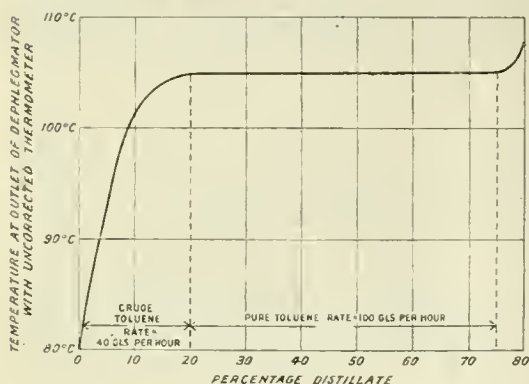


FIG. 4.

Fraction (d) consists of toluene, xylenes, and higher homologues, and is the most difficult fraction to deal with. It will be noted from the curve (see fig. 5) that a comparatively high percentage of heavy toluol is produced and is returned for reworking. When working for solvent naphtha (a mixture of xylenes and higher homologues) the use of vacuum or even of open steam at the end is advantageous, but should be avoided if possible when working for xylenes.

Economy in working can be obtained by observing a definite rotation. It has already been pointed out that the distillation of standard benzol is stopped when the temperature rises after the separation of the pure benzene. After successively working two or three charges in this manner the

Fractionation of Crude Xylene.

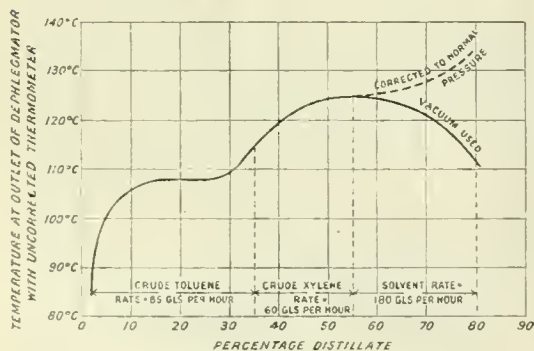


FIG. 5.

combined residues have accumulated toluene, and the still may then be charged with light toluol, to be followed by crude toluene. This rotation avoids unnecessary working of intermediate, thus saving both time and steam.

The rates indicated by the curves will necessarily vary with the plant and must be determined for each plant, but they indicate what has been done and show a marked improvement on the practice which obtained previous to the war.

Another point of note is the difference between the separation of benzene from benzene-toluene mixtures, and of toluene from toluene-xylene mixtures. From experiments made by the authors, working on pure compounds in the laboratory, it would

appear that no such difficulty should exist, that is to say, there are no properties inherent in the three hydrocarbons, such as the formation of constant boiling mixtures, which would cause this difficulty. The cause must therefore lie in the plant, probably in the dephlegmation, and might possibly be overcome by the use of salt solution or oil as a dephlegmating medium.

In conclusion, it may be of interest to record differences in the working of paraffinoid crude benzols and those free from paraffin. When dealing with such benzols, in addition to greater care being necessary in the washing process, involving greater loss, a further difficulty is observed in the fractionation. When working for standard benzene it is necessary to "sweat off" the fore-runnings slowly in order to prevent a high paraffin content in the benzene, and to reduce correspondingly the rate on the intermediates when working toluene from crude toluene.

It is very noticeable that in the case of products free from paraffin the "cuts" for pure benzene and toluene are clear and definite, whilst in the case of paraffinoid products a larger percentage of the distillate has to be rejected both at the beginning and end of the pure fraction, although the paraffin content is as low as 0.5%. For instance, fig. 2 shows that when the temperature reaches 78° C. pure benzene can be at once separated and the separation continued until the temperature rises. In the case of a paraffinoid product as much as 10% at the beginning and 5% at the end has to be rejected. The authors attribute this difficulty to the formation of constant-boiling mixtures of the aromatic hydrocarbons and the paraffins; some such mixtures are known, that of benzene and normal hexane being typical.

Another feature in the working of the paraffinoid benzols is the formation of a deposit on the coil in the still, which reduces the heating capacity to a detrimental degree. More thorough washing of the benzol does not prevent this. The nature of the deposit cannot easily be ascertained, because it is rapidly decomposed to a highly carbonaceous compound of indefinite composition.

Frequent cleaning of the still is necessary to maintain the conditions required for successful working.

## THE PROSPECTS OF FOUNDING A POTASH INDUSTRY IN THIS COUNTRY.

BY KENNETH M. CHANCE, M.A.

I have already indicated\* the lines upon which potash can be recovered in this country in large quantities and at low cost, and I will endeavour in this paper to give some account of the work which has actually been done to lay the foundations of an industry which, if properly developed, should render this country self-supporting not only in those potash compounds such as commercial chloride, carbonate, and caustic which are required in large quantities as raw materials for agriculture and other important industries, but also in those finer chemicals such as those quoted above refined to a condition of chemical purity and the permanganate, ferricyanide, and other salts of potassium of which the manufacture in the past has been practically, if not completely, confined to Germany.

### Blast furnaces—preliminary investigations.

In the autumn of 1914 the Halberg-Beth plant installed by Fraser and Chalmers, Ltd., at the works of the North Lincolnshire Iron Co., Ltd., for the

\* See two papers read at Middlesbrough on January 15th, 1918 "The Cleaning of Blast Furnace Gas for Boilers and Stoves," by A. Lennox Leigh, and "The Commercial Aspect of the Recovery of Potash in the Manufacture of Pig Iron," by Kenneth M. Chance.

purpose of cleaning their blast furnace gases, came into operation. A sample of the dust recovered from the gas was found to contain considerable quantities of potash and of cyanide, and an advertisement was inserted in the technical press offering quantities of this dust for sale. The attention of the British Cyanides Co., Ltd., was attracted to this advertisement and a preliminary examination of the dust led that company to suggest a working arrangement by which a joint investigation should be made by them with the North Lincolnshire Iron Co., Ltd., into the possibilities of producing the dust and working it up into commercial products. This suggestion was accepted and all was ready to commence before the close of 1914, but owing to delay due to alterations in the cleaning plant, it was not until April, 1915, that deliveries of the recovered dust could be sent to the factory of the British Cyanides Co. at Oldbury. This dust was found to contain much less soluble matter (including both potash and cyanide) than the original samples, and upon investigation into the cause it was discovered that the pump used to spray the gases with water in the pre-coolers had originally been out of order but had been put right in the course of these alterations, so that a great deal of the soluble potash was now being taken out of the gas in this preliminary cooling operation and carried away in very weak solution in the cooling water.

#### *Installation and operation of dry cooling plant.*

The next step was to introduce some method of cooling the gas without the direct application of water, and for this purpose a Green's economiser was ordered and installed.

This plant came into operation in the early days of 1916 and the next three months were spent in overcoming the usual mechanical troubles inseparable from starting up all new plants, in the course of which it was found that upon the temperature of the water entering the economiser depends to a large extent its smooth working, and it was better to let the cold water meet the hot gases and not cold gases. At the same time experience had been gained in sampling and measuring the gas and in operating the Halberg-Beth plant itself, while at Oldbury the problem of producing potassium carbonate of great purity from the recovered dust had, after several failures, been solved.

#### *Analysis of dust.*

The following is the analysis of a typical sample of dust derived from the Halberg-Beth plant at Seunthorpe in the early months of 1916, when the economiser plant was in operation:—Soluble in water: 5.58% KCN, 14.66%  $K_2CO_3$ , 23.23%  $KHCO_3$ , trace KSCN, 1.13%  $K_2SO_4$ , 3.66%  $KHCO_3$ , 10.90% KCl, 3.97%  $Na_2CO_3$ , 0.48%  $Fe(CN)_3$ , 0.36% Zn and Pb as sulphides; soluble in hydrochloric acid: 11.68%  $Fe_2O_3$ , 2.23% ZnS, 10.39%  $CaCO_3$ , 1.05% MgO, also 8.91%  $SiO_2$  and 2.18% carbon.

It may be of interest at this stage to give some figures of the alkali and chlorides contained in the dust recovered from the plant during the months which led to prematurely optimistic views being formed. The total alkali and chloride are expressed in terms of potash, the actual proportion of soda to potash being found constant at about 10 to 90; i.e., from the figures given below about 10% should be deducted to obtain the actual percentage of potash in the form of carbonate and chloride.

In May (before the discovery of the effect of the sprays in carrying away the soluble salts, especially the very soluble carbonate) the percentage of alkali as  $K_2CO_3$  and of chloride as KCl were each in the neighbourhood of 22% of the dust.

The following table gives the corresponding figures from July, 1915, to February, 1916:—

	1915					Dec., 1915- Jan., 1916.	1916
	July	Aug.	Sept.	Oct.	Nov.		Feb.
Alkali as $K_2CO_3$	43.31	42.07	43.66	41.85	38.02	40.02	37.85
Chlorides as KCl	15.96	15.28	14.14	14.95	15.94	14.17	11.86

By the end of March, 1916, it was thought that all difficulties had been overcome, and a report was sent to the Director of Optical Munitions giving details of this new source of production of pure potassium carbonate, which had then already been supplied for some months to the largest maker of optical glass in the country.

Hardly had that report reached its destination, however, when the quantity of soluble potash in the dust began seriously to decline. The average test for March showed 30.29% alkali as  $K_2CO_3$  and 12.07% chloride as KCl, for April 26.29% and 16.22%, and for May 24.27% and 17.42% respectively, while, at the same time, the proportion of soda to potash in that alkali had doubled, and the proportion of chloride to alkali, which had been nearly constant at 3:8 rose until it was 2:3.

An immediate investigation into the cause of the decline was set on foot, which took the form of daily tests of the gas from both of the furnaces then in blast and a comparison of these tests with the analysis of the slags drawn from those furnaces during the same period and with the proportion of coke to ore charged into the furnaces. It was clearly demonstrated by this investigation that the potash charged into the furnace and issuing from it both in the gas and in the slag was sufficient to produce upwards of 200 lb. as  $K_2CO_3$ , together with 25 to 30 lb. as KCl per million cubic feet of gas, so the next step taken was to make a careful analysis of all the available information and to hold a conference on the possibility of carrying out experiments in a blast furnace without detriment to the quantity or quality of the iron produced.

Numerous suggestions were considered, including that of introducing into the furnace some basic material which would flux at comparatively low temperatures, but it was decided to commence by determining the effect of increasing the proportion of lime to silica in the charge. Mr. Leigh pointed out that this would inevitably necessitate an increase of temperature in the furnace, and the results were carefully charted in order that the relative influence of increased base and increased temperature might be determined.

These preliminary experiments failed to provide any information that could be regarded as conclusive, but they indicated that the temperature of the furnace had some influence upon the quantity of potash volatilised into the gas in the form of carbonate.

Mr. Leigh thereupon undertook to carry out a series of short experiments whereby the temperature of the furnaces should be raised for periods of two or three days at a time, and daily samples of the gas, dust, and slag from those furnaces were analysed for their potash contents.

#### *Increased production of potassium carbonate.*

These experiments were commenced on August 5th, 1916, and their issue was hardly ever in doubt, as will be seen from fig. 1. Daily tests of the gas from both furnaces had been taken from the middle of March to that date, and the maximum weekly average of those tests had shown less than 60 lb.  $K_2CO_3$  in the gas and had fallen as low as 23 lb. per million cubic feet during the week ending April 22nd, the average for the week ending





tests from the same furnaces for the week ending August 6th and the period in question:—

*Lb. Alkali expressed as  $K_2CO_3$  per million cubic feet.*

Period.	No. 2 Furnace.			No. 4 Furnace.		
	Highest.	Lowest.	Average.	Highest.	Lowest.	Average.
Aug. 1-6	66.1	30.5	48.6	38.3	12.4	21.3
Oct. 1-18	122.0	84.0	81.4	123.0	63.0	94.6

After October 18th the furnaces from which these results were recorded resumed normal working and the alkali in the gas (expressed as  $K_2CO_3$ ) rapidly fell to the normal figure of about 40 lb. per million cubic feet. In November the experiment was repeated, and the alkali again raised to an average of more than 100 lb. for a week towards the end of that month, with a high record of 140 lb. on November 20th from one furnace. Similar measures were then taken in a furnace at the Scunthorpe works of John Lysaght, Ltd., and although no records so high as these were obtained, the alkali in the gas was increased to an even greater extent in proportion to the normal amount found in the gas at these works, which is greatly below that at the North Lincolnshire Iron Company's works.

These figures are open to criticism in that they are unaccompanied by accurate records of the temperature of the furnaces and of the actual weights of lime and silica charged into them. The conditions under which we were working rendered it impossible to obtain such records. The blast furnaces were not fitted and could not be fitted with pyrometers. In ordinary blast furnace practice it is impossible to analyse every charge for lime and silica and to weigh the contents of each with that minute accuracy which would be necessary for such records. Above all, these experiments were carried out in time of war and the labour required for such purposes was not available.

Although, therefore, there is a mass of figures available in the form of daily tests and records, which can be made public if its publication can serve any useful purpose, the information provided is not conclusive. Indeed it is probable that no really conclusive proof of the relative influence of base and temperature upon the volatilisation of potash in the form of carbonate from a blast furnace can be tabulated unless a blast furnace is specifically designed and constructed for a thorough research into this problem, although one further experiment which we carried out may possibly have some bearing upon it.

Soda ash was added to the furnace regularly in suitable quantities over a period of three or four days to the extent of five tons. The effect upon the production of potassium carbonate was at the best inconsiderable—indeed it is questionable whether it had any effect at all—and was in marked contrast to the immediate effect resulting from the addition of salt to the blast furnace of which some account is given below.

Perhaps the strongest evidence as to the influence of the combination of high temperature with high basicity in the slag is furnished by an analysis of the gas and slag produced in the manufacture of ferro-manganese, where both the proportion of lime to silica and the temperature at which the furnace is operated are considerably higher than in normal blast furnace practice. In this case the slag contains the merest trace of the potash charged into the furnace, practically the whole of which is volatilised into the gas in the form of carbonate, cyanide, and chloride.

It is therefore permissible to assume that if the same methods could be applied to the production

of pig iron the same results would be obtained and potassium carbonate would be readily available in large quantities, but unfortunately the cost of operating a blast furnace in this manner is so great as to be prohibitive for the manufacture of pig iron, and some other method of volatilising the potash in the ordinary blast furnace had, for this reason, to be found.

#### *A wider demand for potash.*

Up to the close of 1916 the requirements of potash that had made themselves urgently felt were all for potash in the form of carbonate, especially for making optical glass, and its production in that form had therefore been our sole objective under the auspices of the Controller of Optical Munitions, Mr. Esslemont. About that time, however, the vital need for potash in a form suitable for use on the land was made known to Mr. Esslemont, who therefore asked me to draw up a scheme for meeting those demands, thus placing the problem of quantitative and cheap production in the forefront. The first suggestion was obviously the collection and distribution of the dust drawn from the stoves and boilers at blast furnace works, because the process of combustion removes all impurities that are harmful to agriculture from this dust, which consists of carbon, lime, and potash. But at the best, the collection and distribution of this dust could only be regarded as a temporary expedient and partial stop-gap and could not form the basis of an industry which would render this country self-supporting in providing potash at reasonable prices, because the quantities available are insufficient and the proportion of potash in the dust too low. It was therefore necessary to find some other source of supply.

#### *Consideration of other methods of production.*

I have already narrated how at times the production of dust from the Halberg-Beth plant of the North Lincolnshire Iron Co., Ltd., had fallen off either in quantity or in quality to such an extent as made it appear extremely doubtful whether permanent supplies of potash at prices which could compete with German production after the war could ever be collected as a by-product of the manufacture of pig iron. In the meantime, the British Cyanides Co., Ltd., was endeavouring to produce some of those refined potassium compounds the manufacture of which either had never yet been achieved in this country, or had been killed by German competition, and it had become a matter of vital urgency to that company, before the national need was fully recognised, that its supplies of raw material should be secured. Problems therefore such as the recovery of potash from felspar, from alunit, or as a by-product of the manufacture of cement, had all been carefully considered in the early days of the war by the directors and staff of that company.

It may not be out of place to give the reasons which led us in those early days to continue to devote time and money to the blast furnace problem in preference to any of these others, although the British Cyanides Co. had already experienced to the full in the recovery of cyanide from coal gas the difficulties of persuading a large body of individual manufacturers of the benefits to be derived from the central collection and refining of a by-product of their ordinary manufacture, and we were not therefore in any way attracted by the prospect of having to repeat this propaganda work in the recovery of potash as a by-product of pig iron manufacture.

The recovery of potash in a gas-cleaning plant at a blast furnace has one enormous advantage over all other native sources of supply in that the



value of the main product of that plant, namely clean gas, is in itself sufficiently great to provide a handsome remuneration for the capital outlay involved in erecting it and the cost of its operation, so that the cost of recovering potash already in the gas in the form of dust is *nil*. The second consideration is that the main items of cost in volatilising potash from an ore in which it is present in insoluble form, namely cost of raw material, labour, and fuel, are, in the case of a blast furnace, already incurred and reckoned as items of cost in the manufacture of the main product, pig iron. The third consideration is that, to the best of my knowledge, there is no bed of feldspar or similar ore in this country which can compare in magnitude or convenience of situation with the soluble potash deposits in Stassfurt and Alsace, while the cost of producing soluble potash from such insoluble ores must inevitably compare unfavourably with that of refining the soluble deposits above referred to.

#### *The development of the salt process.*

In fig. 1 it will be seen that there is a straight line representing lb. KCl per million cubic feet of gas, which remained practically constant at about 30 lb. throughout the wide variation of  $K_2CO_3$  recorded in the course of these experiments.

Investigations at the works of John Lysaght, Ltd., extended the range of variation in the alkali contents of the gas down to zero, but the quantity of KCl remained practically unchanged.

There emerged, therefore, the notable fact that while the alkali in the gas varied from 0 to 140 lb. per million cubic feet, the volatile chloride remained practically constant. The natural inference was that the chlorides in the furnace charge were practically constant and were all volatilised into the gas, so that if additional chlorides were artificially fed into the furnace they would combine in the zone of reaction with those bases having the most volatile chlorides. It followed that as the most readily volatilised of all the chlorides is that of potassium a large proportion of the potash charged into the furnace would be volatilised into the gas in that form.

This theory had already met with some confirmation from an examination of the proportions of potash to soda in the total chlorides already found in the gas, which varied from 85% to 90% of potash and 10% to 15% of soda, whereas the proportions of potash and soda in the furnace charge were approximately equal.

The North Lincolnshire Iron Co., Ltd., thereupon consented to try the experiment of adding salt to one of their furnaces in sufficient quantity to volatilise the whole of the potash in the furnace. This was done in February, 1917, and the success was instantaneous, the chlorides in the gas rising from about 30 lb. per million cubic feet to upwards of 200 lb. expressed as KCl, while the proportion of potash to soda in the total chlorides was not decreased but remained at about 9 to 1 and the alkali in the gas was unaffected. The actual potash in the gas as alkalis and chlorides combined was therefore raised from about 60 lb. to upwards of 200 lb. per million cubic feet of gas.

The use of salt has been repeated in several blast furnaces in other parts of the country since that time and, while the quantity of potash volatilised naturally varies with the quantity charged into the furnace, in no case has the addition of salt failed to more than double the quantity of potash found in the gas, while in others it has trebled or quadrupled it. (See Eng. Pat. 112,333 of 1917: this J., 1918, 88 A.)

The extended use of salt in blast furnaces has up to the present disclosed, so far as I am aware, no adverse results as regards the manufacture of pig iron.

#### *The cost of producing potash by the salt process.*

It is obviously impossible to give more information on this subject than can be deduced by the application of technical knowledge to the main facts regarding the process which have now been made public, but some general deductions may be drawn. The cost of cleaning the gas must be debited to the cost of the pig iron, and the cost of the raw material at the blast furnace works is therefore limited to the cost of salt, of which about three tons is required for the production of four tons of potash in the dust. For purposes of comparison with pre-war figures, £1 per ton of salt delivered to the blast furnace works, or 15s. per ton of potash, should be ample allowance. The next item of cost is that of carriage in bulk to a central factory, and for this 5s. per ton of dust, or say 12s. per ton of potash, should suffice. The cost of potash in the raw material delivered to the central factory may therefore be estimated at 27s. (say 30s.) per ton.

From this stage the cost of production of potassium chloride differs only from that of producing sodium chloride in the following particulars:—1. Lixiviation of the dust as compared with pumping the brine. 2. Elimination of impurities. 3. Scale of operations. Of these items of cost the last is by far the most important, and it is only possible to draw any comparison between the cost of production of these two chemicals if the scale of operation is comparable. An output of 500 tons per week, which is the figure that I have given as the minimum size for each central factory, would be considered small for a salt works, but it is a scale of production sufficiently large to justify a comparison of this nature, and at the same time the distribution of the blast furnace industry in this country is such as to justify the anticipation that where the use of gas cleaning plants is general, central factories on this scale can be erected at suitable points so that the cost of carriage of the dust to them will not on the average exceed the figure of 5s. per ton indicated above.

We have therefore the following information available as to the cost of production of potassium chloride by this process:—(a) Cost of raw material delivered to central factory, say 30s. per ton KCl. (b) Cost of refining; comparable to (say for example double or treble) the cost of production of common salt. (c) Cost of bagging and distribution. (d) Profit to the blast furnace owner and to the refiner.

Two points arise from this:—The first that the process of manufacture is thus strictly comparable to that of any heavy chemical, and therefore its cost will inevitably depend very largely upon the scale upon which that manufacture is conducted. The second that by the use of suitable quantities of salt in blast furnaces, coupled with the installation of efficient gas cleaning plants, a raw material is recovered as a by-product of the pig iron industry from which the manufacture of potassium chloride can be carried out on a sufficient scale to meet the requirements of this country and at a price which, on a pre-war basis of cost, is less than the lowest figure at which German potassium chloride has ever yet been marketed in this country, namely £8 per ton, after providing a margin of profit to the blast furnace owner and to the refiner.

At this point I should like to pay tribute to the large part taken by the directors and staff of the North Lincolnshire Iron Company, Ltd., and especially by Mr. Leigh, in this work. I am only now beginning to realise the difficulties in which Mr. Leigh must frequently have found himself in meeting suggestions which were based upon experience of large scale experimental work on furnaces designed for a totally different purpose and from lack of experience of blast furnaces. The credit of



meeting and overcoming these difficulties is due to Mr. Lelgh, ably seconded by Mr. Langdale and Mr. Parker. Valuable assistance was rendered by Mr. Williams, of John Lysaght, Ltd., in confirming our results. The actual research work on the spot was entrusted to Mr. Dingley, under the supervision of Mr. Rossiter, Chief Chemist to the British Cyanides Co., Ltd., who was responsible for the control of this work both at Oldbury and at Scunthorpe.

#### *Two other sources of by-product potash.*

Apart from the blast furnace industry there are two other industries in this country from which potash may be recovered as a by-product, namely, the manufacture of cement and the washing of raw wool.

**Cement.** By use of salt in the cement kiln, whether in the crystalline form or as sea water, there is ample evidence both in this country and from the United States to show that potash in considerable quantities is volatilised into the waste gases from the kiln, and in the latter country particulars have been published of plants that have been erected for the recovery of the dust from these gases, the dilution of the potash therefrom and its crystallisation from concentrated solutions.

In both countries the greatest difficulty has proved to be the construction of cheap and efficient plant for extracting the potash from the gas. Various methods of washing the gas have been tried, and the removal of the dust by electrical precipitation and in a bag plant of the Halberg-Beth type has also been attempted.

According to one authority a combination of electrical precipitation and the collection of the dust in the form of a slurry has been found successful in producing potash at a profit in the abnormal conditions that exist at the present time, but in looking to the future it must be remembered that the gas from a cement kiln has no value beyond its potash contents, while the value of blast furnace gas is enormously increased by the removal of the dust from it. The former industry thus labours under the disadvantage of having to debit the whole cost of the cleaning plant to the potash recovered by it, while that cost is borne by the increased value of the gas from a blast furnace, and, therefore, unless some counter-balancing advantage can be discovered and utilised, the cost of recovering potash must always be greater from a cement kiln than it is from a blast furnace. It is probable that this compensation will be found in the condition of the dust itself, which contains the potash in a form in which, owing to its being recovered from gas which has been burnt, and to the harmful impurities, such as carbonates and cyanides, having been thereby destroyed, it is suitable for direct application to the land. The best line of development for the recovery of potash as a by-product in the manufacture of cement would therefore appear to lie in the direction of the recovery of the dust in the dry state for direct sale as a fertiliser containing about 25 to 30% of potash as  $K_2O$ , in the form of the chloride and sulphate, with lime as the preponderating impurity.

**Suint.** The third source of by-product potash in this country is the wool-combing industry which is centred in Bradford.

The method of washing the raw wool generally adopted is to scour it in hot water with alkali and soap in three or four different vessels, thereby removing the potash in very weak solution contaminated with large quantities of fatty matter and other impurities in addition to the soap and alkali. In many cases the fatty matter is now recovered from these solutions, but the liquors are run to waste as having no value.

By a preliminary washing of the wool in cold water, the potash can be recovered from it as carbonate in a solution which is contaminated with a small proportion of the natural fats of the wool. This practice appears to have been more widely adopted in the past than it is now, because at a time when both potash and soda were more costly than they became during the latter part of the last century, it was a profitable undertaking to recover the potash in this form, concentrate it, and thus provide the alkali for scouring the wool in the second stage.

Owing, however, to purely physical difficulties of concentration, due to the fatty contamination of the weak potash solutions, the recovered potash has always taken the form of an impure mixture of carbonate and sulphate, and as such has been of little commercial value, while the fats present in the weak solution have been destroyed in the process of concentration.

If these difficulties can be overcome there is no doubt that potassium carbonate of great purity can be recovered from the preliminary washing of wool with cold water, and the fats rendered of value in the form of gas and oils.

#### *The work that has been accomplished.*

The salt process is at work at more than one blast furnace works in this country, cleaning plants are extracting the potash from the gas at those works in the form of dust, the first factory for treating the dust has been erected and is now producing potassium chloride at the rate of some fifty tons per week. There are in addition cleaning plants in course of erection at other blast furnace works, and the factory referred to, which is situated at Oldbury, is designed for a total output of from 400 to 500 tons of potassium chloride per week, and as supplies of raw material become available, will be brought up to that scale of production.

Arrangements have been entered into with one of the leading chemical companies engaged in electrolytic work for the manufacture of caustic potash in bulk from the chloride by electrolysis, and in a short time caustic potash from this native source will be available both in the commercial form and in the pure state, and at the same time chloride, refined for druggists' use, will also be available.

The problems connected with the manufacture of the following salts of potassium in their pure state have been overcome, and they are all now being produced on a scale sufficient to meet the essential needs of the country in time of war:—

Potassium carbonate testing upwards of 99%  $K_2CO_3$ .

Potassium bicarbonate testing upwards of 99·5%  $KHCO_3$ .

Potassium ferrieyanide testing upwards of 99·8%  $K_3Fe(CN)_6$ .

Potassium permanganate testing upwards of 99·8%  $KMnO_4$ .

Admittedly at present prices are high and the ordinary consumer who cannot make out a case for war necessity has to go short, but now that the initial difficulties of manufacture have been overcome, and alternative methods have been worked out to permit of advantage being taken of potash in the form either of the chloride or the carbonate, whichever be the more readily available, or the cheaper, and now that arrangements have been made for the mother liquors from one process to be used as the raw material for another, a gradual improvement, both in the quantities of these chemicals that are available and in the price at which they can be purchased in the open market, may be confidently anticipated.

Some indication as to the magnitude of the difficulties which had to be overcome may perhaps be worth mentioning. In the first place the problem



of producing potassium carbonate suitable for use in the manufacture of optical glass and also for druggists' use from the impure flue dust was especially difficult on account of the crude nature of the raw material and the exceptionally high standard of purity demanded in the finished article, and three experimental works-scale plants were erected and discarded within less than six months before a method was evolved for the manufacture of a product which has fully satisfied the exacting requirements of these two industries.

In the manufacture of ferricyanide we soon found that nothing short of chemical purity would be of any real value, and that high standard was only attained by laborious experiment on the elimination of impurities. In the case of permanganate, a plant was laid down for its manufacture by the text-book method of fusing caustic potash with manganese dioxide and oxidising the soluble manganate with carbon dioxide on a scale of production which in the early stages was regarded as ample for all possible requirements. Those possible requirements were, however, doubled, then quadrupled, and have recently been multiplied by eight, so that a less cumbersome method had to be devised and installed at short notice without jeopardising the scanty supply of potash that was available in the form of carbonate, or the rate of production of permanganate that had already been reached. At the present time the only form of potash used in the manufacture of both potassium permanganate and ferricyanide is the chloride.

In working out these technical problems, erecting the factory of the Potash Company to treat the flue dust, and remodelling one of the factories of the Cyanides Company for the production of pure potassium salts, I was able to employ the organisation built up by the latter company prior to the war, and I wish to mention the invaluable assistance afforded me in this work by Messrs. A. E. Doherty, Manager, L. A. Fullagar, Chief Engineer, and E. C. Rossiter, Chief Chemist of that company. Developments of this nature in time of war are attended with difficulties infinitely greater than in normal times, but such difficulties only serve as spurs for further effort on the part of these men and the staff and workmen under them.

#### *The work now in progress.*

Although the arrangements which have already been made are sufficient to ensure the production of potash in this country to an extent which before the autumn of this year will meet the requirements of the country for all essential purposes in time of war, other than explosives and agriculture, the needs of agriculture are so great and, in these times, so paramount, that every ton of potash which is used during the next few years in the chemical industry will, in a sense, be decreasing the potential food supplies of this country.

At the same time it is hardly an exaggeration to say that the present output of 50 tons per week of commercial potassium chloride represents but 1% of the potential supplies now going to waste in this country, and the plants now in course of construction will only serve to render available about one twentieth of that waste.

The engineering problem which transcends all others in importance is that of designing a really cheap and thoroughly efficient plant for removing the fine potash fume in the dry state from large volumes of gas both from blast furnace and cement kiln. Experimental work on the lines which have proved successful in solving the problems that have already been described is now being actively pressed forward, and as soon as these preliminary experiments have been taken sufficiently far to indicate the correct design for a unit capable of handling one million cubic feet of gas per hour, trial plants

on that scale will be constructed and erected at blast furnace and cement works where facilities for such trials have been generously offered.

The qualifications essential to success in such a plant are that it shall be simple to construct, erect, and operate, that its construction shall not make undue inroads upon materials that are difficult to obtain, that its erection shall not throw heavy demands upon skilled labour, its capital cost (apart from mains and connections) shall not exceed £5000 for each unit of that capacity at the inflated prices current to-day, and its operating cost shall not, under existing conditions, amount to  $\frac{1}{2}$ d. per 1000 cubic feet of gas.

The plant must be practically fool-proof when handling gas under the wide variations of temperature and vapour density which are inseparable from ordinary blast furnace practice and it must be capable of being constructed and erected within the limits of time imposed by the acquisition and erection of the mains and connections required to bring it into operation.

Those are briefly the objectives which have been laid down, and until they are reached the foundations of a potash industry in this country will not have been so securely laid as to exclude competition from abroad.

The second problem which is now being tackled is the treatment of the suint liquor which is being conveyed in quantity from Bradford to Oldbury for experimental work on a practical scale in the plant which has already proved its suitability for experimental work of this nature, and considerable progress has been made towards its solution.

#### *Conditions essential to success.*

When these problems have been solved, as they assuredly will be, the formation of a strong and healthy potash industry will be cleared of the chemical and engineering difficulties which have hitherto compelled this country to pay tribute to Germany for every ton of potash that it consumes.

The combination under one control of all the German potash interests has created one of the most absolute and powerful monopolies that the world has ever known, and we can be very sure that nothing will be left undone to prevent that monopoly being broken.

To bring about a similar combination of interests in this country must inevitably be far more difficult than in Germany because of the widely diverse industries from which these potential supplies of by-product potash must be drawn and because each of these great industries as a whole is composed of a large number of individual firms. In fact, the comparative unimportance of by-product potash to any one of these firms as a source of revenue in the future, when prices are brought down to a normal level, renders it absolutely essential that the plant installed for its recovery shall be cheap of construction and simple in its operation and that the producer shall be able to dispose of the crude product easily and automatically to central refining factories and shall not be burdened with the complications of an intricate process of chemical manufacture.

It is these considerations, no less than those to which I have drawn attention earlier in this paper, which have forced me to the conclusion that the production of potash in this country on a scale and at a cost which will enable German competition to be faced, can only be effected by the unification of all the varied interests involved; and the more deeply that the problem is studied the more clearly does this stand out as the only possible road to success.

#### *DISCUSSION.*

Capt. C. J. GOODWIN said that in considering the question of a national potash industry it was important to consider what would be the position

after the war; the only basis of comparison available at present was the pre-war basis, and he hoped it would be possible to show that the industry could be established without the necessity of import restrictions or similar measures. Considerable doubt existed as to the actual cost of potash manufacture in Germany before the war; it had been given on various occasions as between £4 and £6 per ton. Mr. Chance had stated that the cost of gas cleaning should not exceed  $\frac{1}{2}d.$  per thousand cub. ft., or 7s. per million cub. ft., and the corresponding quantity of potash recovered, viz. 200 lb., was worth about 15s. If the cost of cleaning the gas materially exceeded that figure the margin of profit would be greatly reduced. A great deal of work had been done in the last few years on the treatment of felspar; the problem was particularly applicable to those parts of the Empire where blast furnaces were perhaps not available and in countries where there was unlimited scope for agricultural development. In that connection the volcanic deposits in Italy, which contained leucite, were of great interest. He also referred to the Ashcroft process, in which such rocks were treated at high temperatures with common salt, with a recovery of about 85% of the potash. He understood that the cost of producing potash by this process was less than the pre-war price of German potash. This and similar processes appeared to be similar in principle to the blast-furnace process described by Mr. Chance, each depending on the reaction between sodium chloride and a potassium compound in the absence of oxygen.

Mr. C. J. WATERFALL said that the possibility of utilising felspar appealed strongly to those in the West of England, where there were extensive deposits of that mineral.

Dr. E. F. ARMSTRONG emphasised the point which was made throughout the paper, that the whole question was an economic one—that the potash would have to be recovered at a cost which would permit us to compete with natural salts. Mr. Chance appeared to be somewhat apprehensive as to the possibility of getting enough blast furnaces and similar works to purify their gas to produce sufficient raw material for a really large plant for extracting potash. He suggested that it was in matters of this kind that societies like their own and the Association of British Chemical Manufacturers, and ultimately, if necessary, the State, should help. It should be made compulsory that these cleaning plants were attached to blast furnaces, but at the same time it must be made sufficiently profitable for the blast furnace companies to do this. It was frequently said that the manufacturer had no right to make any profits beyond a bare 5%; if that doctrine made headway it would be impossible to establish the potash or any other industry in this country. These very excess profits which were being taken away from blast furnace owners might be usefully employed in wiping off the capital cost of these cleaning plants.

Dr. F. M. PERKIN pointed out that some blast-furnace dusts contained large quantities of iron dust and iron oxide. How would such dusts be treated in the process described by Mr. Chance? He agreed with Dr. Armstrong's remarks about the employment of capital; the great German dye industry was largely built up by wiping out excess capital, and the capitalisation which appeared on the books of the great German dye and chemical firms was very small compared with the actual amount of money that had been spent on the plant.

Mr. C. S. GARLAND said that it must not be expected that blast furnace dust would be obtained for nothing. As in the case of other waste products formerly considered as useless, when it was realised that it was of value the price would certainly rise.

Dr. S. MIALl suggested that a committee might be appointed by the Society, in conjunction with the Association of British Chemical Manufacturers, to confer with Government departments with a view to obtaining the organisation indicated by Mr. Chance.

Mr. PATTERSON pointed out that whilst it was quite true that it would pay the blast-furnace owner to have clean gas, he would not, in order to get clean gas in the ordinary way, go to the same expense as if he were desirous of extracting potash. For instance economisers had to be put in instead of the usual wet-cooling plant, and these were much more expensive than the cooling apparatus in the original Halberg-Beth process. Therefore the blast furnace owner would have to be paid that extra expense. At the present time probably not more than 10 or 15% of the dust produced in blast furnaces was treated. Much of it was deposited in the tubes and the downcomers, and it was only the fine suspended matter that was treated.

Mr. F. SOUTHERDEN asked whether the author could give figures of the cost of recovery of crude dust from the furnace gases and also whether experiments had been tried in the use of this crude dust for fertilising purposes.

The PRESIDENT said that he was possibly one of the earliest to have found potash in blast furnace dust. In 1878, when chemist to some blast furnaces in Nova Scotia, he had analysed the dust from some Siemens-Cowper stoves and found a considerable amount of alkali, especially potash. The fact had been of no importance at that time, but the present significance of the matter had been made evident by Mr. Chance. He thoroughly endorsed the view that the cleaning of dust in some way or other paid for itself. Many years ago he had been concerned in installing in Sweden one of the first blast furnace gas engines; within six months of putting in that engine, the piston rod was worn  $\frac{1}{4}$ -inch all round and was very soon useless, owing to the scouring action of the dust in the gas. As long as the old method, still largely used, of burning the gas under boilers was used, very crude cleaning was sufficient, but for use in a gas engine, the most efficient cleaning possible was required, and blast furnace owners had spent very large sums upon efficient cleaners. He agreed with Mr. Patterson that hitherto wet cleaning had been found to be the cheaper, but there was no evidence that that would remain the case. The Cottrell electric process was exceedingly promising. As regards the use of salt in the blast furnace, he wondered whether it would not eat the top bricks a good deal, and it occurred to him that a cheap waste product like calcium chloride might be found equally efficient.

Mr. CHANCE, in reply, said that a large amount of work had been done in America on the Cottrell process, but the capital cost was very high and it certainly did not come within the conditions he had laid down in the paper. The difficulty appeared to be with the extraordinarily fine fume. At first he had thought it would be possible to settle the fume out, but in a test it still floated at the end of a week. If the gas were kept hot it was possible to deposit the fume, but if it were allowed to cool so that the specific gravities of the gas and the fume were approximately the same, it was impossible to remove the fume by settling, and the difficulty with the Cottrell process was that if this very fine fume was to be deposited on the tubes or plates, it was necessary so to lower the velocity of the gas that the size of the plant became enormous and the cost became prohibitive. Also when this fume was once deposited on the plates or tubes the great difficulty at the low



velocities was to prevent it being carried on. That had been overcome in America, he understood, by a double system of plant whereby the gas was passed first over plates or through tubes at a fairly high velocity, and on the way practically all of the heavy dust was recovered and a certain amount of the fume. In the second plant a stream of water trickled over the plates or tubes. The objection to that, however, was that the vapour density of the gas was raised very considerably, and the heating properties were largely reduced; it also resulted in the dew point being lowered, and vapour was deposited. With regard to adding salt to blast furnaces, there had been a good deal of evidence one way and another, but the actual putting in of the salt made no difference except that it increased the quantity of potash. In a paper read at Middlesbrough about 15 or 20 years ago it had been shown that the bricks from an ordinary blast-furnace became so saturated with potash that on the inside they contained from 40 to 50%, and even on the outside wall of the furnace from 5 to 10%. Their experience only went back to January, 1917; one of the furnaces had not shown any sign of deterioration due to the use of salt, nor did he find any objection was raised by blast furnace owners. In reply to Dr. Perkin, iron dust fell out in the downcomers, but it did not reach the cleaning plant, or only in very rare cases. He was not at liberty to give any further details as to costs. In regard to the cost of cleaning referred to by Capt. Goodwin, the figure of  $\frac{1}{2}$ d. per unit only referred to cleaning of cement kiln gases. With the Cottrell process 0.05d. or 0.06d. was said to be the cost, and he believed that claim was quite justified, except that he did not know at what rate they reckoned depreciation of plant. One of their present problems was to design a cleaning plant which would get over the difficulties that had been pointed out. The present economiser was too costly. The Green's economiser was made for a steam pressure of anything up to 200 lb., and therefore they were using a plant which was far too costly for the purpose. The difficulty at present was that there was no plant on the market for cleaning gases thoroughly, with the possible exception of the wet cleaning plant, and he did not think that blast furnace owners favoured that type of plant. There were only one or two in use in this country, but it was more commonly used in the United States. For dry cleaning there was no plant on the market except the electric process and the Halberg-Beth plant; both of these used a very large amount of steel and, incidentally, were very costly. With regard to Government assistance, he mentioned that half the shares in the British Potash Company were owned by the Government and they had Government assistance to a very large degree. The suggestion that a committee of the Society should be formed was a most valuable one, and the time might come when it would be of the utmost assistance to the potash industry, but at the present time the trouble was not lack of Government assistance, except perhaps in regard to labour. The chief trouble was that the proper plant was not available.

#### SOURCES OF LOSS DURING THE MANUFACTURE OF CONCENTRATED AMMONIA LIQUOR.

BY ARTHUR MARSDEN.

Several very instructive papers\* have recently appeared in the technical press on the subject of concentrated ammonia liquor, and the object of this paper is to describe the sources of ammonia

loss present when making that product, together with some of the remedies applied, and it is hoped that the suggestions may be productive of further improvements in the efficiency of the process.

The general principles of manufacture are well known and need not be described here. In pre-war plants the method largely used was that of "fixing" all the carbon dioxide and hydrogen sulphide in the liquor by means of lime. This has now been replaced by the process of "decomposing" the liquor, prior to distillation, at a temperature below boiling point, in order to liberate as much of the carbon dioxide and hydrogen sulphide as possible together with the least amount of ammonia. The conditions necessary to effect this end are, in practice, often difficult of attainment and vary in different types of plant.

After leaving the heater or economiser the liquor usually flows direct to a "decomposer" or "desulphuriser" and during its passage down that apparatus the evolution of carbon dioxide and hydrogen sulphide takes place progressively due to rising temperature, until, towards the bottom, the temperature is high enough to liberate ammonia, much of which is absorbed again in passing upwards through the cooler trays. The ammonia passing away with the outlet gases is dealt with in different ways, according to circumstances.

The remarks which follow deal in particular with a plant of the Wilton type, consisting of a combined still, 5 ft. 4 in. internal diameter, and liming vessel, capable of dealing with 26,000 gallons of 7 oz. liquor per 24 hours, together with a 13-tray desulphurising still 5 ft. 4 in. internal diameter; a catch tower to arrest, by means of water, the ammonia driven off from the desulphuriser; an exchange liquor heater; reflux and final condensers, and the usual accessories. The still has 15 trays, including one "liming" and one anti-priming section. Each tray has one rectangular serrated hood running across the centre, and is provided with suitable hand holes for removing the hood. The liming vessel is a circular cast iron vessel 3 ft. 6 in. internal diameter and 7 ft. high. It is fitted with a live-steam distributor at the bottom.

The desulphuriser gases may be dealt with in various ways. In some cases they are returned to the foul-main, but difficulty has been experienced owing to the formation of ammonium carbonate in the pipe conveying the gases. Deposits of naphthalene have also given trouble, especially if light tarry matter entered the desulphuriser with the liquor. In any case complete freedom of the liquor from tarry matter is most desirable. In other cases where a sulphate of ammonia plant is arranged in conjunction with the liquor concentrating plant, the desulphuriser gases are passed directly to the saturator. That method also appears to have objections because, unless the sulphate plant is in operation, apart from the liquor plant, the "bath" needs much attention if the formation of "blue-salt" is to be avoided, also, any naphthalene driven forward is liable to cause a dirty, dark-coloured salt, which can only be remedied with difficulty. The treatment of the gases by means of an acid bath offers, on the other hand, an advantage in the case of liquors containing much pyridine which, being basic in character, is arrested by the acid, whereas when a catch tower fed with water is used, as in the case of the plant here dealt with, much pyridine passing to the purifiers may become very objectionable and render the washing of the gases by oil necessary.

For the purposes of this paper no account has been taken of any losses occurring during the production, storage, or delivery of the crude liquor to the plant, although those phases of what may be termed the "ammonia question" provide much

\* Gas J., 140, 412, 554, 603; 141, 398.



material for investigation, and are receiving attention.\*

The plant here dealt with was put to work in October, 1917, and worked satisfactorily as far as the quality of the concentrated liquor produced was concerned, which averaged 28.12%  $\text{NH}_3$  and 0.45%  $\text{H}_2\text{S}$  by weight, but it soon became evident that considerable losses of ammonia were taking place, the usual works' stocks indicating an efficiency of conversion, from crude to concentrated liquor, in the neighbourhood of 80%. As a result the plant was run under test for 24 hours, maintaining as far as possible the usual conditions of working, but taking careful series of readings and measurements. The figures obtained showed that, of the total ammonia supplied to the plant as crude liquor, 90.1% was obtained as concentrated liquor. This improved result when compared with ordinary working was doubtless due to the increased and constant supervision during the test. The results obtained were as follows:—Average rate of liquor flow to plant, 63.1% of rated capacity. Average steam on still (first tray), 18 lb. per sq. in. Temperature of gases at outlet of desulphuriser 68.1° C., and at outlet of last reflux 83.2° C. Water required to keep catch tower outlet clean, 73 gallons per hr., or equal to 10.6% by vol. of liquor used. Lime used, 2½ cwt. per ton of 25% liquor. Crude liquor supplied, fixed  $\text{NH}_3$  0.16%, free  $\text{NH}_3$  1.55%; total, 1.71% = 7.88 oz. liquor.  $\text{H}_2\text{S}$  0.20%. Desulphurised liquor, 1.39%  $\text{NH}_3$ , 0.088%  $\text{H}_2\text{S}$ . Effluent, 0.010%  $\text{NH}_3$ . Concentrated liquor made, 25.21%  $\text{NH}_3$  and 0.42%  $\text{H}_2\text{S}$  by weight.

It will be seen from the above that the amount of water required to keep the catch-tower outlet clean was rather large, and not conducive to steam economy in the desulphuriser.

The various sources of ammonia loss were investigated and remedied as far as possible, but, unfortunately, it is not possible to give any figures relating to an improved efficiency of the plant because, before sufficient trials could be made, the plant, or rather portions of it, were changed over to the manufacture of sulphate of ammonia by order of the Ministry of Munitions, thus obviating, as will be seen, the chief sources of loss present when making concentrated liquor. The losses of ammonia in connection with a plant of the type in question may be due to the following causes:—(a) Desulphurisation. (b) Imperfect liming and boiling in the stills. (c) Pumping desulphurised, reflux pot and concentrated liquor. (d) Storage tanks. (e) Loading of rail tanks. (f) Leaks in the apparatus. These respective sources of loss will now be dealt with in order.

(a) There certainly remains much work to be done on the "decomposition" of ammoniacal liquor both from a general and an individual point of view and, as mentioned by Parrish,† the physico-chemical problems involved form "a subject which is peculiarly captivating to the student of applied chemistry and physics." In the present case lack of time has prevented any detailed enquiry so far. The steam pressure on the desulphuriser bottom tray averaged, over the period of working, 19 lb. per sq. inch; the average  $\text{H}_2\text{S}$  content of the liquor supplied being 0.21% and the total ammonia content 1.70%, while the desulphurised liquor averaged 0.061%  $\text{H}_2\text{S}$  and 1.48% total  $\text{NH}_3$ . The finished liquor, as previously stated, contained 28.12%  $\text{NH}_3$  and 0.45%  $\text{H}_2\text{S}$ . From these figures the hydrogen sulphide distribution works out as follows:—

	% $\text{H}_2\text{S}$ .	% Removal.
Removed by desulphuriser	2.81	70.8
.. .. lime ..	0.71	17.8
Remaining in conc. liquor	0.45	11.3
	3.97	99.9

\* Alkali, etc., Works Report, 1911.

† Gas J., 140, 557.

No systematic tests were made on removal of carbon dioxide. The average temperature at the top of the desulphuriser was 69.5° C., a supply of cold liquor being admitted to the top tray for purposes of control. The liquor from the catch tower was pumped into the third tray from the top and the hot main liquor supply into the fifth.

The catch tower has five trays 36 in. diameter and 8 in. deep. The total height including the bottom box is 5 feet. In this portion of the plant the most serious ammonia losses occurred. The condition aimed at was the minimum cold water supply requisite to retain all the ammonia, in order to avoid throwing extra work on the desulphuriser. Although a test cock was fixed in the last tray but one and instruction given to the attendants to test the gases there with litmus paper every half hour, ammonia escaped at times to the purifiers. At the time of testing, the tower might have three sections cold while a few minutes later, with the water supply unaltered, a change in the desulphuriser conditions would send forward a rush of gas and ammonia would be lost. In order to keep a check upon the working of the tower, a thermograph was connected with the last tray but one in order to record to within 0.5° C. the temperature of the liquor in that particular tray and so, indirectly, to indicate whether ammonia was passing. An improved efficiency is expected from the use of this instrument, but the plant was closed down before this was proved.

(b) With regard to the ammonia stills themselves, during the time the plant was in work it was impossible, owing to lack of boiler power, to work the plant to its full capacity. The lime mixer required cleaning out about once a fortnight. The effluent from the stills during the period of working averaged 0.011%  $\text{NH}_3$ .

(c) The losses which occur due to pumping are difficult to determine and may attain considerable proportions. An Evans pump, 5 in.  $\times$  4 in.  $\times$  6 in., is fitted for pumping the desulphurised liquor and needed packing every two or three days at the liquor end. After trying various types of packing special S.E.A. rings were fitted and they had remained tight for three weeks, when, as previously mentioned, the plant was changed to the manufacture of sulphate of ammonia. The concentrated liquor pump, with ordinary care, and owing to not being used so frequently, gave little trouble. An Evans pump, 4 in.  $\times$  3 in.  $\times$  4 in., fitted for the purpose of dealing with the hot liquor from the reflux pot, gave much trouble. In order to avoid considerable leakage this pump had to be packed daily, due to the pump rod being rapidly attacked, apparently owing to the action of cyanides, and so developing a "shoulder." In the course of three months the rod was reduced in diameter by  $\frac{1}{2}$  in. where in contact with the liquor. When the plant is restarted other types of rod will be experimented with.

(d) The storage tanks, two in number, are connected by 2-inch pipes with a small apparatus known as a "bubbler," the function of which is to deal with the ammonia gas expelled from the tanks. It resembles a small still and has four trays 21 inches in diameter; when fed with a dribble of water from a  $\frac{1}{2}$ -inch pipe it is quite capable of dealing with the gases normally expelled, the weak liquor thus formed flowing to the reflux pot and thence to the lime mixer. Should the storage tanks become warm, however, ammonia escapes from the bubbler unless the stream of water is largely increased; an apparatus with a larger area would have been preferable. Careful attention should therefore be given to the outlet temperature of the final condenser. No trouble has been experienced by choking of the bubbler trays as, fortunately, the water supply is fairly soft.

(e) This source of loss is one which, at first sight,



seemed of small consequence, but upon investigation yielded surprising results. Rail tanks are usually provided with a loading or delivery pipe which reaches to within 2 or 3 inches of the bottom, while the displaced gases escape through a smaller hole in the top of the tank. It was at first thought that, by merely connecting this outlet with a 40-gallon cask of water, the ammonia escaping from the tank would be absorbed. However, after loading a tank an olfactory examination of the cask contents was unpleasant. Other casks were therefore prepared containing definite volumes of sulphuric acid and determinations carried out on the total ammonia displaced upon loading six 10-ton rail tanks. The figures obtained showed an average loss equal to 15 gallons of 25% liquor per tank. This figure must only be taken as approximate, because many factors, such as rate of pumping, strength of liquor, and atmospheric conditions influence the result. The tests were carried out during the month of February, the atmospheric temperature being about 50° F., while the liquor in storage was 65° F. In order to avoid the loss of ammonia while loading tanks, a pipe was permanently carried to the "bubbler," which was fed with extra water during loading, the rail tank being connected to the pipe by flexible hose.

(f) Frequent examination is necessary, because small leakages readily develop into large ones and often become all the more difficult to rectify. All gauge cocks should be moved daily because if once fixed they readily break, being made of cast iron. Careful warming up of the plant is one of the best methods for preventing numerous small leaks on the still and reflux condensers.

No attempt, as indicated by the title, has been made in this paper to exhaust the numerous problems arising in the manufacture of concentrated liquor, but merely to point out various sources of loss and suggest how they may be minimised. It must also be remembered that much remains yet to be accomplished in the design of plant, for although many plants will yield a product of specified quality they will only do so at the expense of disproportionate quantities of steam and cooling water.

In conclusion the author desires to express his thanks to Mr. J. Wesley Whimster, Engineer and Manager of the Bath Gas Light and Coke Company, for permission to publish particulars relating to the above plant.

#### DISCUSSION.

Mr. P. PARRISH remarked that the problem of the desulphurisation of ammoniacal liquor had hardly been approached scientifically. Most makers of plant had assumed that a long column still, designed on the lines of an ammonium sulphate still, would answer satisfactorily as a decomposer. No attempt had been made to dissect the respective operations involved in the desulphurisation of gas liquor. He had found that, within limits of 3 lb. pressure—i.e., about 6 in. mercury gauge—above atmospheric pressure, there was a practically coincident relationship between the boiling-point of water and the point of almost complete dissociation of the ammonium sulphide and ammonium hydrosulphide present in ammoniacal liquor. Further, to conform to the Ministry of Munitions specification in respect of concentrated ammonia liquor—viz., 25%  $\text{NH}_3$  with a maximum of 0.5%  $\text{H}_2\text{S}$ —it was necessary that the gas liquor in the terminal tray of the decomposer should attain a temperature coincident with the temperature at absolute pressure, to which the liquor in the bottom, or terminal, tray was subjected. Having established this, it was readily conceivable that appreciable volumes of steam, ammonia, carbon dioxide, and hydrogen sulphide were released. It would appear that the most judicious arrangement

of plant was one which dissected the operation of desulphurisation into three parts—dissociation or decomposition; cooling; absorption. The dissociator should preferably comprise a still with about five trays, affording adequate area for the ascent of the gases, and equally adequate space for the descent of the liquor. To facilitate the recovery of the ammonia released in the dissociator, it was necessary to control the temperature of the gases leaving the latter apparatus. This could best be effected by cooling the liquor, by passing it through a multitubular cooler, so as to attain efficient absorption of the ammonia in the absorbing vessel. The absorber should be constituted of about eight trays in all, softened water being admitted to the top tray; the gas liquor to be desulphurised should be introduced to the fifth tray from the top. Plant designed on this basis, he believed, would be capable of fulfilling the assigned functions efficiently, and more economically in point of steam consumption than the majority of decomposers supplied hitherto. As regarded the losses of ammonia, he thought that the author had insufficiently emphasised the loss likely to arise from the effluent liquor from the ammonia still. He advocated the use of a pump provided with multiple glands for the concentrated ammonia liquor, so as to obviate loss of ammonia at this point.

Mr. MARSDEN, in reply, said he agreed that Mr. Parrish's method of desulphurisation was a good one; but as there were a large number of plants of the Wilton type—very similar to the one he had described—in use, he thought the difficulties he had described might help some of those who had to work these plants. At the same time, he agreed that Mr. Parrish's method would be preferable to the large type of desulphuriser fitted by the makers of the Wilton plant. The percentage of ammonia in the effluent during the test was taken by means of a Whitehead sampler, which took samples every hour.

#### NOTES ON THE CATALYTIC AND THERMAL SYNTHESSES OF AMMONIA.

BY E. B. MAXTED, PH.D., B.Sc.

The direct combination of nitrogen and hydrogen with formation of appreciable percentages of ammonia may take place under two entirely distinct sets of conditions. Firstly, according to the well-known Haber synthesis, nitrogen and hydrogen are allowed to react, at a moderate temperature and at a high pressure, in the presence of a suitable catalyst such as iron. In the second place it has recently been shown by the author that the percentage of ammonia in equilibrium with nitrogen and hydrogen at a given pressure firstly sinks with increasing temperature in accordance with Haber's observations, then passes through a minimum value, and finally rises once more until at, for instance, the temperature of the high tension arc, the percentage of ammonia in equilibrium with its component gases at atmospheric pressure has risen to nearly 2% by volume, a value through which it has already passed on the other (i.e., the decreasing) side of the equilibrium temperature curve at a temperature of about 320° C.

It is proposed, in the first part of the present paper, to discuss quite briefly several points of interest which have arisen in the course of work on the Haber synthesis of ammonia, the second part of the paper being devoted to some description of the methods employed and results obtained in connection with the measurement of the ammonia equilibrium at high temperatures.

#### Part I.—Catalytic synthesis.

From the point of view of efficiency it would appear desirable to operate the synthesis under

such conditions that each litre of catalyst space gives the greatest possible yield of ammonia per hour, in other words so that the number of kilos. of ammonia per litre of catalyst space per hour, is at its maximum value. This is undoubtedly true in so far as it concerns the desirability of employing as active a catalyst as possible, provided of course that this catalyst has sufficient power of resistance to possible traces of "poisons" in the circulating gases; but it requires considerable qualification where a high yield of ammonia is obtained by means of rapid circulation.

If, in this connection, the progress of ammonia formation with increasing time of contact between the gas and the catalyst be noted and the yield of ammonia calculated for each case, it will be seen, as is well known, that the maximum yields of ammonia are obtained in conjunction with a short time of contact and consequently with a low percentage of ammonia per passage.

Table I., which records a typical result with an iron-potash catalyst at 550° C. and at 150 atmospheres pressure, is illustrative of this point.

The improved results over and above those reported last year are due principally to a modification in the method of working.

TABLE I.

Time of contact of gas with catalyst in seco. ds.	Percentage of ammonia formed.	Yield of ammonia in kilos. per hour per litre of catalyst space.
0.34	0.65	2.7
0.56	0.94	2.3
1.8	1.8	1.4
3.6	3.2	1.25
7.2	4.7	0.91
10.8	5.1	0.65
24.5	6.8	0.38

The highest possible yield of ammonia for a catalyst chamber of given size is thus obtained with a very high speed of circulation and with a minimum value for the percentage of ammonia formed per passage.

It must, however, be remembered that any decrease in the percentage of ammonia formed per passage involves a corresponding increase in the number of passages of the gas mixture through the catalyst chamber while each kilogram of ammonia is being formed, and that this necessitates an ever increasing amount of work in order to compensate for the deficiencies of the heat exchangers, also of those connected with the refrigeration system where this method of ammonia elimination is employed in preference to absorption by water.

It will accordingly be seen that the most economical conditions for the synthesis do not correspond with those necessary for a maximum yield per hour, but that an optimum rate of circulation exists, which can easily be calculated for a given system from measurements, with various rates of gas flow, of the temperature differences at like ends of the exchangers employed.

The next point which appears to call for attention is that of the most suitable working pressure. It is obvious that a low-pressure system presents many conveniences in working, principally in connection with the possibility of employing larger and simpler furnaces than are applicable for higher pressures. On the other hand any increase in the pressure employed leads to a corresponding increase not only in the value of the equilibrium ammonia percentage for a given temperature but also in the velocity of formation of ammonia. Further, since the power required for the initial compres-

sion varies directly as the logarithm of the pressure used, a high-pressure system will entail very little extra expenditure of power for compression over and above that required for a system working under a comparatively low pressure. Thus, if we take the power required to compress a gas mixture to fifty atmospheres as unity, that required to compress up to 100, 150, and 200 atmospheres, respectively, is 1.2, 1.3, and 1.35. Lastly, a high-pressure gas exchanges heat better than one at a low pressure and requires therefore less expenditure of energy in the exchangers, while, on account of the smaller size of the complete plant, radiation losses are comparatively small.

The relative values at various working pressures for the percentage of ammonia per passage and yield in kilos. per hour per litre of catalyst space with the same catalyst and under the same conditions as those employed for Table I. may be seen from Table II.

TABLE II.

Velocity of flow of gas in cubic metres per hour at N.T.P. per litre of catalyst space.	50 Atm.		150 Atm.		180 Atm.	
	% NH <sub>3</sub> formed.	Yield in kilos. per litre per hour.	% NH <sub>3</sub> .	Yield.	% NH <sub>3</sub> .	Yield.
20	2.2	0.34	4.85	0.75	6.7	1.0
40	1.2	0.37	3.7	1.1	5.4	1.65
60	0.8	0.37	2.9	1.35	4.5	2.1
80	0.65	0.4	2.25	1.4	3.9	2.4
100	0.55	0.42	1.8	1.4	3.5	2.7

It will be noted that even at 50 atmospheres pressure it is possible to obtain a fairly high percentage of ammonia per passage, associated with a moderate amount of power for compression, by providing the plant with a series of relatively large catalyst chambers, which may be of simple design. There are, however, disadvantages of the nature already discussed and it would appear more economical in practice to employ as high a pressure as is compatible with ease of working, 180 atmospheres being found very suitable.

TABLE III.

Catalyst, iron-potash. Pressure=150 Atm.

Time of contact in seconds.	T = 530° C.		T = 580° C.	
	% NH <sub>3</sub> .	Yield in kilos per litre per hr.	% NH <sub>3</sub> .	Yield.
0.6	0.96	2.1	1.5	3.5
1.0	1.3	2.0	2.4	3.3
1.5	1.7	1.7	3.2	2.9
2.0	2.05	1.4	3.8	2.6

Thirdly, dealing with the question of a suitable working temperature, it must be borne in mind that, as the synthesis is usually carried out, the percentage of ammonia formed per passage never approaches the equilibrium value for the temperature and pressure used, and thus, within limits of course, reaction velocity will play an even more important rôle in the determination of the amount of ammonia formed during the time of contact than the absolute value of the equilibrium ammonia percentage. If, on the other hand, the temperature employed be so high that the desired percentage of ammonia unduly approaches the equilibrium percentage, the increase in reaction velocity due to increased temperature will no longer compensate for the decreased equilibrium ammonia content; further, at unduly high



temperatures the catalyst becomes less active owing to sintering.

The advantage of increasing the temperature, notwithstanding the decrease in the equilibrium ammonia percentage, is shown in Table III. for temperatures approximating to 530° and 580° C. respectively.

In concluding these notes on the Haber synthesis, I should like to thank Mr. G. F. Hanna for his assistance in the determination of the experimental data given above.

#### Part II.—Thermal synthesis.

While the possibility of a thermal synthesis of ammonia is certainly of considerable general interest at the present time, it would be just as certainly premature to discuss any possibility of its technical application, especially with the very limited amount of knowledge which is available. The nature of ammonia equilibria at high temperatures and the methods which have been employed for their determination appear, however, to deserve discussion, and it is felt that the following notes may well be given without apology as to their only indirect technical interest.

It appears desirable in the first place to give some description of the determination of the approximate value for the ammonia equilibrium at the temperature of the high tension arc. In order to obtain this value it is necessary to "freeze" equilibrium by an extremely rapid cooling of the gas mixture from arc temperature to below that at which ammonia decomposes with measurable velocity. This rapid cooling is effected only with considerable difficulty when a comparatively large arc is employed, but may be carried out extremely simply with an arc of small dimensions. For the measurement of equilibrium the arc is allowed to burn within a capillary tube in order that the whole of the gas, which during

terminals of an induction coil to approach until the spark discharge becomes transformed into a small and apparently continuous high tension arc flame of high temperature. Spark discharge as such has only a comparatively feeble effect on the combination, as was observed, for instance, by Deville and later by Donkin, and it is found that energetic formation of ammonia only takes place when the electrodes are brought so near together that a small more or less continuously burning high tension arc flame is formed instead of a spark.

Equilibrium at arc temperature was determined by observing the progress of the reaction,  $3\text{H}_2 + \text{N}_2 \rightleftharpoons 2\text{NH}_3$ , from both sides. The apparatus employed is illustrated diagrammatically in fig. 1. It consists of a graduated U-tube, A, provided with a three-way capillary tap at the top of its graduated limb and with a small outlet cock at its bottom near the bend.

This tube, which has a capacity of 100 c.c., is connected, as shown in the figure, to a capillary tube, B, which is bent twice at right angles, and passes by way of a second capillary three-way stopcock, C, to the small absorption vessel, D. D contains very dilute ammonia-free sulphuric acid and is provided with a wide side-neck and with a stopcock for filling and emptying respectively. At a convenient place in B two platinum wire electrodes, F and G, are inserted, leaving a spark gap of the required size, which for the measurements about to be described was 0.5 mm., the tube containing this spark gap being 0.6 mm. in internal diameter. E is a tap funnel containing mercury and provided with a removable jet, by adjusting the size of which the gas contained in A may be caused to flow at the required rate through the small arc and into the dilute acid contained in D.

The determination of the percentage of ammonia formed with a given time of contact with the small arc was carried out by filling A with a nitrogen-hydrogen mixture (25% nitrogen, 75% hydrogen) through its three-way cock, this being effected by allowing the mercury, with which A was originally filled, to flow out of the small tap provided for that purpose at its bottom. This gas was then driven at the required rate through the arc by allowing mercury to flow into A from the tap funnel, E, by way of a jet of suitable size. The tap, C, is, for determinations of ammonia formed, left in constant communication with D, and may if desired be omitted. At the end of the experiment the arc is stopped and the dilute acid in D, containing the ammonia produced, is run into a tall cylinder, and the ammonia estimated by means of Nessler's reagent. It was found inadvisable, on account of frequent fractures, to employ a heavy discharge for arc formation, the most satisfactory conditions being obtained with a "two inch" coil supplied with a primary current of about 3.5 amperes from the laboratory 220-volt main.

Fig. 2, bottom curve, shows the percentage of ammonia formed with various times of contact and it will be seen that a gas containing 1.5% of ammonia by volume is produced with a contact of 0.004 second. It is difficult to estimate the temperature of the small high tension arc formed, which, however, for the purpose of obtaining rough comparative figures for the various times of contact has been assumed to be about 3000° C. The rise in the ammonia yield with increasing temperature at high temperatures (in contradistinction to the fall with increasing temperature at moderate temperatures) is to be noted, and it will be seen further that sufficiently rapid cooling for the retention of the ammonia produced is effected

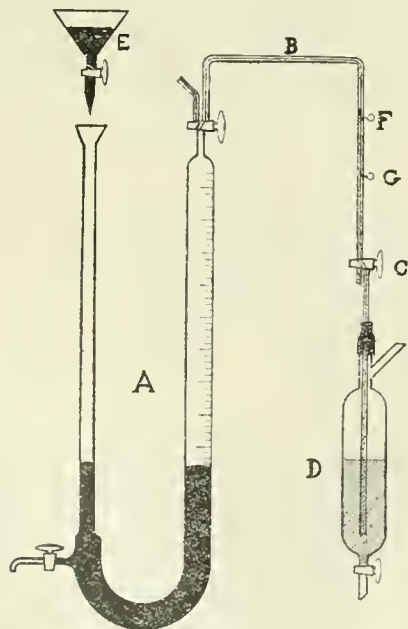


FIG. 1.

the course of the experiment is passing through the capillary, may be subjected as uniformly as possible to the heat of the arc flame. It has been found that a very convenient arc of small dimensions may be formed by causing the secondary

In this case by contact with the relatively cool walls of the capillary.

This synthesis curve would seem to indicate that the equilibrium ammonia content for a hydrogen-nitrogen mixture under the above conditions is between one and two parts per cent. by volume.

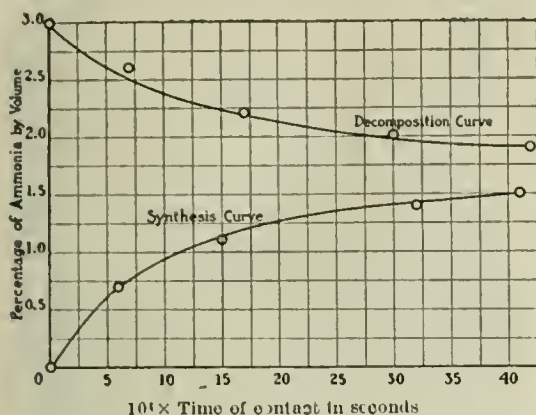


FIG. 2.

Accordingly it was considered interesting to study the effect of passing through the small arc a nitrogen-hydrogen mixture, as before, but containing 3% of ammonia. This ammonia content, being above the equilibrium value, sank instead of rising after contact with the arc, the experimental results obtained with various times of contact being shown in the top curve (decomposition curve) of fig. 2. In this case, the gas was, by operating C, only allowed to pass through D during the actual experiment. It will be seen that the equilibrium ammonia percentage appears to lie at about 1.7% for atmospheric pressure under the conditions studied. Finally, an attempt has been made in fig. 3 to

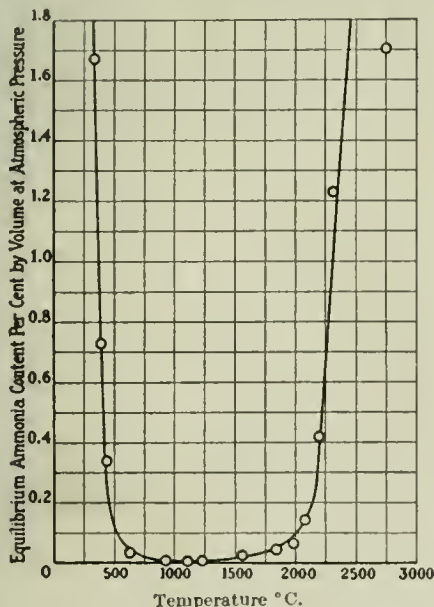


FIG. 3.

plot out the complete experimental ammonia curve up to about 3000° C. The figures both for temperature and for equilibrium ammonia content are very rough approximations only, except for temperatures below 1000° C. where ammonia formation

is effected catalytically according to Haber's measurements. The values for temperatures ranging from 1000° to 2300° Abs. have been determined by the sudden cooling of a nitrogen-hydrogen mixture in flames burning under water in a manner already described in detail (this J., 1918, 105 T).

While it is not proposed to discuss in the present paper the influence of pressure on the thermal synthesis, it is, however, interesting to note in connection with these earlier results that an increase in pressure from about one-tenth of an atmosphere to one atmosphere has given the anticipated increase in the amount of ammonia formed.

## SCIENTIFIC GLASSWARE.

BY DR. MORRIS W. TRAVERS, F.R.S.

Before the war the whole of the light hollow glassware used in our chemical laboratories was imported into Great Britain from enemy countries, and practically the whole of the glass tubing and the glass instruments were obtained from the same sources. In August, 1914, supplies were cut off; and though the stocks held by some dealers in these goods were perhaps unusually large, it soon became clear that, should the war last for more than a few months, our industries, our public health organisations, and our hospitals would be seriously handicapped.

A few days after the outbreak of war I met my friend Mr. Douglas H. Baird, Chairman of Messrs. Baird & Tatlock (London), Ltd., who explained the position to me, laying particular stress upon the importance of chemical glassware in the steel industry, upon which a heavy strain was likely to be thrown, and upon the demand for glassware by the Army Medical Department and the hospitals. He told me that his firm were already considering the possibility of manufacturing scientific glassware upon a scale sufficient to carry them over the war, and were endeavouring to secure the co-operation of other dealers. He asked me if I would assist him, and I readily agreed, though I admitted that I had no wider experience of chemical technology than the average academic chemist. I may say that now, after spending almost the whole of my time during four years actually in the works, I approach the great problem of science and industry in a more humble spirit than may have possessed me when I was a professor of chemistry. I am inclined rather to blame academic chemists for the neglect of industry than the manufacturers for the neglect of science.

Mr. Baird's negotiations with other dealers came to nothing, and his attempts to interest certain glass manufacturers in scientific glassware were equally fruitless, so he decided to finance the undertaking himself. Professor H. B. Baker, F.R.S., had in the meantime very kindly placed at my disposal a laboratory in the Imperial College of Science and Technology, to enable me to complete a piece of scientific work which I had commenced in India, and in the month of October, 1914, in conjunction with Mr. Baird and Mr. T. Allen, a director of Baird & Tatlock (London), Ltd., I commenced an investigation on the glasses manufactured in Germany. Mr. Baird and Mr. Allen supplied me with a very large number of samples of glass, and with the fullest information as to their origin, qualities, and the opinions of their clients upon them. Thus, with regard to glasses for "lamp working," that is to say working with the table blow-pipe, I was able to obtain the opinions of skilled glassworkers, who used this material in very large quantity, "Jena" glass, "R" glass, Bohemian glasses, and glasses used for minor apparatus, such as test-tubes, and for heavy hollow ware, such as desiccators, were all reviewed and analysed, and at the end of four months I had obtained a fair insight



into the nature of the glasses used in making scientific glassware. A very considerable number of complete analyses were made at the time, and the analytical work carried out afterwards at the works upon thermometer glasses, lamp glasses, etc., extended our knowledge of the subject. I must remark here upon the very slight amount of information which was to be gained from scientific and technical literature upon the subject of glass.

It was decided to erect a glass factory in Walthamstow, adjacent to Messrs. Baird & Tatlock's scientific-instrument factory, and before commencing operations Mr. Baird secured the services of Mr. F. Winchurch, a highly qualified glassworker, as technical expert, and to his initiative, skill, and endurance such success as we have attained to is largely due. In the month of March, 1915, a small oil-fired furnace, capable of founding glass in quantities of about 1 cwt. at a time, was erected on the site of the works, in a temporary shed, and Mr. Winchurch and I proceeded to experiment on the basis of my analyses of glasses and raw materials. Batches were made up first from chemicals corresponding as nearly as possible to the constituents of the glasses, and then using commercial materials such as china clay, felspar, etc. Tube and hollow articles, such as beakers, were made from these glasses, and their properties were tested.

We now decided that we could make a soda-potash-lime-alumina glass, equal to the best German lamp-working glass, and a resistance glass similar to the "Jena" resistance glass, but modified in a manner calculated to remove the objection that the "Jena" glass gave up zinc to acids. My judgment upon this point has been confirmed by the results of a careful investigation recently carried out by Professor Turner and his staff in the Department of Glass Technology, Sheffield University, which has proved that "Duroglass" resistance glass is the most highly resistant to chemical reagents of any glass yet placed upon the British market.\*

We next proceeded to build a larger oil-fired furnace for pots holding about 2 cwt. of glass. At that time Government was anxious for manufacturers to adopt oil fuel, but our experiments led to the conclusion that, though oil-firing was applicable to tank furnaces it was not suitable for pot furnaces, and the rapid increase in the price of oil during the second quarter of the year 1915 completely negated any advantage which might arise from the use of it. The experiments were therefore brought to a conclusion. By means of this furnace we were, however, able to manufacture glass in sufficient quantity to experiment further on the blowing of hollow ware, and it did good service in enabling us to manufacture some articles which were urgently required by Government in connection with gas warfare.

We had now to erect a furnace capable of producing glass at the rate of at least 5 tons per week, with the least possible delay, and we decided upon a type, actually in use, which was based upon the principle employed in connection with the gas-firing of horizontal gas retorts. It was a four-pot furnace, for 36-in. by 38-in. covered pots, each contained in a separate arch, and heated by gas from coke in a producer. The gas, issuing from nostrils, as in the gas-retort setting, met the secondary air, and burned below the siege, upon which the pot

in each arch rested, and the flames passed up channels at the side of the siege, surrounded the pot, and escaped through a flue at the top of the arch into the stack. The furnace was non-recuperative, and consequently not an economic one, but it cost little to build, and was erected and at work in six weeks. After I had entirely redesigned the setting, so as to burn the gas at the sides of the arch, and a little below the level of the top of the siege, instead of underneath the siege, it gave excellent results. It is still at work, and three, and sometimes even four, pots of tube metal, which is by no means too easy to plain, can be obtained from each pot every week. It does not give us much trouble now that we understand it. Later, we built a six-pot furnace of the same type, which has given good results, so far as output of glass is concerned, but which is very extravagant of fuel. We are now installing two Hermansen recuperative furnaces in a new glass-house.

In speaking of furnaces the efficiency is often expressed in terms of glass founded per ton of fuel consumed, but the expression is misleading, for the fuel consumption cannot be materially reduced while the pots are being worked, and the rate at which they are emptied depends upon the nature of the goods being blown. Two men will empty a half-ton pot of glass in a few hours making five-litre flasks, but six men might work for a couple of days out of a similar pot blowing small articles.

Unless the furnaces run well metal will run short, and what is much worse, the metal will be bad. But if the glass pots will not stand the heat, and continually crack, work may be brought to a standstill altogether. During the past year or two a great deal has been heard about the inferiority of English pots generally, and the cause of trouble with pots has been attributed to the fact that the demand has obliged manufacturers to use them before they are properly matured. However, having made a systematic study of the question, I have come to the conclusion that the fault lies mainly, though not entirely, with the glass makers rather than with the pot makers. The most usual complaint is that the pots split down the front, either through, or below the lip. The reason is as follows. The green pots are first heated in a separate furnace, the pot arch, usually direct fired with coke or coal to a temperature, which is not as a rule determined, but which probably rarely reaches 1100° C. The pot is then transferred to the glass furnace, and the front is built up, leaving the working opening, which is closed by a loose clay stopper. As soon as the pot has apparently attained the temperature of the furnace it is filled.

Now, the firing of a green pot in a furnace in which the pot is exposed directly to the flames is obviously a delicate process, and when one considers that the stoking is carried out by two or three shifts, it is somewhat remarkable that so large a proportion of the pots survives the process at all. Indeed, unless the pot is carefully made, and allowed to mature for a long time, it will not survive. If the firing were carried out in a muffle kiln, much trouble would probably be saved, and danger to the pots averted. Provided that sufficient care is taken, there is, however, no reason why the pot should be not quite sound when it is set in the furnace.

Now, when the temperature of the pot arch does not exceed 1100° C. the pot, when set in the furnace, is still in the biscuit condition, and if the temperature is then raised to 1300°–1400° C., as is usual in the case of any modern type of furnace, the material of it will become semi-vitreous in appearance, and the change will be accompanied by an increase of density from about 1.5 to 2.2, and by consequent shrinkage of the pot, the material of which will also become very much more

\* Prof. Turner informs me that, as the result of a recent investigation, he has found that a 500-c.c. flask, after boiling for 130 hours with strong hydrochloric acid, during which time 9 litres of the acid was evaporated, dissolved to the extent of about 75 mgrms., or about 0.0001 gm. per sq. dem. per hour. There was no selective solution of the constituents of the glass, which, so far as it dissolved at all, dissolved as a whole. Using the purest acid obtainable, the amount of each of the constituents of the glass which dissolved in the acid was, with the exception of the silica, less than the quantity of the same substance which was originally present in the acid.



highly resistant to the action of molten glass. However, if the ordinary procedure is adopted, while the back and sides of the pot become completely altered, the front remains in the biscuit condition, and owing to strains set up by the shrinking of the back and sides, cracks appear in the front, sometimes starting from the lip of the pot, sometimes appearing below it. When a small crack forms, owing to the low resistant qualities of the biscuit clay, it rapidly increases. Further, when the front of the pot is not properly burnt it rapidly corrodes, scaling from the inside, and being washed away on the outside by the glass which runs down between the pot and the pad below the lip.

When we set a pot in the furnace it is now our practice to build a temporary 9-inch wall, 9 in. from the front of the pot, so as to cover the opening of the furnace completely, and to leave the pot without touching it for three days. The temperature of the whole pot rapidly rises to the full heat of the furnace, and it becomes completely and uniformly burnt. The upper part of the wall is then pulled down and the front is built up for working. Special tools are used in this work, for the whole of the pot is white hot, but the operation presents no special difficulties. Though a pot is out of work for three days, the fact that its life is lengthened and that it does not scale, compensates for the loss of time.

During the war a good deal of work has been done in connection with the refractories used in the glass industry. This work has, however, generally been undertaken from the point of view of the laboratory, and not of the works. I cannot help thinking that the problems connected with glass pots should first have been attacked with a view to rectifying the faulty methods of burning the pots which are in common use, so as to enable glass makers to utilise to the best advantage the excellent material supplied to them by the British pot makers. No improvement in the material can compensate for unscientific works practice.

The making of glasses for use in the manufacture of scientific apparatus opens a very large field for research. Investigations on the composition of glasses which have been carried out by ourselves and others during the past few years have supplied us only with the bare minimum of necessary information. On the series silica-alumina-lime-soda-potash, with or without boric acid, there remains a vast amount of systematic work yet to be done by the chemist and manufacturer working in co-operation. We know, for instance, that a pure silica-alumina-lime-soda glass does not work as well before the blowpipe as a glass containing potash, and also tends to devitrify, particularly if it has been in stock for some time. But what is the proportion of potash to soda which will give the best result? Only a research conducted by the chemist, the glass manufacturer, and the practical glass-blower can tell us. Again, with certain proportions of potash the glass becomes difficult to free from minute seeds. Combinations of the same constituents for the manufacture of Petri dishes, and similar articles which must be highly resistant to the action of superheated steam, and as clear as "flint" when blown, also require investigation.

Glass is at the best a compromise, and this fact never becomes more evident than when one is considering the composition of the so-called resistance glasses. I should here like to call attention to the excellent work on the properties of these glasses which has been carried out by Professor Turner and his colleagues in the Department of Technology of the University of Sheffield, which will prove of great value to the manufacturer. However, while the manufacturer should do his utmost to provide the chemist with glassware which uniformly resists attack by ordinary chemical reagents, it must not be forgotten that the quality of the articles manufactured depends, not merely upon the chemical

composition of the glass, but upon properties which can at present only be determined in the glasshouse. If a glass melts easily it does not follow that it will plain easily, that it will be possible to work it over a sufficiently wide range of temperature to allow of economic production, or that it will not tend to become "cordy" upon the slightest provocation.

There is scope for a considerable amount of research on the mixing of "batch," and in connection with the mixing of batch and cullet. In the case of the batches used for making flint glass the roughest procedure may give excellent results; but while china clay may be used with invariable success as a source of alumina, when the batch is mixed mechanically, hand mixing of the batch containing china clay will produce, not glass, but a substance closely resembling pudding. I believe that considerable advantage is gained by grinding such materials as china clay with one other constituent of the batch. According to British glasshouse tradition, the cullet must be sieved to remove powdered glass, and therefore the grinding of cullet is a process which need not be considered. Actually, the cullet is sieved to remove accidental dirt, to which too little attention is paid in our glasshouses. Our observations lead us to the conclusion that considerable advantage may be gained from grinding the cullet, and mixing it with the batch.

The study of the formation of "cords" in glass is well worth attention. I believe that there are at least two classes of cords, but those which find their origin in ill-digested particles of clay, or scale from the pot, or from the material of the batch, may be ruled out of the investigation. A cord of this kind in a piece of tube, or hollow blown article, will be found to end in a little mass of jelly-like glass, sometimes surrounding an opaque particle. The glass-maker will say that these cords are due to "lack of heat," which is certainly true in the sense that if the pot is hot enough during the found, even fairly large particles of clay will completely dissolve. However, during the working of a pot of metal, the glass sometimes "goes cordy," and remains cordy, even when worked with a ring in the pot, and sometimes goes clear again in a truly remarkable manner. This is possibly a surface phenomenon, and is worthy of the attention of those who have made a study of colloids. I am collecting data on the subject, and shall be glad to hear of others who are interested in it.

Scientific glassware, made in the glasshouse, includes hand-made articles, articles blown in the mould and finished by hand, and articles which are blown in the mould and finished outside the glasshouse. Probably no foreign workman is the equal of the English craftsman at handwork, but though I hope we shall never see the famous English glasshouses abandoning art for industry, I do hope to see a less rigid adherence to old-fashioned methods of working, in connection with our new industry. The glass-worker's chair, as a machine, seems to me to be a very crude appliance for giving rotary motion to a blowing iron, and to the glass on the end of it, and should rank with the bow drill, and other implements of bygone ages. The glass-worker will tell you that he has more power over the glass while running the iron up and down the chair, but this appears to be one of those traditions which it is so difficult to overcome. In finishing such articles as beakers and flasks, and in many similar operations, which are carried out in the finishing shop, the chair has been abandoned for the machine, or mechanical arrangement, by which the article is rotated about its axis, without lateral movement, with the result that a comparatively unskilled girl can, after a few days' training, carry out operations which would otherwise have to be placed in the hands of a highly skilled workman. Further the girl's work on the machine is the more



satisfactory. Similar mechanical methods must be introduced into the glasshouse.

Except in the case of electric lamp bulbs, very little light hollow ware was manufactured in this country before the war, and, as may well be imagined, a good deal of experimental work had to be done before satisfactory methods were arrived at. Since I have become a glass manufacturer I have been amazed at the variety of common articles which the chemist and bacteriologist make use of. The result of this is that, while some few sizes of beakers and flasks can be made in fair quantity, the trade mainly consists in manufacturing comparatively small quantities of a large variety of articles. As neither dealers nor users hold large stocks, and are always asking for supplies in dozens of articles at short notice, the manufacturer is at a disadvantage. Were it possible to standardise chemical and bacteriological glassware, both the consumer and manufacturer would benefit. Owing to the variety of the goods to be manufactured, and the comparatively small demand for them, the introduction of automatic and semi-automatic machinery into this branch of the trade is at present hardly practical politics.

The question as to the thickness of the beakers and flasks has been the subject of careful consideration. Bohemian ware was always very thin, as the glass from which it was made was not resistant to heat. With resistance glasses it is possible to make beakers and flasks very much heavier than the Bohemian ware, and in this respect we, and the American manufacturers, have followed the practice of the makers of the Jena glassware. A 500-c.c. beaker should be about 0.8 mm. thick, and a 1000-c.c. beaker 1.0 to 1.2 mm. thick, but the articles must, of course, be evenly blown. Such articles as graduating flasks and X-ray bulbs must be blown exact to weight.\*

The satisfactory annealing of the hollow-blown ware gave us more trouble than the whole of the other processes put together. When a beaker or similar article is blown, it is first annealed, then cut to the right height, flanged, badged, and finally re-annealed. The scientific part of the annealing process has been carefully studied by Mr. F. Twyman, of Messrs Adam Hilger, Ltd., who has also perfected an optical strain viewer, with which all our goods are tested before issue. However, the practical treatment of the subject was not easy, particularly as we were hampered by tradition.

The lehr which is used in most English glasshouses is an arched chamber upwards of 40 feet long, with a fire-box on one side, at the hot end. The articles when placed in the pan are heated directly by fire, and as the pans are drawn down the lehr they gradually cool off. This method serves to anneal even complicated hand-worked ware made from flint (lead) glass, but it proved totally inadequate for resistance beakers and flasks. This is somewhat surprising, for since the goods are light they should anneal easily. However, I believe that we have here to consider the existence of a skin effect, which may possibly be connected with the formation of cords to which I have already referred. This effect is not taken into consideration in dealing with the general problem of annealing, but its existence may account for the fact that unless the glass is thoroughly soaked at the annealing temperature it is not properly annealed. In the case of scientific glassware we are dealing with an enormous bulk for very small weight, and as the goods must pass very slowly through the hot part of the lehr, unusually large lehr accommodation is required. This is a fact which we altogether failed to recognise when we designed our first glasshouse.

The cost of town gas being prohibitive, we heat our lehrs by means of producer gas. The gas enters a space below the bed of the lehr, which is formed of a brick arch, and then passes up ports at the side. Secondary air enters through slides at the front, and meets the gas at the bottom of the ports, and the length of the flame issuing from the ports, and consequently the temperature of the lehr, can be increased or reduced by closing or opening the air slides. The temperature of the hot part of the lehr can be regulated by this means to within 10°, and as the hot gas from the producer heats the bed of the lehr from below, a uniform temperature can be maintained. The fall of temperature along the lehr is regulated by taking away the hot gases into a flue, through openings which are closed by dampers. Resistance glass may be removed from the lehr at 300° C.

It is neither necessary, nor possible, to anneal chemical hollow ware as perfectly as glass which is required for optical purposes. However, uniformity in the output is absolutely essential if the manufacturer is to inspire confidence in his customers, and therefore the problems connected with annealing demand his closest attention.

When a beaker, or similar piece of apparatus is blown, it resembles a bottle, and the next stage in the process is to cut it to the proper height. The process by which this is done is well known. The article is slightly scratched with a diamond, and placed on a revolving table, while a fine blowpipe jet plays upon it at the height of the scratch. If the goods are well annealed, and evenly blown, they crack off quickly and evenly, provided that the jet is properly adjusted. Badly annealed goods and badly blown goods crack off so as to leave jagged edges. Thus the efficiency of production depends very largely upon efficient first annealing.

The flanging and lipping of beakers, and the flanging of flasks, are operations which are carried out entirely by machines operated by girls. The designing of these machines did not call for a vast amount of ingenuity, but in perfecting the details it was necessary to study the processes very closely in the works. There is scope for the engineer in the finishing shop as well as in the glasshouse.

Space will not admit of a lengthy discussion of the problems of the graduating shop, but I should like to call attention to the very great scope for research in this direction, and to the curious fact that scientific literature includes very few papers dealing with the accuracy of volumetric measurements. Unfortunately, in this, as in other branches of the trade, a policy of secrecy acts as a bar to progress, and in establishing our department, which was undertaken in order to meet a national demand, Mr. F. C. Harris and I had to start at the beginning, and work out the various processes. Sir Richard Glazebrook, F.R.S., and Mr. Stott, of the National Physical Laboratory, kindly placed at our disposal data for the compilation of correction tables for determining volumes by weighing water, but this is the only information which we have been able to obtain from the outside.

Provided that the operator is supplied with tables from which the corrections for barometric pressure, and for air and water temperatures, can be read off easily, that due care is taken with the weighings, and that proper machines are used in reading and marking the level of the meniscus, there is no difficulty in manufacturing accurately marked flasks, and similar pieces of apparatus. However, the graduating of cylindrical vessels, such as measuring cylinders and burettes, presents greater difficulties. In the first place glass tubes, even when most carefully drawn, are never truly cylindrical, and the same remark applies to measuring cylinders, when blown in the mould. Before graduating, a 50-c.c. burette is usually marked at six points, so as to divide it into five lengths, each

\* My attention has recently been called to the shrinkage of moulds used in blowing graduated flasks. That cast metal articles, when repeatedly subjected to heat and cold, do shrink is well known.



corresponding to a capacity of 10 c.c. Each length is then divided into 100 parts, but as the distance between the fixed points is more or less unequal, the lengths corresponding to 0.1 c.c. must change suddenly in passing each of the fixed points. The graduation is, therefore, at the best an approximation. A machine has been designed which, it is hoped, will make it possible to smooth out the irregularities automatically, by means of a mechanism retarding or accelerating the motion of the carriage of the dividing machine, and actuated by a cam working against a flexible steel bar, the curve in which corresponds to the correction curve for the tube. When once the curvature of the steel bar has been adjusted to correct the setting of the machine at the fixed points, the graduation of the whole length will be possible without further adjustment. The plans are in the hands of Messrs. Munro, of Tottenham, who have also made for us a machine for graduating measuring cylinders and similar instruments, based on the principle of similar triangles. One of these machines has been in use in our works for some time, and others are being made for ourselves, and for other members of the trade.

A good deal of work might well be done with a view to improving the ordinary dividing machine, in which the carriage is moved by means of a screw, driven by a ratchet wheel and a pawl attached to the divided head. The setting error may, of course, amount to  $1 \div$  (twice the number of teeth on the ratchet wheel), and as the number of teeth is usually only 200, the error may be 1 in 400, which is too great for accurately graduated glassware. The accuracy with which the machine can be set must be increased, and means must be found to eliminate the "overthrow" of the screw, which introduces irregularities, unless the operator is very careful. Even mechanically driven machines are liable to this error, unless in perfect adjustment.

I may mention here a very useful, and simple method which we employ in determining volumes. A rod of pure nickel is bored and the hole is rounded at the bottom. The end of the rod is then carefully ground till the capacity, which is determined by filling the vessel, so formed, with mercury, and pressing a polished glass plate into the top, is exactly the volume required. In calibrating an instrument, like a burette, the glass tube is filled with water and the height of the meniscus is marked. Ten c.c., or any other volume, of mercury, is then poured into the burette from the nickel measure and, the sides of the tube being wet, the water meniscus will rise by an amount equal to the amount which it would fall if the same quantity of water were allowed to flow from the apparatus. The method, which is only a modification of one which is well known, has been proved to be one of very high accuracy, and might well be applied in the laboratory for the checking of glass instruments.

Anyone possessing mechanical ingenuity will find in the lamp-working shop a field for investigation. Manufacturers of electric lamps have for long been accustomed to employ machines for flanging glass tubes, and for sealing together the parts of the lamps, and the principles upon which their methods are based are capable of wide extension. However, while in the electric lamp factory the machines are generally designed to perform only specified functions, machines of a more "universal" type are required for use in the general lamp-working shop.

There is perhaps less difficulty in designing machines than in getting them made under war conditions.

Grinding, polishing, stoppering, and similar operations, which, in this country, are usually per-

formed by highly skilled workmen using very crude tools, demand the attention of the engineer.

In reviewing the work which has been done, and the results which have been attained, it must be admitted that we, and the other firms which are working on similar lines, are yet far from being able to supply all the varieties of scientific glassware previously imported into the country. As to whether the industry will survive the war is a question which I do not propose to discuss. It has, however, been scheduled by Government as a "key industry," and in establishing the Department of Optical Munitions and Glassware, Ministry of Munitions, we believe that the Government has in view, not only the development of the industry for war purposes, but its permanent establishment in the country. We hope that this department, which has succeeded in gaining the confidence of those engaged in the industry, will be able to play an important part in the work of reconstruction.

#### DISCUSSION.

Dr. CHARLES A. KEANE said that one of the principal difficulties in founding the scientific glassware industry was the financial question. The manufacture of test-tubes, for instance, had been a home industry in certain parts of Austria and Germany, carried out in small communities where families obtained their glass from the glass makers, blew the test-tubes at home and brought them back at a price many times less than we could produce them at present. Although they could not hope to establish these home industries here, yet they would like to be able to look forward to a cheapened method for making such essential commodities as test-tubes, at moderate prices. He asked what material was used for the moulds in which beakers, etc., were blown. With regard to the suggestion that the number of the sizes of flasks and beakers made should be reduced, he said that although it was very convenient to have a large range of these, it was open to discussion whether it was really necessary, and if they could help the glass industry and cheapen the products by accepting, at any rate as a temporary expedient, a few standard sizes, their work would not be seriously crippled.

Dr. F. W. RIXON asked if Dr. Travers had attempted to reproduce the effect obtained by superimposed layers of different types of glass one upon the other, also whether he considered he had obtained a soft glass tubing which was as workable before the blow-pipe as the ordinary glass obtainable in pre-war days.

Mr. W. J. A. BUTTERFIELD referred to the committee formed some years ago by the Society to deal with questions of standardisation of plant and apparatus and arbitrary methods of analysis, and expressed the hope that the question of standardisation of glassware would be referred to some committee of the Society in co-operation with the glass manufacturers. In regard to the question of thickness of glass, he said that for two or three years before the war he had been in the habit of using glass made at Ehrenfeldt, which was very much thicker than the Jena glass; it was equally good for high temperature work but was very much stronger. There was very often a thin patch in Jena glass which was apt to fall out when the vessels were used for violently bumping liquids or liquids which distilled with explosive violence; that did not happen with the Ehrenfeldt glass, but he believed the competition of the firm in question had become so severe that the Jena firm had bought it up.

Dr. BRISCOE supported strongly the suggestion that great simplification of laboratory stocks could be obtained by standardising and reducing



the number of sizes of glass apparatus. A range of some four sizes of beakers, say 250, 400, 700, and 1000 c.c., would probably cover all practical requirements, and he thought that the glass maker might take the matter into his own hands. It might also be possible to eliminate the smaller sizes of round-bottomed flasks; for all practical purposes the smaller sized flat-bottomed flasks were very much to be preferred. With regard to soft glass tubing for working with the blowpipe, he said that he had had considerable difficulty in obtaining glass which was really suitable for this purpose, most of the available supplies having a great tendency to devitrify.

The PRESIDENT referred to the great benefit that had been derived from the work of the Engineering Standards Committee in simplifying specifications for rails, girders, etc. The question of chemical standards was a matter in which their Society should take the lead, and he sincerely hoped the matter would not be lost sight of. In listening to the paper he had been struck by the fact that an industry working in such an exceedingly crude fashion should have survived. It seemed difficult to imagine that so much should be done by hand. Why should a boy be employed to blow into the end of a tube? Surely it was possible to produce a blast by mechanical means that was at least as reliable and probably cheaper. Obviously the only way of competing with domestic industries such as Dr. Keane had mentioned was to dispense with labour as much as possible and work in factories on a big scale with mechanical appliances, and he had not the least doubt that the solution of the commercial problem, at any rate, lay in that direction.

Dr. TRAVERS, in reply, said that in this country we certainly could never introduce cottage industries such as obtained in Germany and Austria, and he should very much regret to see it done. The Japanese were making test tubes extremely cheaply, and that manufacture was being developed as a cottage industry. He believed that success or failure in this industry depended upon organisation. They had had to discover the methods adopted in Germany in making this glass, and in 99 cases out of 100 it had not proved difficult. The moulds at present were being made of brass for the reason that although the material was dear, it was much cheaper to finish a mould which was cast in brass than one cast in iron, the saving in labour amounting to about 75%. Their work was blown dry, although this was not the English practice as a rule. They did not use wet methods because there was less skin effect owing to less chilling, and therefore less annealing was required. The question of the thickness of glass was entirely one of care in annealing. In reply to Dr. Rixon, he said that he had heard recently that superimposed glasses were not a success because the thicknesses of the superimposed layers had to be kept within certain narrow limits; outside those limits an explosive mixture was obtained. He was glad that the idea of standardisation had been received sympathetically; if some of the sizes now made were ignored it would be possible to work with automatic and semi-automatic machinery and reduce the price very considerably. In reply to the President, apart from automatic and semi-automatic machinery in which blowing was done entirely mechanically, pressure blowing had been used for many years in the glass industry particularly for blowing very large articles like carboys and big globes from which watchglasses were cut. In the tube shop the boy was only blowing for a few minutes; when not blowing he was engaged in picking up glass and dropping it on the ladder to prevent it chilling, so that mechanical aid

would not pay in that case. Tube-drawing machines were in use in America for drawing tubes of comparatively small size, but there were none, so far as he knew, in this country. He believed it would be difficult to develop machines for drawing tubes more than  $\frac{1}{2}$  in. diameter. In regard to soft glass tubes, he said that they had copied the German potash-soda tube, and the British product was now as good as the German. It must be a soda-potash glass containing 13% of soda and 7% of potash.

## THE SEPARATION AND USES OF CACAO SHELL.

BY A. W. KNAPP, B.Sc., F.I.C.

The regulations recently issued by the Food Controller concerning cacao shell in cocoa have brought this subject again into prominence. Since the famous legal case in 1910 when a cocoa powder containing 18% of shell was found to be genuine, it has received little attention, and now for the first time in England the amount of shell that may be present in cocoa has been carefully defined.

The quantity of shell produced every year is sufficiently great to make the subject of interest to those who have to consider the scientific use of waste products. By calculation from the official returns on cacao, the world-production of cacao shell is found to be about 36,000 tons per year, of which Europe consumes 22,000 tons. The consumption in Great Britain in 1916 was 4773 tons.

The raw cacao beans of commerce are about the size of almonds and have a thin skin or shell. This averages about 12.5% by weight; the percentage varies with the size and method of production of the beans. Thus, when the shell has been washed, as in the case of the cacao from Ceylon, it amounts to about 8%, whereas with Trinidad cacao, which is clayed, it varies around 15%.

In the cleaning of the beans a small amount (about 0.2%) of loose shell fragments is separated. The shell cannot be easily separated from the bean in the raw state, but after roasting, the shell no longer adheres to the bean. It has apparently always been the custom to remove the shell, and to use only the kernels for the preparation of cocoa or chocolate; thus Willoughby in his "Travels in Spain" (1664) writes "They first toast the berries to get off the husk," and R. Brookes in his "Natural History of Chocolate" (1730) says:—"The Indians . . . roast the kernels in earthen pots, then free them from their skins, and afterwards crush and grind them between two stones."

After roasting, both the shell and bean are crisp and brittle, and the small hard radicle, or germ, is loose. All that is necessary to get them in a suitable condition for separation is to crush the bean with as little breaking down to powder as possible, so that the shell is in large flat plates and the bean in large solid fragments (nibs). This is frequently accomplished by passing through rolls at such a distance apart that the bean is cracked without being crushed. It may also be effected by using a serrated cone revolving in a serrated conical case. It is usual to pass the broken bean into a germ separator; in these machines use is made of the uniform size and rod-like shape of the germ to effect separation. The germs so obtained naturally contain some nib and fine shell, and this mixture is known as "smalls." The large nib passes on to the husking machine, in which the nib and shell are separated by winnowing in a powerful current of air, the large nib falling through the current, whilst the shell is blown into another compartment. Both nib and shell pass down revolving cylindrical screens, encountering a larger and larger mesh as they proceed and thus

being separated into various sizes. The current of air carries about 0.2% of the material as dust into a settling chamber. The large shell contains a fair amount of nib and is graded and separated. Starting with 100 parts of raw cacao beans 10½% of shell is produced. The total "smalls" obtained approach 4%, these "smalls" containing about 36% of shell. As a result of these separations and the loss which occurs on roasting, only 78.5% of usable nib is obtained, and this contains about 2% of shell. Continuous vigilance is required to keep the product up to this standard. The husking machine and shell purifiers occupy a considerable space. Crushing and separating machines to deal with 1 ton an hour occupy 2200 cubic feet, or roughly to handle 1 lb. of roasted beans per hour requires 1 cubic foot. The space occupied by the "smalls" machine, shell graders, and purifiers would add 50%. We have never heard of any other method of separation of the shell being used.

The price of cacao shell has undergone an extraordinary increase in the last two years. Thus in 1912 the average price was 65s. per ton; in 1913, 1914, and 1915, 70s.; in 1916 it rose to 90s.; in 1917 to 128s.; whilst in May, 1918, it stood at 310s. per ton. The variation in price is even greater than appears from these figures. To appreciate fully the rise we have to deduct the bagging expenses, which are high, shell being a bulky material. Thus the above shell, which is practically free from cocoa, weighs only 9½ lb. per cubic foot (or 32 lb. when ground to powder).

There are other grades of cacao shell from which the manufacturer has not so completely separated the cocoa, and these are more highly priced, e.g.,

Grade.	Cacao nib present.	Price per ton (Sept., 1916).
1.	less than 1 %	120s.
2.	2.8 %	130s.
3.	10.0 %	150s.
4.	15.0 %	200s.

The following are the most representative analyses:—

#### *Analyses of Cacao Shell.*

Shell.	Unroasted. Average.	Roasted. Average.	Roasted. Average.	
Water ..	12.51	4.50	4.87	% 9.30
Fat ..	4.23	4.40	2.77	3.83
Ash ..	10.20	7.30	10.48	8.26
Nitrogen	2.19	2.50	2.34	3.00
Fibre ..	16.71	14.00	15.63	13.85
Analyst ..	Zipperer	Booth, Cribb, and Richards	Winton, Silverman, and Bailey	Smetham

Cacao shells have long been sold in small quantities in Ireland under the name of "miserables" for the preparation of a table decoction. But it was not till this year that they were sold under fancy names at fancy prices, as much as 2s. per lb. being paid in some cases. Whilst a water extract has, no doubt, a small food value, cacao shell should be regarded as a substance capable of producing an

interior stimulating drink rather than as one giving a food beverage. In this connection reference should be made to a recent paper by J. L. Baker and H. F. E. Hulton on "The Analysis of 'Cocoa Teas'" (Analyst, 1918, 43, 189). Cacao shell contains on an average 1% of theobromine (the figure given in most published analyses being too low), and this is probably its most valuable constituent when used to prepare a drink. Its proper use is as cattle food; for this purpose until the last six months it was low in price.

Smetham (J. Lancashire Agric. Soc., 1914) calculated the "food units" as 102, which places cacao shell above maize and meadow hay.

Mr. W. L. Dubois has sent us the following figures, obtained in America, showing the digestible nutrients in 100 lb. of shells:—Protein 1.53 lb., fibre 6.45 lb., nitrogen-free extract 40.6 lb., fat 4.91 lb., fuel value 111,079 calories (1 lb. gives 4401 B.Th.U.). These analytical results have been supported by practical feeding experiments in America, in Germany (see Zipperer), and in Turin by F. Faelli, who obtained an increase in the daily average yield of milk. J. E. Lucas (Bull. Agric. Intell., 1913) obtained 20% decrease in amount of milk and 20% increase of fat content. In 1916 it was reported that horses in Germany were poisoned by being fed on cacao shells (2½ lb. per meal). It was suggested that this was due to the theobromine present in the shell. We feel that this is doubtful, considering that cacao shells have so long been used in compound feeding cakes without complaint. It suggests however, that it is probably unwise to use a high percentage of it in a diet.\*

Early in 1915 the transport difficulties were so great that manufacturers of cocoa could not get rid of their shell and hence some thought was given to ways of using it. It has been used as fuel. Its calorific value is a little greater than that of wood (varying from 7400 to 8600 B.Th.U.), but being very light it needs careful management. It is most effectively used on a gas plant, the only objection being that the tar which it produces has a nauseating odour. The charred residue can be used as a manure. The shell itself has been used as a manure (see Annual Report of the Experimental Farms in Canada, 1898, 151, and 1899, 851). In experiments at Bournville it was found to decompose in the ground very slowly, and Mr. J. Lodge recommends that the decomposition should be hastened by placing the shell in a heap, soaking well with water, and turning several times previous to use on the land. Used in this way it gave excellent results both as a manure and as a lightener of heavy soils.

The fat in cacao shell can be extracted by solvents, and as "shell fat" is seen on the market from time to time; this is presumably a regular practice on the Continent. This solvent-extracted fat has an unpleasant taste and an odour like tobacco, which renders it unfit for edible purposes. With theobromine fetching fifty shillings a pound the extraction of the theobromine from shell appears a feasible proposition. We know of no firm actually doing this, although presumably either shell or germ is the source of the theobromine now sold. As an experiment we ourselves extracted some sixty pounds, which we had no difficulty in selling.

Shell can be made to give an extract which is equal to some of the coffee substitutes at present sold, and many other applications have been

\* See "Cacao Shells as Fodder," by A. W. Knapp, Tropical Life, 1916, 154.



suggested, but the use of shell which is the most interesting and regrettable is in cocoa and chocolate. In Belgium, Switzerland, Austria, Germany, and America, it is illegal to put shell in cocoa or chocolate, the shelled bean being used. In Great Britain until this year the amount of shell that might be present in cocoa and chocolate had not been defined. The Food Controller has now issued regulations which state that "no person shall manufacture cocoa powder except such powder as contains no more than 5% of shell." A manufacturer "may sell as Grade A. cocoa powder any cocoa powder which contains not more than 2% of cocoa bean shell." From the point of view of the public and the manufacturer the figures are well chosen, for when every reasonable effort is made on a commercial scale to separate the shell from the nib, about 2% of shell is left in. These figures have, however, placed the analyst in a difficult position, for there is no process which will accurately determine such small quantities of shell as 2% and 5%, and with such processes as are available he will need to draw conclusions from his results with considerable caution. Of the many processes that have been suggested we have most confidence in the fibre determination, but the natural variations in shell and in nib are so great as to make the detection of 5% of shell uncertain (see also Baker and Hulton, Analyst, 1918, 43, 197-201).

In conclusion I wish to thank Mr. N. P. Booth for a number of useful suggestions.

#### DISCUSSION.

Mr. D. LLOYD HOWARD asked whether the feeding of cacao bean shell to cattle affected the flavour of the milk in any way.

Mr. C. J. WATERFALL asked whether the author had tried any other mechanical process for separating the shell than that which he had described. He had tested a great many samples of nibs for cattle feeding purposes with very good results, and had never heard any suggestion of taint being produced in the milk. What method was used to estimate the theobromine?

Mr. MILNES asked whether there was any great difference in the specific gravities of the shell and the nib.

Dr. H. W. BYWATERS enquired whether the author had obtained more theobromine from the roasted shell than from the unroasted shell. One would imagine that during the roasting a good deal of the theobromine would be eliminated; if that were so it was surprising to find that so much remained in the roasted shell. He suggested that another possible use for the shell was in the manufacture of paper. At the present time it would seem to be more profitable to use cacao shell as a foodstuff than as a manure.

Mr. KNAPP, in reply, said that he knew nothing as to the effect on milk supply through the consumption of cacao shell by cattle. His own view was that it should be used as an appetiser rather than as the main portion of the diet. For the estimation of shell he had tried the levigation process of Macara, which depended on the fineness of grinding of the shell; any difference in the specific gravity in water could easily be overcome by finer grinding. He had found very slightly more theobromine in the roasted shell than in the unroasted. Theobromine was very difficult to sublime; it required a much higher temperature than was necessary when roasting cocoa. He had tried various processes for estimating theobromine and thought that the Dekker process was undoubtedly the best.

#### OCCURRENCE OF MOULD IN COCOA BUTTER.

BY LILY BATTEN AND HUBERT W. BYWATERS.

Cocoa butter is distinguished among fats by its resistance to influences tending to produce rancidity or mouldiness. A case of extensive growth of a mould in a specimen of cocoa butter is therefore interesting and noteworthy. The specimen in question was a large block of butter weighing about 28 lb., and it had probably been expressed from the cocoa beans several months before it came under our observation. On being broken, it was noted that towards the centre of the block, and extending from the upper to the lower surfaces, were a large number of dull black patches, intermingled with streaks of a brownish yellow tinge; the butter had a granular appearance, the whole somewhat resembling a matured Stilton cheese.

An accidental contamination with dirt was at first suspected, but on microscopical examination the dark material was found to consist of innumerable hyphae of various kinds, and greenish blue conidia of a fungus. It was difficult to identify the individual genera in this mass, so sterile prune-juice agar medium was prepared, poured into Petri dishes, and subsequently inoculated from various parts of the infected material. In this way colonies of the moulds were obtained and *Penicillium glaucum* and pink yeast were found to occur, but the greater part of the original mass was found to consist of a species of *Aspergillus*. A pure culture of the latter was obtained, and it is believed to be *Aspergillus oryzae*. According to Lafar (Tech. Mycol., 1910, Vol. II., Pt. II., p. 308) this species is of practical importance as a saccharifying fungus, and has been cultivated for centuries in Japan for the production of saké from rice. It grows rapidly on a large variety of liquid and solid media, and is easily cultivated even at room temperature, the optimum being above 30° C. The peculiarities of the conidiophores, sterigmata, and conidia enable the species to be distinguished with comparative ease from most others, but it is similar to *Aspergillus flavus*, except that in the latter the walls of the hyphae and conidiophores bear irregular outgrowths. The clavate or spherical globule exhibits no definite line of demarcation from the smooth stem. The sterigmata are radial, and bear numerous large spherical conidia measuring about 0.006 mm. in diameter.

On breaking open the block of infected butter, drops of clear liquid, apparently water, were observed to be present in some of the larger vesicles. Chemical examination showed the infected part to contain 0.13% of moisture, and it was thought possible that the spores of the fungi had found their way into the butter by this means. To settle this question, the following investigation of the conditions of growth of *Aspergillus* was carried out:—Cocoa butter in sterilised Petri dishes was inoculated from the infected butter, and it was found that the mould would not grow on ordinary cocoa butter at any temperature from room temperature up to 33° C., which is its melting point.

Evidence of the facilitating influence of water on the growth of the mould in the infected block was then sought by preparing a series of sterile Petri dishes, and into them placing (a) ordinary cocoa butter, (b) sterilised cocoa butter, and (c) solidified emulsion of cocoa butter and water (containing about 30% of water). These were then inoculated with the spores of the fungus. In addition, small blocks of the infected butter were placed in contact with blocks of (a) ordinary cocoa butter and (b) solidified emulsion of cocoa butter and water. The dishes were then kept at various temperatures from 15° C. up to 33° C., but at the end of three months in no case could fresh growth of the fungus be observed.

However, colonies of fungoid growth had actually developed on a large block of the solidified emulsion of cocoa butter and water which had been kept at room temperature for the same time, and on reconsideration it appeared probable that the comparatively thin films of cocoa butter and water emulsion in the Petri dishes would rapidly become dry during the incubation, and any initial growth of the fungus would soon cease owing to the lack of moisture. This view was corroborated by the fact that in a dish where the block of medium was about as thick as the depth of the dish would allow, growth of the fungus was eventually observed at a temperature of about 27° C. The water content of this butter was 2.4% which is therefore sufficient to enable the mould to grow.

In all cases growth of the mould appeared to become quiescent after a comparatively short time, although subsequent re-inoculation into a fresh plate was again followed by spasmodic growth. In order to ascertain if the arrest of growth is due to lack of water or another substance, a series of blocks of cocoa butter containing from 0–20% of water in an emulsified state were prepared, and the fungus introduced into the centre of the block, instead of being inoculated into the surface layers. Under these circumstances, however, growth continued to be slow, no matter how large the proportion of water present, but it was rather quicker at 27° C. than at either 12° C. or 17° C.

These experiments seem to indicate that although water is necessary for the growth of the mould, yet the cessation of growth is due to a lack of some other food substance. Although nitrogen could not be detected in the original block by the usual tests, it was thought possible that the addition of a nitrogenous substance might render the cocoa butter more suitable for the propagation of the fungus. Sterile Petri dishes were therefore prepared containing a medium of cocoa butter mixed with a small quantity of sterile prune-juice agar. These were inoculated, and, on incubation at 27° C., vigorous fungal growth was observed in less than a week. Fructifications were also formed in the following days, with development of characteristic conidia.

These results show that no fear need be entertained of ordinary cocoa butter becoming mouldy from the cause under investigation, provided it is kept free from water. If, however, water finds its way into the cocoa butter—and especially if the water contains substances, probably of a nitrogenous nature, which can serve as food for the fungus—then there is a real danger of the cocoa butter becoming mouldy.

Chemical investigation showed that the acid value of the mouldy butter was about 13, but this was largely due to the presence of the fungoid material, for the acid value of the butter after filtering through paper was only 3.8. This figure, although comparatively low, nevertheless indicates a certain amount of free fatty acid in the butter, and suggests the probability of the appearance of rancidity if the growth of the fungus is unchecked.

The greater part of the experimental portion of this investigation was carried out in the Department of Botany of the University of Bristol.

#### DISCUSSION.

Dr. S. JUDH LEWIS said that it might happen even with perfect storage that the brown paper in which the 28-lb. blocks were wrapped, or the sack containing a number of such blocks, might have a growth upon them. In the one or two instances in which he had seen mould growth, water had always been present. The figure given by the authors of 2.4% of moisture in cocoa butter emphasised the importance of keeping out water in the manufacture, particularly as cocoa butter was liable to contain cocoa material, which would be

sufficient to supply the nitrogen which the authors had found necessary for the development of the mould.

Mr. J. ALLAN said that there was one point in connection with the storage of fats which accentuated the difficulties arising from the presence in them even of very small quantities of water. It was well known that the fats which were solid at ordinary temperatures had a great tendency during solidification, particularly if it were prolonged, to develop a condition which was known as "seed," i.e., the mass became more or less granulated. Usually in solidification of fat under these conditions, the portion which solidified first possessed little or no structure; the granular structure, which was generally in the centre, was almost akin to crystallisation. At the same time the water in the fat tended to pass towards the centre of the mass, resulting in a higher percentage of water in that part. Consequently the mere estimation of the original water content of the fat was not a safeguard against such conditions as had been put forward in the paper. If it was intended to store fats of this kind for any length of time, care should be taken to avoid this granulated structure. It was not infrequent to find it in parcels of high-grade tallow arriving from Australia or America, even though care had been taken during cooling and packing to get them as nearly as possible homogeneous in texture, and red, green and black moulds were often found in the centre of the packages.

Mr. A. W. KNAPP asked in what manner the water was extracted from the mass. Was the water distributed in the form of an emulsion or was it distributed in fissures in the mass? He also asked what was the ash content of the fat, i.e., if any alkali had been used in its preparation.

Dr. BYWATERS, in reply, said that the particular sample of cocoa butter had been filtered after it had been prepared and pressed in the ordinary way, and as far as he could judge it was quite free from cocoa matter, so that the nitrogenous material must have arisen from some other source. Cocoa butter was a fat which presented very great difficulties in cooling, because a large amount could be kept for 12 hours after it had reached air temperature without any sign of solidification occurring. It assumed a granular appearance very readily. When the most complex of cocoa butters was broken up it was easy to see the granules which Mr. Allan had described. When a sample of cocoa butter was broken open, even though it was only a small piece of a block, fine drops of water could be seen in some of the vesicles, but a portion which was not in the granulated form contained only a trace of moisture. The ash of cocoa butter was practically nil.

Miss BATTEN, replying to a question asked by Mr. Easton, said that they had used about 10 grms. of agar to 500 c.c. of prune juice, that having been prepared from one pound of prunes.

#### CIDER APPLE JELLY.

BY B. T. P. BARKER.

After the outbreak of war in 1914 one of the chief objects on which the attention of those engaged in agricultural research was focussed was the avoidance of waste of any agricultural material of food value. The cider apple crop that season afforded a particularly good example for study, since the yield was heavy, and the price of cider apples so low that thousands of tons of fruit were wasted because the farmers did not consider it sufficiently profitable to gather. During September, 1914, the basis of the method of converting this fruit into a palatable product for human consumption, which is described



in this paper, was worked out: but the occasion for utilising it on a commercial scale did not arise until 1917. The manufacture of the jelly was organised by the Bristol University Research Station at Long Ashton, where most of the actual work was carried out.

The immediate object of the work was to utilise for human consumption the 10% or so of sugar (approximately  $\frac{1}{2}$  invert sugar and  $\frac{1}{2}$  sucrose) in the fruit. The most satisfactory plan proved to be the production of a form of apple jelly by concentration of the juice without the addition of sugar. With cider fruit fetching 20s. per ton and less, a cheap and palatable jam substitute was thus produced.

Cider apples may be divided into three main groups:—(a) sour, (b) sweet, (c) bittersweet. The sour are distinguished from the other two classes by their relatively high content of malic acid, the amount in the average case well exceeding 0.45% of acid in the juice. The acidity of sweet and bittersweet apples is normally much below that figure. The only difference of importance between the two latter classes is that the bittersweets contain appreciable quantities of tannin, which occurs only in very limited quantity in the sweet varieties. For present purposes these two classes may be treated as one, since their mode of use from a food point of view is identical.

Hitherto it has only been the sour varieties which have been drawn upon to any extent to make good any deficiency in the crop of ordinary table apples. Their utilisation presents no difficulties, the quantity of acid contained being sufficient to furnish the necessary cooking qualities for culinary purposes, and the "setting" or "jellying" properties for jam-making. Arrangements were therefore made in the West of England for the 1917 season to deal extensively with this class of fruit for the production of apple pulp for jam-making, and the major portion of the apples pulped at the respective centres organised there as well as those despatched to other stations in other parts of the country were of this type.

The other types, however, have not been utilised previously to any extent on a commercial scale for food purposes, and the work in this direction which has been carried out during the 1917 season must, therefore, be regarded as largely of an experimental character. The form of product which it was decided to make was the type of apple jelly evolved as the result of experiments carried out at Long Ashton in 1914.

*Method of making jelly.* The general method of making the jelly has already been outlined. The actual details of the process, as generally carried out, are as follows:—

The fruit to be dealt with should consist mainly of sweet or bittersweet varieties or a mixture of both types. A limited proportion of sour apples in the mixture is permissible, but it should in no case exceed one-third of the total quantity, and not more than one-sixth should be allowed if the jelly is made without added sugar. The object is to secure that the percentage of malic acid in the finished jelly shall be between 1 and 1.5%. The exact amount desirable is a matter of taste; for most palates, an amount below 1% results in a jelly too insipid and sickly sweet in flavour, while anything exceeding 1.5% is too sharp. It is obvious that the degree of acidity requisite in the unconcentrated juice must depend upon the extent to which the concentration is to be carried, *i.e.*, upon the percentage of sugar. Approximately 65% of total sugar is needed in the jelly: hence the quantity of sugar in the juice before evaporation, whether natural sugar only, or natural *plus* added sugar, determines the extent of concentration required. At Long Ashton it was the practice to test the acidity and the sugar content of every lot of juice used, adjusting the acidity to the

calculated quantity needed by the addition of sharper or less acid juices as the case might be.

Natural apple juice in an average season contains about 10% of total sugar. The juice, therefore, must be reduced to about  $\frac{1}{4}$  of its original volume, if no other sugar is added. It is found in practice more economical to add approximately the same amount of sugar as occurs naturally in the juice. The extent of concentration required is then reduced by one-half, a larger yield of jelly is obtained from a given weight of fruit, and there is a considerable saving of time. The quality of the jelly is also considered by most people to be superior. The added sugar may be either cane sugar, glucose, or corn syrup. In most cases during the past season a mixture of equal parts of cane sugar and corn syrup was used and gave very satisfactory results.

The requisite plant for jelly-making consists of a mill and press, for extraction of the juice from the fruit, and an evaporator, for the concentration of the juice.

*Mills and presses.* Ordinary cider mills and presses serve perfectly well. At Long Ashton an American type of "grater" mill and a hydraulic press, used at the Research Station for cider-making, were available. The yield of juice per ton of fruit with these machines averaged 150–170 gallons according to the period of the season.

*Evaporators.* It has been possible as a result of the season's working to obtain important information as to the most efficient type of evaporator for this particular purpose. The simplest form is the ordinary steam-jacketed jam-boiling pan. This is the least satisfactory type of apparatus, since it is not a continuous working form, and the whole of the juice dealt with in one operation has to be subjected to a high temperature until the volume is reduced to the required point. This involves a relatively long period of cooking with consequent partial caramelisation of sugar and, therefore, considerable darkening of colour and the acquisition of a more or less distinct burnt flavour. The "setting" property of the jelly is to some extent affected. The process is also comparatively slow. The usual type of steam-heated vacuum boiling pan is an improvement, but is also open to the objection of not being continuous in its working. Caramelisation is, however, considerably reduced, and colour and flavour accordingly are improved. The speed of evaporation is increased, but the output does not reach that of a continuous working apparatus.

Another type of evaporator used was one constructed on the lines of certain American juice evaporators. It consists of a long narrow wooden trough divided into two compartments by a partition. The two compartments are entirely separated from each other by this at one end, the fresh juice being fed into the one division at this end and the outlet for the concentrated juice being situated at the corresponding point in the other compartment. The two compartments communicate at the far end, where the partition does not extend quite the full length of the trough. The bottom of the trough is made slightly sloping so that the incoming juice flows along the first compartment to its point of communication with the second, and thence returns along the latter to the outlet. Three copper steam pipes are fixed just above the floor of the trough in each compartment, and these are heated by steam at 80 lb. pressure. By regulation of the rate of feed of juice the desired degree of concentration is obtained. A wooden cover is fitted over the whole trough and a central flue to carry off the evolved steam from the boiling juice is provided. This evaporator has the advantage of being continuous in its working, but proved to be difficult to regulate. Variations in steam pressure affected the rate of flow to such an extent that constant attention was required, and a number of other causes contributed to produce such serious



irregularities in the flow that occasionally the steam pipes near the outlet would become partially uncovered, leading to burning of the concentrated juice, while at other times juice incompletely evaporated would be delivered. It was with great difficulty, therefore, that anything approaching uniformity in the product was obtained. The speed of the apparatus proved to be much slower than had been anticipated, and the cost of concentration was accordingly relatively high. The quality of the product was, on the whole, fair; the colour was generally rather too dark and the flavour at times indicated traces of burning. The jelly as a rule set moderately well, but showed a tendency to be hygroscopic, and during damp periods in December liquefied somewhat. Under drier conditions this is now setting again. The principal drawbacks of the apparatus are the slow rate of output, the high cost for concentration, and the difficulty of securing uniform working.

The apparatus mainly used at Long Ashton was the Kestner evaporator. In essentials, it consists of a continuous series of copper tubes, on the interior surface of which the juice travels as a thin film. The tubes are heated externally by steam under pressure, and the juice in its passage through them is gradually concentrated. The most suitable pressure found during the past season's working with apple juice was about 30 lb. A special arrangement at the delivery end of the tube system provides for the separation of the evolved steam from the concentrated liquid. The machine is a continuous working one. It can be operated with the treated liquid under ordinary or reduced pressure. No vacuum pump was provided for the first season's trials at Long Ashton, and consequently the whole of the work was done under ordinary pressure: but there is no doubt that the reduced pressure would give decidedly superior results as regards quality of product, rate of output, and cost of evaporation. For future work in this direction it is likely to be adopted. Another improvement which should in future be incorporated is to pre-heat the juice before it enters the evaporator by means of the steam given off by the juice.

*Condition of juice.* Experience in the making of cider has shown that the yield of juice is determined by the condition of ripeness of the fruit at the time it is milled and pressed. Both unripe and over-ripe fruit—particularly the latter—yield less juice from a given weight of apples than fruit which has reached a well-ripened condition. The "setting" quality of the juice is similarly much affected by the state of ripeness of the fruit. The juice from unripe or much over-ripe fruit sets less well after concentration than that from perfectly ripe apples. Up to a point a certain amount of over-ripeness may assist rather than hinder "setting," and it is probable that the fruit should be left over to a somewhat more advanced state of maturity than is desirable from the point of view of yield of juice. If the maximum "setting" quality is aimed at. It is preferable, therefore, that the fruit should be thoroughly well matured before extraction of the juice, even if a portion is distinctly over-ripe, rather than it should be milled unripe, unless steps are taken to ensure good setting by the use of pectin extract, concerning which information is given below.

*Extraction of juice.* There are no points of particular importance in connection with the extraction of the juice which require notice. The methods found most satisfactory in cider-making apply equally in this case.

*Supplies of juice.* In preparing quantities of juice for evaporation it is desirable to provide at one time no more than is required for one day's working of the evaporator. Juice more than 24 hours old is liable to show the beginnings of fer-

mentation, particularly during the warmer parts of the season, and, when this occurs, its "setting" qualities suffer owing to changes in its pectin constituents. An attempt was made to overcome this difficulty by treating the juice with sufficient sulphur dioxide to arrest fermentation for a few days: but, although the juice can be retained fit for jelly-making in this way even for so long a period as a fortnight or more, the jelly does not set readily and the method cannot be recommended except for emergencies caused by an over-supply of juice. At the same time it is desirable to provide at the start of each day a sufficient bulk of juice to last throughout the day. If this is pumped into one large vat, it ensures that the whole of the day's output is uniform in character. At Long Ashton two blending vats, holding about 750 gallons each, were used, the contents of the two sufficing to keep the evaporator running for a working day of about 15 hours.

*Standardising acidity of juice.* The acidity of the supply of juice provided is brought to the required standard by blending as already described. It has been shown that this standard is determined by the degree to which concentration is to be carried, i.e., according to whether sugar is or is not added. As mentioned in the beginning of this paper, the method found most advantageous is to raise the content of total sugar in the unconcentrated juice to approximately 20% by the addition of cane sugar or corn syrup or a mixture of both, in which case the standard of acidity is taken as about 0.35% of malic acid and concentration is carried to about  $\frac{1}{4}$  of the original volume.

*Use of sugar.* When supplies of sugar in some form are available, its use is advised for the reasons already given. If cane sugar is exclusively used, the flavour of the jelly is sweeter and its consistency generally firmer. Corn syrup alone, consisting of a mixture of glucose, maltose, and dextrin, yields a less sweet article, and there is a tendency for the jelly to be more sticky or treacly. A mixture of cane sugar and corn syrup in equal parts gives results little, if any, inferior to cane sugar alone.

Jelly made without added sugar is more intense in flavour, its colour is deeper, and particular care to avoid caramelisation during the making is necessary. It is, however, possible to make a palatable article without added sugar; but the yield of jelly from a given weight of fruit is considerably less, and when the price of apples is about £3 per ton or upwards the cost of the jelly works out at a slightly higher rate than when sugar is added. The latter point simply resolves itself into a question of relative prices of apples and sugar.

It is better to add the sugar before than after concentration of the juice, since, although more steam is required for concentration, it is not easy to dissolve the sugar and obtain an even mixture in the thick, syrupy, concentrated liquid.

*Use of pectin extract.* The investigations on pectin compounds which have been conducted at Long Ashton during the past four years have shown that the following three conditions are necessary for a proper "gel" formation, viz., (a) the content of total sugar present in solution—whether cane sugar, glucose, or some other suitable carbohydrate, either singly or in mixture—must approach 60–65%; (b) an acid of some kind, preferably malic, citric, or tartaric acid, must be present in quantity approximating at least to the equivalent of 1% malic acid, and (c) a suitable form of pectin must also be present in quantities of 0.5% or upwards. In many samples of apple juice dealt with in the course of this work, with the acidity raised to the standard adopted as being most suitable for jelly-making, these three essential conditions were easily obtained in the finished product when the fruit handled was in reasonably uniform condition as regards ripeness and was milled when well ripened.



Even when the fruit was somewhat over-ripe no serious trouble was experienced; but badly over-ripe fruit as well as unripe or unevenly ripe samples caused uncertain results, mainly on account of pectin deficiencies. Since in practice the condition of the fruit generally is very variable, it was considered advisable during the greater part of the season to add a limited quantity of a pectin extract to the juice before evaporation.

The extract was made by passing waste steam from the evaporator into a mass of pressed apple pomace, plentiful supplies of this being available. After being steamed for some time, the mass was placed under the press and as much liquid as possible extracted. This fluid, which was thick and mucilaginous and contained considerable quantities of pectin, was added to the juice at the rate of 15 gallons of extract to 100 gallons of juice. When this method was adopted, the jelly could be relied upon to set well, provided that the juice was not over-cooked in the evaporator. With the extract an additional quantity of sugar was used (15 lb. of sugar to 15 gallons of extract) to avoid dilution of the juice in respect of that constituent.

The use of a crude pectin extract prepared in the manner described is mainly for the purpose of ensuring the proper "setting" of the jelly; but it also affects the yield from a given weight of fruit. Practically it amounts to the addition of 15% to the volume of the fresh juice handled with a corresponding gain in the final amount of jelly. It occasionally results in a still greater increase in the yield of jelly, since some juices without it have to be concentrated to a smaller bulk than that normally requisite in order to give a sufficiently firm "set."

Some preliminary experiments with an improved form of pectin preparation suggest that an appreciable reduction in the cost of production should be possible, provided that the value of sugar as compared with that of the fruit does not vary materially from the ratio obtaining during the 1917 season. The preparation in question also promises to simplify the problem of making the jelly set and to yield a superior product.

The addition of the pectin extract does not reduce the strength of the flavour to an appreciable extent. The acidity is slightly reduced, but this can be adjusted by modifying the standard of acidity in the unconcentrated juice by suitable blending.

*Clearing of juice.* The fresh juice as it is received from the press is a turbid liquid containing variable amounts of particles of apple pomace in suspension. The removal of the coarser particles is essential and is readily accomplished by straining the juice, as it is pumped into the blending tank, through a filter bag made of canvas or other suitable material of moderately fine mesh. By that means the juice, while still turbid, is freed fairly completely from suspended solid matter, the turbidity being mainly caused by pectin compounds in solution. A further filtration of the juice is arranged as it is fed to the evaporator, the end of the suction hose of the supply pump being covered with fine-meshed straining cloth.

The turbidity of the juice disappears almost entirely in the final stages of its concentration in the evaporator. This is believed to be due to the interaction of the sugar, acid, and pectin contents, and the comparatively sudden change from opacity to relative transparency which occurs apparently at or about a given point of concentration is regarded as indicating that the "jelling" stage has been reached. In practice it constitutes a safe guide.

The jelly prepared in this way without more elaborate attempts at clearing is semi-transparent when cold, a certain amount of haziness developing as it cools.

*Concentration of juice.* A supply pump provides a continuous flow of juice into the evaporator, the rate of which can be regulated as required. This is determined by the specific gravity of the concentrated juice. It is considered necessary that the jelly should possess a total sugar content of 60–65% to ensure adequate keeping and setting properties. This is equivalent to a specific gravity of 1.35 at 15° C., which corresponds to about 1.3 at the temperature of the concentrated liquor as it is collected from the evaporator (about 95° C.). The whole rate of working of the apparatus is therefore controlled by the specific gravity of the concentrated juice. This is taken at frequent intervals with a hydrometer, the standard gravity adopted being 1.3 for the hot liquor. When the gravity reading is too low the rate of supply of juice to the evaporator is slowed down, and when it is too high the supply is increased. Another means of controlling the rate of concentration is to regulate the steam pressure of the evaporator. By the combination of this method with that of regulation of the supply of juice, it is not difficult with the Kestner apparatus to maintain the specific gravity of the concentrated liquor at any point required. Control is similarly obtained with the American type of evaporator, but exact regulation is more difficult, and closer and continuous attention is needed in this case.

*Output.* As regards output the Kestner evaporator at Long Ashton was capable of dealing with 100 gallons of the prepared juice per hour under fair average conditions of working, giving an average production of about 275 lb. of jelly per hour. Under specially favourable conditions the quantity of juice per hour used advanced occasionally to 110–120 gallons. These figures refer to cold juice only. In a trial where the juice was preheated to 75° C. before entering the evaporator, the juice supply per hour easily reached 125 gallons. Probably an appreciable increase in these figures would have been recorded had a larger steam boiler been available. That used had to be run at full capacity the whole time in order to maintain the steam pressure in the evaporator near 30 lb. per sq. inch.

## ANALYSIS OF COMMERCIAL SACCHARIN. THE ESTIMATION OF O-BENZOYLSULPHONIMIDE FROM THE AMMONIA PRODUCED BY ACID HYDROLYSIS.

BY H. DROOP RICHMOND, F.I.C., AND  
CHARLES ALFRED HILL.

The present investigation was undertaken with the two-fold motive of finding a more rapid method of estimating o-benzoylsulphonimide and of eliminating from the known methods those factors giving rise to a variation of results obtained by different workers.

The principal known methods take from 3½ to 4 hours to complete, and in a factory engaged upon the manufacture the reduction of this time is a matter of considerable importance, while we have found variations as great as 4% by the same method due to personal factors.

The methods commonly employed are applications of the principle established by Remsen and Burton (Amer. Chem. J., 11, 403) and Hefelmann (Pharm. Centr. Blatt., 85, 105) that saccharin is hydrolysed by weak acids to o-sulphonaminobenzoic acid and ammonium sulphonbenzoate and depend upon the determination of the ammonia; the former employed 1.25N hydrochloric acid and the latter 70% sulphuric acid as the hydrolytic agent. Reid (Amer. Chem. J., 1899, 461) studied both these methods in detail, and finally gave preference to the use of hydrochloric acid, which he found under the conditions he prescribed to convert saccharin completely into the ammonium salt of

*o*-sulphobenzole acid, while *p*-sulphonaminobenzole acid and *o*-toluenesulphonamide, the two impurities most commonly present in commercial saccharin, were unaffected. His directions were:—Boil 0.65 gm. of saccharin with 50 c.c. of 1.2N hydrochloric acid for 2 hours in a small flask fitted with an air condenser, evaporate to 10 c.c., and distil off the ammonia into standard acid (minor details here omitted). This method was tried by Reid on pure saccharin and mixtures of known composition, and found to give excellent results, but probably the fact that the final evaporation is a little troublesome has prevented its general adoption. Proctor (*Chem. Soc. Trans.*, 1905, 242) simplified Reid's method by omitting the final evaporation, boiling for 2½ hours, and reducing the strength of the acid to approximately normal; unfortunately he does not appear to have tested this modification against mixtures of known composition, and, as we shall show, the reduction of the strength of acid, and the want of more exact definition of conditions is responsible to a considerable extent for the discrepancies between analysts.

Proctor's modification was so obviously convenient that it has been largely adopted, but we have found that his use of the words "approximately normal" in the description of the method has been interpreted by different analysts who have given us their detailed working directions as a strength varying from 1.2N to 0.7N.

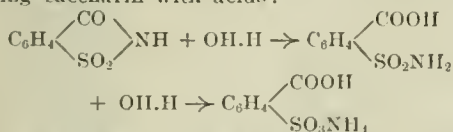
Testoni (*Z. Unters. Nahr. Genussm.*, 1909, 18, 577) heated the saccharin with 5N acid for ½ hour in an autoclave, a method which lacks convenience.

Hefelmann's method of heating with 70% sulphuric acid in a water bath for from 2 to 4 hours has been adopted by the B.P., but only as a qualitative test, and was found by Reid to give very fair results. A modification of this worked out in these laboratories consists in boiling 0.6 gm. saccharin with 10 c.c. of 70% sulphuric acid for a short period (not exceeding one minute), pouring at once into a considerable quantity of cold water, and distilling off the ammonia. This method has the advantage of great rapidity, but at the same time, unless the conditions are narrowly defined and adhered to, is liable to give high results with impure saccharin, and is consequently, as we have found, unreliable except in skilled hands.

All the above methods depend on the comparatively rapid hydrolysis of saccharin by acids to an ammonium salt, and the estimation of the ammonia, and do not discriminate between *o*-benzoylsulphonimide and *o*-sulphonaminobenzole acid, which owing to its considerable solubility in water is a possible but not likely impurity in appreciable amount.

Testoni (*loc. cit.*) has devised a method depending upon the insolubility of the silver salt in alcohol, Proctor (*loc. cit.*) discussed the conversion into salicylic acid by fusion with alkali, and there are numerous methods which depend on the estimation of the sulphur, but these methods are of little use in the examination of commercial saccharin. Gnadinger (*J.A.O.A.C.*, 1917, III, 1, 25) has studied the solubility of saccharin in various solvents.

According to work of Remsen and Burton (*loc. cit.*), the following reactions take place on hydrolysing saccharin with acids:—



In the presence of a large and constant excess of hydrogen ions, both reactions will be approximately unimolecular, and the results should approximately agree with a formula of the type

$$1-x = \frac{K_2}{K_2 - K_1} e^{-K_1 t} - \frac{K_1}{K_2 - K_1} e^{-K_2 t}$$

where  $x$  is amount of saccharin converted into ammonia; total=1; the minus sign comes from the fact that the first stage is a conversion of saccharin into the ortho acid, while the results are expressed in terms of the formation of ammonia from the ortho acid.

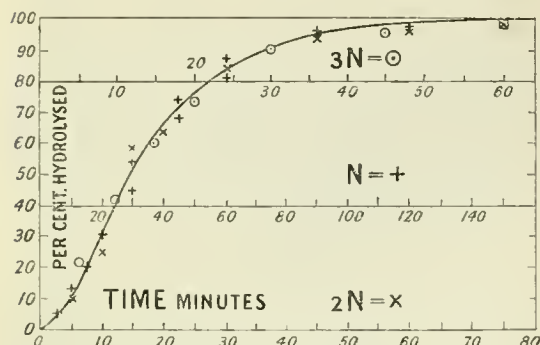
It is evident that Reid's method, involving a change of conditions during the experiment, would not lend itself to a study of the mechanism of the reaction, and it was found that in Hefelmann's method the solution of the saccharin was slow and the time, therefore, could not be accurately determined, but Proctor's modification appeared to be carried out under fairly constant conditions, and our first experiments were a study of the ammonia produced in different times of heating. Our procedure was as follows:—

0.6104 gm. of saccharin was placed in a flask, and 50 c.c. of *N*/1 hydrochloric acid, just raised to the boiling point, added; the saccharin was dissolved in a very short time, and the solution boiled under a reflux condenser for the stated period; the time was, of course, not accurately known, though the error was small; the temperature was not quite constant, as we frequently had evidence of superheating, followed by explosive ebullition, and the concentration would vary slightly in different parts of the solution, but it was hoped that the conditions would be sufficiently constant to enable us to verify the equation given above. The saccharin was prepared from a fairly pure commercial saccharin, by dissolving in slightly less than the calculated quantity of sodium bicarbonate, adding acid equal to 10%, allowing this to crystallise out, and then precipitating about 80% of the remainder; this treatment was repeated, and the resulting product crystallised from acetone (*cf.* Pope, *J. Chem. Soc.*, 1895, 985). It had a melting point of 229° C. The *p*-sulphonaminobenzole acid was twice recrystallised from hot water, the melting point of the product of the first and second crystallisations being 288° C. The *o*-toluenesulphonamide was also carefully purified and had a melting point of 153.2° C. Unfortunately we found that duplicates gave values differing by a considerable amount, and we have therefore only calculated approximately the constants in the equation as  $K_1 = 0.0414$  and  $K_2 = 0.0828$ , and the agreement between those and our experimental results is sufficiently good to show that the mechanism of the reaction is probably that stated above. Two series were carried out with *N*/1 acid, one with pure saccharin, and the other with a commercial article of which the probable content of saccharin was 97.2%; the 2N and 3N series were carried out with good commercial products. From the results with 2N acid it is evident that if the time is multiplied by 2.0 the results agree closely with those obtained by the use of *N*/1 acid, showing that the same law is followed, while with 3N acid the agreement is on the whole good if the times are multiplied by 2.5. A series in which saccharin was hydrolysed by boiling with water at a strength of *N*/15 took approximately 12 times as long to reach the same figures in the early stages as the *N*/1 series.

Time	100% product.	<i>N</i> /1 HCl		2N HCl		3N HCl	
		97.2% product.	Calc.	Calc.	Calc.	Calc.	Calc.
5 mins.	—	4.7	3.5	10.4	11.5	21.9	16.3
10 "	—	13.0	11.5	25.5	31.8	42.4	41.6
15 "	20.2	—	21.4	58.8	50.6	60.1	62.4
20 "	—	30.6	31.8	63.8	65.5	73.8	76.4
30 "	53.9	44.8, 54.0	50.6	84.4	84.1	90.5	91.3
45 "	68.2	73.4	71.3	94.0	95.4	95.3	98.1
60 "	87.3	81.7	84.1	96.3	96.6	98.8	99.6
90 "	95.1	95.0	95.4	98.7	99.3	100.2	100.0
120 "	96.8	97.4	99.6	100.0	100.0		
150 "	98.2, 99.2, 99.2	99.3	99.6	100.0	100.0		
240 "	100.2	99.7	100.0	100.0	100.0		
480 "	99.7	—	100.0				



The rate of hydrolysis is fairly proportional to the strength of acid, though slightly higher with weak acids, and lower with higher strengths. This is shown graphically in the accompanying curve, plotted from the figures in the above table.



It would appear, therefore, that the use of Proctor's method, adhering strictly to normal acid, will give results from 0.5 to 1.0% low; if the wording "approximately normal" is taken to mean 1.2N they will be nearly correct, while if the strength is reduced to 0.7N and the solution boiled for three hours, they will be about 2% low. Further, owing to slight but unavoidable variations of temperature, concentration, and time of effective heating, any method which does not give approximately complete hydrolysis is liable to a fairly large experimental error, and the following are the minimum times of heating which can be depended upon to hydrolyse saccharin with practical completeness.

Strength of acid.	Time in hours.
0.7N ... ..	5
1.0N ... ..	4
1.2N ... ..	3.3
1.5N ... ..	2.5
2.0N ... ..	2
3.0N ... ..	1.5

We have made some experiments on the influence of temperature by hydrolysing saccharin for 2 hours on days when the barometer varied, and these indicated that the reaction follows the usual law of doubling for a 10° C. rise.

It is seen that the rapidity of the method can be increased by increasing the strength of acid; it is evident that as the reaction proceeds in two stages, the first one of which is the slower, any method of increasing this would add to the total speed. It is known that alkali hydrolyses saccharin very rapidly to *o*-sulphonaminobenzoic acid, the action stopping at this stage; our experiments showed that on adding 10 c.c. of 7.5N sodium hydroxide to 0.6104 grm. of saccharin, raising to boiling, cooling, and making neutral, the sweet taste had disappeared, indicating that the first stage of the reaction was over, and we also found that no ammonia was given off on continuing the boiling for 10 minutes. We therefore made a series of experiments by adding 10 c.c. of 7.5N sodium hydroxide to 0.6104 grm. of saccharin, raising to the boil and boiling for 2 minutes, cooling somewhat, adding 15 c.c. of 10N hydrochloric acid (B.P. strength), and boiling under a reflux condenser for various times. As there is only one reaction taking place, the formula for unimolecular reactions should express the results. A commercial saccharin was used in this series. The following were the results, together with those calculated by the formula  $1-x = e^{-0.1612t}$ :

Time.	% Hydrolysed.	Calc.
5 minutes ...	55.2	55.3
10 .. ...	81.8	80.1
15 .. ...	93.3	91.1
30 .. ...	97.2	99.1
45 .. ...	99.9	99.9
60 .. ...	99.5, 99.7	100.0
120 .. ...	100.2	100.0
240 .. ...	100.4	100.0

The calculated results, which agree moderately well, show that approximately 16% per minute is converted by acid hydrolysis.

We have adopted as working conditions 2 minutes' boiling with 10 c.c. of 7.5N sodium hydroxide, taking care not to concentrate appreciably, followed by 50 minutes' boiling after the addition of 15 c.c. of 10N hydrochloric acid under any form of reflux which condenses efficiently without retaining an appreciable amount of liquid, adding a little powdered pumice if desired to promote steady boiling; sodium chloride should not be deposited whilst the liquid is boiling. No correction for variation of temperature due to the usual changes of barometric pressure (740—775 mm.) is necessary. After the heating is completed the liquid is cooled, 75 c.c. of cold water added, and a current of air passed through the upper part of the flask to remove any acid vapour; 15 c.c. of 7.5N sodium hydroxide is added carefully, and the flask at once connected with a distillation apparatus provided with an efficient steam trap, a condenser, and an absorption flask in which is placed 20 c.c. of *N*/5 hydrochloric acid. The distillation is started with a small flame to obviate a considerable rush of air through the standard acid, which would be liable to lead to incomplete absorption of the ammonia; after the air has ceased to bubble through the acid the flame is raised and about 70 c.c. is distilled. The solution is titrated with *N*/10 alkali, using methyl red as indicator, and corrected for a blank test of the reagents. Other indicators may be used, but we prefer methyl red as being the most delicate. No correction is needed for the small amounts of ammonia present in commercial saccharin (not usually exceeding 0.01%), as this is practically all removed by the alkaline treatment. The strengths of the acid and alkali used should not vary more than 2% at most from those given above. With 0.6104 grm. of saccharin the number of c.c. of *N*/10 acid neutralised multiplied by 3 = percentage of saccharin.

The following test experiments were made with mixtures of carefully purified saccharin, *p*-sulphonaminobenzoic acid, and *o*-toluenesulphonamide.

Saccharin.	Calculated composition.			Saccharin found, %.
	<i>p</i> -Compound.	Amide.		
100 ...	0	0		99.9, 100.0
95 ...	5	0		95.0
95 ...	4	1		95.0
94.5 ...	5.5	0		94.7
90 ...	5	5		90.2
90 ...	10	0		90.9
80 ...	20	0		80.6
80 ...	19	1		80.0
70 ...	29	1		70.1
60 ...	38	2		60.0
60 ...	40	0		59.9
40 ...	50	10		40.1
10 ...	90	0		10.2
10 ...	45	45		10.2
0 ...	100	0		0.0, 0.7, 0.5
0 ...	0	100		0.9, 1.7, 1.7

The Proctor method (0.6104 grm. heated for 2½ hours with 50 c.c. *N*/1 acid) was also tried on mixtures.

Calculated composition.			Saccharin found, %.
Saccharin.	p-Compound.	Amide.	
100	...	0	99.2, 99.2
100	...	0	98.9
94.5	...	5.5	93.8
90	...	10	88.6, 89.6
88.8	...	11.2	87.3, 87.8
60	...	39	59.7
0	...	100	0.4, 0.1, 0.1
0	...	0	0.4, 0.4

It is seen that while our alkaline-acid method gives good results, averaging about 0.1% high, Proctor's method averages 0.8% low. In both methods, especially in the alkaline-acid process, there is a slight hydrolysis of the para acid and the amide, the amide hydrolysing the more easily, but the amount hydrolysed is negligible in commercial saccharins.

The following table shows the results with the sulphuric acid methods; the Hefelmann method was carried out by heating 0.6104 gm. with 10 c.c. 70.5% sulphuric acid in a tube immersed in a briskly boiling water bath for 3 hours, stirring vigorously from time to time, and the  $\frac{1}{2}$ -minute method by boiling 0.6104 gm. with 10 c.c. of 70.5% sulphuric acid, heating to boiling in from 1 to 14 minutes and boiling for  $\frac{1}{2}$  minute, and then pouring at once into 75 c.c. of cold water. The temperature in the Hefelmann method is probably a degree or two below 100°, and that of boiling 70.5% sulphuric acid about 160° C., but both are liable to slight variation.

% Saccharin.		p-Compound.		Amide.		Saccharin found.	
						Hefelmann	$\frac{1}{2}$ -minute.
99.5	—	—	—	—	—	99.5, 98.5	99.4
94.5	—	—	—	—	—	94.3, 93.8	94.4, 92.6, 94.4
60.0	39	—	—	1	—	59.6	66.5
0	100	—	—	0	—	2.0	7.4
0	0	—	—	100	—	9.8	37.5, 38.4

A number of comparisons between the Proctor and  $\frac{1}{2}$ -minute methods showed that the results with the latter were about 1% higher than with Proctor's method on saccharins containing 90% and upwards of *o*-benzoylsulphonamide, and quite low in amide.

These results indicate that the sulphuric acid methods are of approximate accuracy only, and we are in substantial agreement with the criticisms of Reid (*loc. cit.*) on Hefelmann's method; the  $\frac{1}{2}$ -minute method has a certain value as a works method, but as we have found that 2–3 minutes' boiling hydrolyses the para acid and the amide practically completely, it requires to be used with great caution.

We are indebted to Miss Florence M. Richards for a number of comparisons between our alkaline-acid method and the Proctor method, illustrating the lower results by the latter; the average difference was 0.6%.

#### Conclusions.

1. Reid's hydrochloric acid method is accurate, but rather troublesome.

2. Proctor's modification tends to low results; it is insufficiently described and leads to discrepancies between analysts. By increasing the time to 4 hours or the strength of acid to 1.5N it becomes reliable.

3. Our alkaline-acid hydrolysis method is rapid, convenient, and accurate, and as it eliminates the ammonia correction saves working time.

4. The sulphuric acid methods are not so good as the hydrochloric acid hydrolysis; the  $\frac{1}{2}$ -minute method is a very rapid approximate method, but requires to be used with caution.

This work was done in the analytical laboratory of Messrs. Boots Pure Drug Co., Ltd., to whom we wish to express our thanks.

## COLLOIDAL ELECTROLYTES: SOAP SOLUTIONS AS A TYPE.

BY JAMES WILLIAM McBAIN.

Soaps are substances of definitely known and simple composition, but their mixtures with water exhibit a remarkable variety of pronounced characteristics. They resemble in many of their properties a very large group of substances of great industrial importance, such as the dyestuffs, gelatin, and protein salts; these, however, are mostly of less simple chemical constitution.

During the six years preceding the outbreak of war, extensive and accurate measurements were carried out in this laboratory with regard to some of the fundamental properties of soap solutions.\* These have led to a comprehensive theory of solutions of such substances which we define as colloidal electrolytes. To this group we expect many inorganic substances will belong, in addition to most organic materials containing eight or more carbon atoms which are capable of splitting off an ion of any kind, or even of taking up one of the ions of an added electrolyte. Even in non-aqueous solutions similar phenomena appear to be of frequent occurrence, and one such case has been studied in order to show how the otherwise typically anomalous results are explained.

The hypothesis to which we have been forced is that all these solutions are salts in which one ion is replaced by an ionic micelle which exhibits high conductivity and great solvation, and which carries a large number of electrical charges. Extensive changes result from alteration in the concentration, so that on the one hand, great dilution may result in the nearly complete formation of simple electrolyte, and, on the other, high concentration or the addition of other substances may lead to the quantitative formation of neutral colloid.

In previous communications it has been shown how the conception of such an ionic micelle of great valency and hydration explains the really high conductivity and great mechanical viscosity at the same time, the proof being based on a simple application of the underlying principle of Stokes' law. The experimental data have further fully established the important theoretical result that colloids, colloidal electrolytes, and electrolytes may participate in true perfectly reversible and reproducible equilibria. This is the case in all soap solutions in which the conditions are defined.

#### Summary of the constitution of soap solutions.

Commencing with the case of very dilute solutions, the soaps are partially hydrolysed according to the following equation:—



The acid soap is in suspension, either coarse or colloidal as the case may be, and it consists of something between the neutral salt NaR and the acid soap NaHR<sub>2</sub>, where R is the radical of the fatty acid. Only excessively minute amounts of free fatty acid can exist as such in any soap solution, even when a whole equivalent excess of fatty acid has been added, so that the equation just given must supersede the ordinary statement of textbooks and other publications. The data which establish this are based upon analysis, conductivity, measurements of electromotive force, and measurements of the free hydroxyl ion by the rate of catalysis of nitrosotriacetoneamine (see Francis, p. 252 r).

\* McBain and Taylor, Ber., 1910, 43, 321; Z. physik. Chem., 1911, 76, 179; Bowden, Chem. Soc. Trans., 1911, 99, 131; McBain, Cornish, Bowden, *ibid.*, 1912, 101, 242; Burbury and Martin, *ibid.*, 1914, 105, 417; McBain and Martin, *ibid.*, 1914, 105, 957; Laing, *ibid.*, 1918, 113, 435; McBain and Bolam, *ibid.*, 1918; McBain, Laing and Titley, *ibid.*, 1918; McBain, Trans. Faraday Soc., 1913, 9, 99; McBain and Salmon, J. Amer. Chem. Soc., 1918.



In decinormal solution or somewhat less, soaps are chiefly composed of simple electrolyte in true solution, with simple ions. Hydrolysis is still present, but only in a very minor degree, since the hydroxyl ion concentration is shown by the two methods just cited to be only about  $N/1000$ . As the concentration increases, the fatty ions rapidly coalesce to form the ionic micelles described above, until in  $N/2$  or  $N/1$  solution the colloidal electrolyte comprises all the soap, hydrolysis being still more insignificant. As the concentration increases the ionic micelle becomes less solvated and conducts even better. The only crystalloidal constituent left is the potassium or sodium ion, as

out the soap in the form of a curd containing some combined water and other sorbed material. An ordinary analysis of such a curd gives but little clue to the composition of the solid part of it, since it is a heterogeneous sponge or felt full of mechanically enclosed mother liquor.

Surprising as it may appear, no data have been published which give information as to how much, if any, of the water and the salts contained in a bar of soap is actually combined in the solid part of the curd, and how much is merely enmeshed. We have established these quantities for several definite cases, and it will be comparatively simple now to extend measurements to all soap curds. This

TABLE I.  
*Equivalent conductivity of potassium soaps at 90° C.*

Concentration ..	1.0	0.75	0.5	0.2	0.1	0.05	0.02	0.01
Stearate, $C_{18}$ .. ..	113.4	112.6	113.9	100.0	96.0	101.7	124.9	147.7
Palmitate, $C_{16}$ .. ..	124.2	127.9	127.0	111.0	107.0	110.8	133.2	171.6
Myristate, $C_{14}$ .. ..	136.2	—	135.4	130.8	121.8	136.6	181.6	224.3
Laurate, $C_{12}$ .. ..	143.2	142.6	146.0	144.2	159.7	195.6	—	233.0
Decoate, $C_{10}$ .. ..	145.9	—	156.3	180.9	200.6	211.9	—	232.4
Octoate, $C_8$ .. ..	148.7	—	168.5	191.0	205.2	219.2	—	239.5
Hexoate, $C_6$ .. ..	149.5	—	177.7	201.2	216.5	227.7	—	245.9
Acetate, $C_2$ .. ..	176.9	183.9	196.6	221.2	236.5	249.5	262.6	270.4

TABLE II.  
*Equivalent conductivities of sodium soaps at 90° C.*

Concentration ..	1.5	1.0	0.75	0.50	0.35	0.20	0.10	0.05	0.01
Stearate .. ..	81.5	88.3	—	76.1	—	77.4	76.0	78.0	125.9
Palmitate .. ..	84.5	84.66	87.48	89.48	87.04	82.38	82.51	88.61	137.7
Myristate .. ..	84.8	94.9	97.6	99.2	—	95.2	96.5	110.4	191.7
Laurate .. ..	96.2	104.2	—	109.5	—	113.4	125.5	152.0	193.9
Acetate .. ..	—	129.7	—	—	—	178.9	195.0	—	228.2

TABLE III.  
*Equivalent conductivities of soap solutions at 18° C.*

Concentration ..	2.0	1.0	0.6	0.4	0.2	0.1	0.05	0.01
K Laurate, $C_{12}$ .. ..	43.1	47.1	46.2	44.2	41.8	44.0	68.7	75.4
K Caprylate, $C_8$ .. ..	42.2	48.6	51.9	56.4	63.1	69.5	—	—
K Oleate, $C_{18}$ (unsaturated)	—	—	37.3	36.7	33.3	29.7	29.6	51.9
Na Oleate, $C_{18}$ (unsaturated)	—	—	21.7	20.8	19.8	20.5	20.6	30.1

even the undissociated soap is now entirely in the form of colloid. Alcoholic solutions differ entirely in their behaviour, as the soap is a simple unhydrolysed electrolyte throughout.

#### *The true composition of soap curd.*

With increasing concentration the dissociation of the colloidal electrolyte decreases, and the composition of the micelle (which being colloidal sorbs, *i.e.*, adsorbs and absorbs, most of the available materials, including probably the neutral colloid) approaches that of neutral colloidal soap. Mere increase of concentration probably never makes this process complete, but it is rapidly effected by the addition of salts of any sort, which in sufficient concentration quantitatively salt

is well known to be an important practical problem for the manufacturer. In the case of sodium palmitate, for example, salted out at 90° C. by a saturated solution of common salt, the combined water amounts to  $NaP + 2.1H_2O$ , which corresponds to 88.0% soap. There is a good deal of enmeshed water even in the best household soaps. The quantitative data will be communicated later.

#### *The hydrolysis of soap solutions.*

All soap solutions contain free hydroxyl ion. The only measurements of this so far published are contained in the work of McBain and Martin by the method of electromotive force, and of McBain and Bolam by the method of catalysis of nitrosotriacetanamine. Great diversity of opinion had

previously prevailed as to whether soaps were scarcely or almost entirely hydrolysed. This was chiefly due to the employment of such unsuitable methods as direct titration in order to study this equilibrium, which was bound to lead to arbitrary and erroneous results.

Our quantitative data show that all soap solutions are alkaline, varying from  $N/3000$  to  $N/300$  for pure soap solutions, down to  $N/30000$  for acid sodium palmitate,  $\text{NaIP}_2$ . Several per cent. excess of alkali hydroxide is required to drive back this slight hydrolysis completely, but, even in the presence of large excess of alkali, not more than a few per cent. of this alkali can be sorbed by the soap. This means that the hydrolysis must be approximately equal to the alkalinity.

The highest soaps are the most alkaline, and the alkalinity is less at lower temperatures until the solution becomes heterogeneous, whereupon the alkalinity increases several fold. The alkalinity is less in the presence of moderate amounts of salt. Probably for these reasons, commercial soaps are all less alkaline than sodium palmitate. Since soaps are never used except in solution, and since free alkali is often injurious to the skin, this question of alkalinity is one that is of direct personal interest to many of us.

It has long been known, especially since the brilliant investigations of Spring, that the alkalinity of soaps has but little to do with their washing power. This property is due entirely to the colloid which is present.

In very concentrated soap solutions the hydrolysis diminishes with abnormal rapidity. This is probably due to the palmitate ions, the subjects of this hydrolysis, having disappeared to form ionic micelle.

#### *The conductivity of soap solutions.*

Some years were spent in this laboratory in obtaining accurate data for one of the higher soaps, and no effort was spared to eliminate and control all sources of error. Since then we have accumulated a mass of data for all the pure soap solutions and for a few mixtures. Other determinations in the literature, mostly published since McBain and Taylor's work, are in substantial agreement, although in some cases the experimental difficulties appear not to have been completely overcome. Some of our results are given in Tables I.—III., in which concentrations are expressed in gram-equivalents of soap per 1000 grms. of water.

The results in these tables show the strikingly anomalous form of the conductivity curves of the higher soaps and also their magnitude as compared with the acetates, the nearest true salts. Sodium and potassium soap solutions are very similar (see the curves given by Bunbury and Martin, *Chem. Soc. Trans.*, 1911, 105, 421).

It is beyond question that soaps exhibit a high proper conductivity, and that this is abnormally high in concentrated solutions. In the case of the higher soaps the conductivity, after passing through a minimum at about  $N/10$ , rises with increase of concentration up to nearly  $N/1$  solution. This is in excellent agreement with the hypothesis of an ionic micelle of conductivity intermediate between that of sodium and potassium ion, which was deduced from other considerations. The replacement of the comparatively slow large true ion by the more highly conducting micelle, and the final gradual falling off in the concentration of the latter as the concentration of the solution is still further increased, are exactly mirrored in these curves. It would be difficult to find another explanation for them.

#### *The osmotic properties of soap solutions.*

It is surprisingly difficult to determine the osmotic behaviour of soaps by any of the usual methods.

We were able to show that the boiling point method is useless, likewise the lowering of vapour pressure, on account of the unavoidable presence of enclosed air. The osmometer methods are obviously useless for a system in which there is an equilibrium between colloidal and crystalloidal forms of the same material, unless a strictly semi-permeable membrane could be found for the purpose.

Together with Mr. Salmon, I devised a dew point method of measuring the "molecular weight," and obtained data for about one hundred soap solutions. This method is very useful, but it is unfamiliar, and has not been applied before to solutions. Incidentally it may be mentioned that the measurements were extended to soap curds and solid soap, with regard to which data will be published later.

For a very few soaps, by taking special precautions, the freezing point method can be used. This leads to values for only one temperature, but the results corroborate the findings of the dew point method. Of course, the osmotic effect is essentially a measure of the large amount of crystalloidal present. Table IV. gives a few selected data for the amounts of crystalloid thus indicated by these two methods, for  $90^\circ$  and for  $0^\circ$  C.

TABLE IV.  
*Total crystalloidal material present in soap solutions.*

Concentration ..	0.1	0.2	0.5	1.0	2.0
K Acetate, $\text{C}_2$ , at $90^\circ$	—	0.38	0.95	1.78	—
Na Acetate, $\text{C}_2$ , at $90^\circ$	—	—	0.93	1.74	—
at $0^\circ$	—	0.38	0.95	1.97	—
K Caprylate, $\text{C}_8$ , at $90^\circ$	—	0.35	0.72	1.24	—
at $0^\circ$	—	0.39	—	1.37	1.69
K Laurate, $\text{C}_{12}$ , at $90^\circ$	—	0.31	0.54	0.66	0.99
at $0^\circ$	0.094	0.132	0.200	0.397	0.393
Na Palmitate, $\text{C}_{16}$ , at $90^\circ$	—	0.27	0.41	0.52	1.04
K Oleate at $0^\circ$ ..	0.049	0.063	0.149	—	—

The first fact about the osmotic data is that the osmotic activity is real, and moderately great. The most interesting conclusions can only be obtained by comparing these data with the conductivity results. The remarkable fact then appears, that in the concentrated higher soap solutions (about  $\text{C}_{12}$  upwards) the whole of the osmotic activity is required to account for the potassium or sodium ion alone, and even that only on the assumption that the conductivity of the negative carrier (our colloidal "ionic micelle") conducts almost as well as the potassium ion. Thus the potassium or sodium ion is the only crystalloidal constituent of these concentrated soap solutions.

A further study of all the data leads to the conclusion, in conformity with theory, that the solvation of the ionic micelle diminishes with increase of concentration, and thus its conductivity increases. Its formula is probably  $(\text{NaP})_x \cdot \text{P}^{(1)} \cdot (\text{H}_2\text{O})_m$ , where  $\text{P}^{(1)}$  is the anion of the fatty acid in question.

#### *The effect of high and low temperature on soap solutions.*

With regard to solutions which are clear and approximately homogeneous, a low temperature favours formation of colloid. Further, this colloid is more hydrated than at higher temperatures, as may be seen by reference to Table IV. The various



adjustments in the equilibria involved when a solution is cooled or heated are made very quickly and are very soon complete to within one per cent. This ensures that definite reproducible properties and effects are always obtainable from a soap solution of any particular composition under any specified set of conditions. As long as the soaps are in solution there is very little difference between potassium and sodium soaps except that the former are always more colloidal.

At room temperature, all but extremely dilute solutions of all sodium soaps above the caprylate ( $C_8$ ) are solid. This solidification withdraws soap from solution and is essentially crystallisation, the mother liquor being mechanically enclosed. Very dilute solutions of sodium soaps are broken down into an aqueous liquid containing some alkali and a trace of soap, and in this are suspended the acid soaps which are the product of hydrolysis.

The potassium soaps solidify at much lower temperatures and they possess a much greater tendency to exist as transparent liquids which are often so viscous as to be almost, or quite, gelatinous. To obtain the same solidification (crystallisation) phenomena as with the sodium soaps, it is necessary to use higher concentrations of the potassium soaps of greater carbon content and to have a much lower temperature. In other words, the potassium soaps are more "soluble" than the corresponding sodium soaps. The soap solutions which readily crystallise can also be more easily salted out by the concentrated salt solutions, though this is an entirely different phenomenon.

It is worth while noting that supersaturation with regard to crystallisation often occurs and may easily be prolonged for years. Inoculation or mechanical shock brings about the solidification to a white solid in a relatively short time. We have only made a beginning with the study of the phenomena of solidification and of salting out, and a considerable extension of this work will be necessary, as it is of direct manufacturing importance.

#### *The viscosity of soap solutions.*

We have made only qualitative observations of this property, but other investigations (and in particular one by Kurzmann deserves mention, *Kolloid-Chemische Beihefte*, 1914, 5, 427) have made exact measurements for a series of cases. The viscosity of soap solutions varies enormously, from about that of water up to a value many thousand-fold greater, giving a perfectly continuous transition from thin fluid, through viscous liquids which can be "span," to a very stiff clear jelly. This change, by the way, is not accompanied by any very great change in the electrical conductivity.

The viscosity is increased in very rapid exponential fashion by increase of concentration, lowering of temperature, or rise within the homologous series of the fatty acids. The addition of any electrolyte such as a hydroxide, carbonate, or chloride in small quantities reduces the viscosity slightly through a minimum and then increases it quite enormously. All of these viscosity changes are quite in accordance with the theory of colloidal electrolytes which is described in detail above.

#### *Influence of position in the homologous series.*

The appearance, washing power, density, and conductivity of the caproates ( $C_6$ ) distinctly mark the beginning of that deviation from the behaviour of the acetate, which rapidly and regularly increases through the other members of the homologous series until the typical character of the higher soaps is attained. At the top of the scale is behenic acid ( $C_{22}$ ) often met with in modern soaps and whose soap solutions solidify at a high temperature. The behenates tend to form nearly undissociated colloid and the solutions often have a distinct structure like those of starch. The

laurates ( $C_{12}$ ) are perhaps the most interesting, as they are just in the middle of the series, the lowest really good soap in whose solutions by suitable adjustment of concentration most of the typical phenomena of the soaps may be produced. This acid is perhaps the most important constituent of industrial soft soaps. Solutions of the sodium salt are usually solid at room temperature. When liquid they, like those of potassium oleate, are viscous liquids or jellies.

Oleic acid ( $C_{18}$  unsaturated) produces soaps *par excellence*. At room temperature the sodium soap solutions are clear. The distinctive behaviour of these soaps is due to the fact that the formation of hydrophilic ionic micelle persists into unusually low concentrations (see for instance Table IV.).

#### *The properties of mixed soaps.*

It will be seen from the above summary that we have now for the first time a fairly definite idea of the constituents of soap solutions. With this picture in our minds we are now able; much more extensively than was possible before, to draw upon the knowledge of colloids in general in order to understand and predict the various properties of soaps and their solutions.

Again, although our work of necessity had to be carried out with pure materials, it is now possible to make predictions with regard to various mixtures. This is essential, since commercial soaps are composed of an almost infinite variety of combinations of fatty acids. It is evident from our theory that the behaviour of mixtures is of course by no means additive.

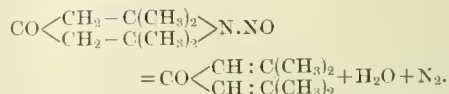
The investigations are still only in their first stages, but it is hoped that the series already completed of laborious and often difficult experiments will have laid a foundation for the elucidation of the industrial and theoretical properties of these very important and interesting substances.

### A METHOD FOR THE DETERMINATION OF THE ALKALINITY OF CERTAIN SOLUTIONS.

BY F. FRANCIS.

For several years the catalytic decomposition of nitrosotriacetoneamine, and similar substances, under the influence of hydroxyl ions has been studied in the Chemical Laboratories of the University of Bristol by my students and myself. (*Chem. Soc. Trans.*, 1912, 101, 2358; 1913, 103, 1722; 1915, 107, 1651.)

Substances of this class break down, when acted upon by alkalis, with the evolution of nitrogen and the formation of phorone or its analogues; in the case of nitrosotriacetoneamine itself the reaction may be expressed as follows:—



Although in the case of some of the nitrosoamines and in certain concentrations of hydroxyl ions there appears to be a simultaneous evolution of small quantities of oxygen, this does not interfere with the method.

It was found that the velocity of this decomposition depends on the concentration of hydroxyl ions, and consequently an estimation of the rate at which nitrogen was evolved constitutes a method for the determination of the alkalinity of those liquids in which—owing to colour or for other reasons—it is inconvenient or impossible to carry out the estimation by any of the usual methods such as the use of an indicator or electro-motive force. My colleague, Dr. J. W. McBain, for instance, has used this method for the determination of the alkalinity

of certain soap solutions. The decomposition is that expressed by the unimolecular equation,

$$K = \frac{2.303}{t^0} \log \frac{P_\infty - P_0}{P_\infty - P},$$

where  $P_0$  is the pressure reading at the time taken as zero,  $P$  is the reading at time  $t^0$ , and  $P_\infty$  the infinity reading. The minute is taken as the unit of time.

The rate of evolution of nitrogen is most accurately followed by pressure readings, and the apparatus and method have been described in detail (Chem. Soc. Trans., 1915, 107, 1653); a much simpler apparatus may be employed and the volume of the evolved gas measured in a burette instead of reading its pressure, although this method is not quite so accurate as the former.

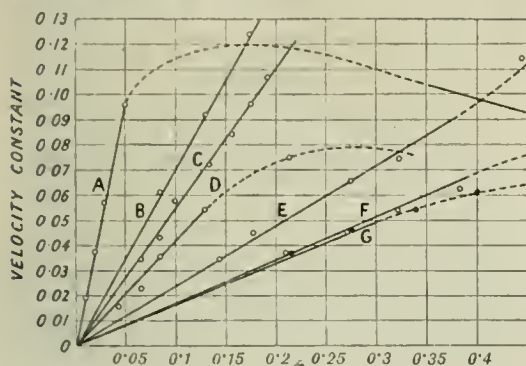


FIG. 1.

Concentration of hydroxyl ions, N.

#### Catalysis of Nitrosoamines with Small Conc. OH'.

Nitrosoamines of—

- A. Triacetanamine at 30°
- B. Iso-butyldiacetonamine at 50°, E. at 40°.
- C. Vinylidiacetonamine at 50°, F. at 40°.
- D. Valeryldiacetonamine at 50°.
- G. N-Butyldiacetonamine at 40°

Actual values for the velocity constant, K, for a series of hydroxyl ion concentrations, together with values for several of the nitrosoamines, and deter-

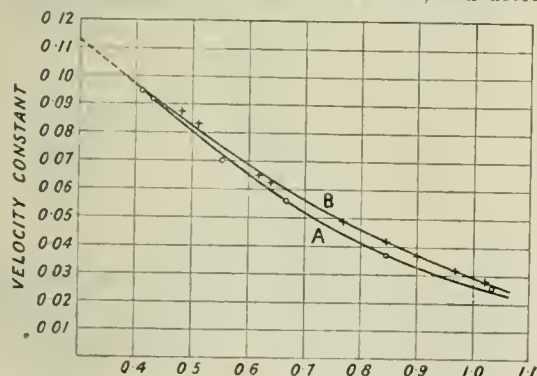


FIG. 2.

Concentration of hydroxyl ions, N.

#### Catalysis of Nitrosotriacetanamine with High Conc. of OH'.

t = 30°.

- A. Sodium hydroxide.
- B. Potassium hydroxide.

minations of the temperature coefficients of these reactions, will be found in the papers mentioned above.

Fig. 1 shows the relationship between the velocity constant and concentration of hydroxyl ions for a

series of nitrosoamines. The dotted portions of the curve indicate that the constants are drifting, and in such cases a mean value has been taken. Since nitrosotriacetanamine and nitrosovinylidiacetonamine are more easily prepared than the others, either one or the other of these substances is most suitable for the determination of the concentration of hydroxyl ions up to 0.4N.

The following table summarises the various data for these two substances for small concentrations of hydroxyl ions. It will be noticed that the relationship between velocity constant and concen-

tration of such ions—*c.g.*,  $\frac{K}{[OH]}$ —in such regions is expressed by a straight line. In the table R is the value for this constant, 1.92 for nitrosotriacetanamine at 30° C., and 0.171 for the vinyl derivative at 40°.

Nitrosoamine.	Region of OH' concentration.	Value of R.	Range of temp.
Nitroso-triacetonamine	up to 0.05N	$\frac{t-30^\circ}{10} \times 1.92 \times 2.35$	30°–40°
Nitrosovinyl-diacetonamine	up to 0.4N	$\frac{t-40^\circ}{10} \times 0.171 \times 2.2$	40°–50°

In fig. 1, it will be seen that in the case of all the nitrosoamines investigated, as the concentration of hydroxyl ions increases there comes a point when the constants "drift," on further increase this ceases, and with increasing concentration the velocity constant falls. The following table gives the region of concentration for which the previously mentioned nitrosoamines are suitable:—

Nitrosoamine.	Conc. of OH' at which K commences to drift.	again becomes constant.	Maximum conc. of OH' ions for which nitrosoamine is suitable.
Nitroso-triacetonamine	about 0.05N	about 0.35N	up to 1.4N for both NaOH and KOH
Nitrosovinyl-diacetonamine	about 0.40N	{ about 1.4N about 1.55N	up to 2.0N for KOH up to 1.55N for NaOH

In higher concentrations a curve of standard results may be made from the data already given (Chem. Soc. Trans., 1915, 1660). Fig. 2 gives the relationship between the velocity constant and high concentrations of hydroxyl ions for the case of

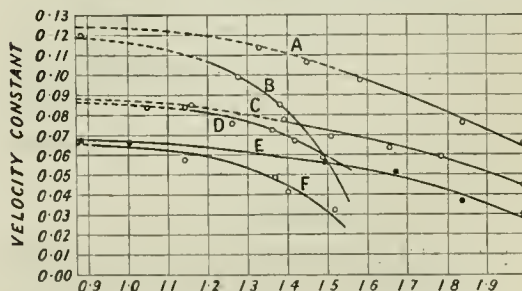


FIG. 3.

Concentration of hydroxyl ions, N.

#### Catalysis of Nitrosoamines with High Conc. OH'.

t = 40°.

Nitrosoamines of—

- A. Isobutyldiacetonamine with KOH.
- B. " " " NaOH.
- C. Vinylidiacetonamine " KOH.
- D. " " " NaOH.
- E. N-Butyldiacetonamine " KOH.
- F. " " " NaOH.



nitrosotriacetanamine which has been more closely examined than the others. It will be noticed that the curve for hydroxyl ion concentration from sodium hydroxide does not quite coincide with that from potassium hydroxide; probably this is due to neutral salt action. Velocity determinations of the decomposition of other nitrosoamines in concentrated hydroxyl ion solutions are shown in fig. 3.

The effect of neutral salts on the velocity constant of the decomposition of nitrosotriacetanamine when the concentrations of hydroxyl ions are small, is shown in fig. 4, where the percentage retardations are plotted against the concentration of the neutral salts used.

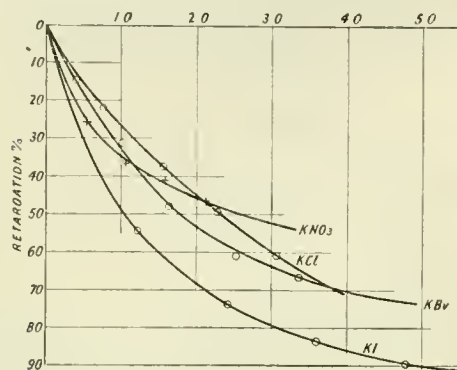


FIG. 4.

Concentration of salt,  $N$ .

Effect of Neutral Salts on Catalysis of Nitrosotriacetanamine. Conc.  $\text{OH}' = 0.0455\ N$ ,  $t = 30^\circ$ .

The group of curves in this case may be compared with a corresponding set for nitrosovinylacetanamine where the velocity determinations were made at  $50^\circ\text{C}$ . (fig. 5). Comparison shows the very

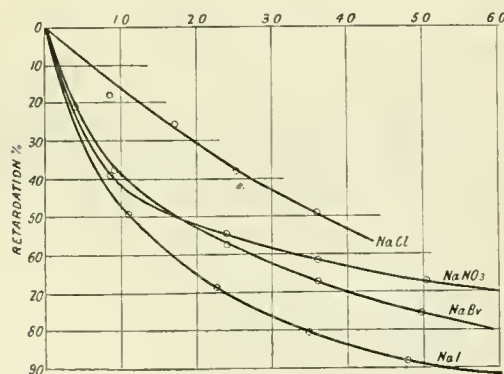


FIG. 5.

Concentration of salt,  $N$ .

Effect of Neutral Salts on Catalysis of Nitrosovinylacetanamine. Conc.  $\text{OH}' = 0.1602\ N$ ,  $t = 60^\circ$ .

striking similarity that exists between the two sets of observations, which, it will be noted, were carried out with different nitrosoamines and at different temperatures.

The effect of neutral salts on the velocity constant in high concentrations of hydroxyl ions is shown in fig. 6. As in the previous figures a dotted curve indicates that the constants are "drifting" and a mean value has been taken. It is possible that the addition of such salts as

potassium iodide and thiocyanate may depress the dissociation of the potassium hydroxide and hence accelerate the reaction to such an extent that the concentration of hydroxyl ions falls to that region

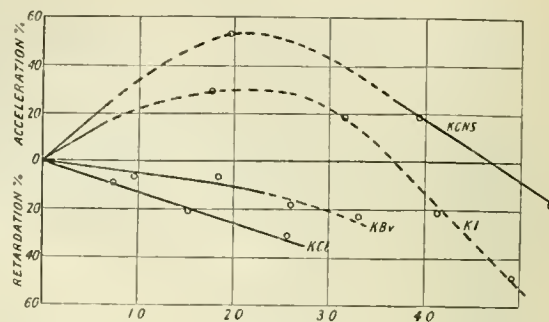


FIG. 6.

Concentration of salt,  $N$ .

Effect of Neutral Salts on Catalysis of Nitrosotriacetanamine. High Conc. of  $\text{OH}'$ ,  $t = 30^\circ$ .

which I have termed the "drift" region, and possibly beyond where the relationship between velocity constant and concentration of hydroxyl ions is expressed by a straight line.

It follows from this brief sketch of the effects of neutral salts, that when this method is employed for the determination of alkalinity in the presence of a large excess of such substances, a due allowance must be made in the value of the velocity constant which may be found, and the method may lose some of its convenience as a means of estimating the concentration of hydroxyl ions in such solutions. The following table gives the relationship between the hydroxyl ion concentration, and that of sodium hydroxide and potassium hydroxide at  $30^\circ\text{C}$ : from  $1\ N$  upwards the temperature is  $40^\circ\text{C}$ . At low concentrations the dissociation of potassium and sodium hydroxide is very similar.

Conc. of base $N$ .	Conc. $\text{OH}' N$ for $\text{KOH}$ .	Conc. $\text{OH}' N$ for $\text{NaOH}$ .
0.005	0.00195	—
0.02	0.0177	—
0.2	0.175	—
0.4	0.335	—
0.8	0.630	0.585
1.0	0.77	0.705
2.0	1.36	1.15
3.0	1.78	1.39
4.0	2.06	1.51

#### DISCUSSION.

Dr. J. C. CATN said that the apparatus was very similar to one which he had used but which had been much criticised on account of the possibility of supersaturation of nitrogen in the liquid unless it was exceptionally well stirred by a mechanical agitator.

Dr. RIXON asked whether there was any difference in the constants obtained with the forms of apparatus described.

Dr. H. W. BYWATERS asked if the apparatus could be used to determine the hydroxyl ion concentration of blood or similar complex substances.

Prof. FRANCIS replied that the question of supersaturation depended upon efficient shaking. The whole of the apparatus was placed in a thermostat and shaken vigorously, and unless this shaking was vigorous, supersaturation would vitiate the

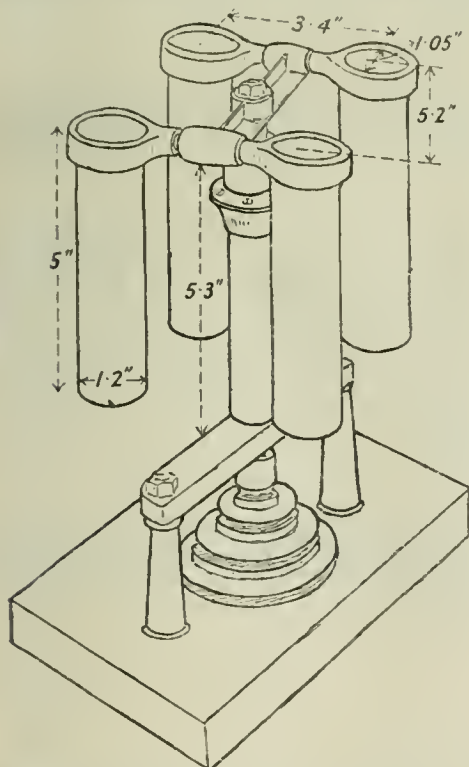
results. In reply to Dr. Rixon, he said the volume measurements in both cases were carried out under atmospheric pressure, so that the results were comparable. It was possible to measure extremely small concentrations of hydroxyl ions by his method, but it had never been used for physiological liquids.

#### THE APPLICATION OF THE CENTRIFUGE IN COMMON LABORATORY PRACTICE.

BY F. W. RIXON, PH.D., M.Sc.

An attempt has been made to construct and test a centrifuge that would be in price within the reach of the smallest laboratory, and at the same time allow an application of ordinary laboratory methods and quantities in its usage. Any motive power may be employed, the most satisfactory being a motor provided with a substantial starting resistance that will not burn out if the motor is run for some time at low speed. A small sixteenth horse power motor has been used with success.

Hand or foot drive may be used. A convenient and readily procured gearing can be made by replacing by a pulley the emery wheel of a grinding machine, and lengthening the handle. The drive is carried by a round leather belt (sewing machine belt) to a stepped and grooved pulley mounted on the driving spindle of the centrifuge, which is of simple construction and can be built by any capable mechanic. The spindle is practically an elongated back wheel bicycle bearing, one ball bearing being sunk on a heavy iron base, the other mounted on a stout tube which surrounds the spindle for about



half its length and is then screwed into a stout cross-piece supported on two pillars which are bolted to the heavy iron base. Immediately below this cross-piece the stepped and grooved pulley is fixed on the spindle by a set screw. To the upper end of the spindle an arm is fixed by means of a nut; this arm carries in plain bearings at each end

a rod which screws at each end into collars on which are fixed thin brass tubes of such size that the usual 6 in.  $\times$  1 in. test tube is a snug fit with about an inch projecting above the collar. A small pad of cotton wool is at the bottom of each tube. The full load of this model is therefore four such test tubes.

Modifications in design suggested by a lengthy use of the model are a tripod mounting for the top spindle in place of the present tube and cross bar, and in place of the arm, a disc with attachments for a greater number of tubes, variable at will.

The centrifuge should be firmly fixed to a stout bench and have some protective covering—a cage of stout galvanized wire netting with one side cut away slightly for the drive answers this purpose. The belt is always used crossed.

Ordinary test tubes, if selected carefully, serve most purposes; where comparatively high temperatures have to be used in operations subsequent to centrifuging quartz test tubes are substituted.

Further details can be seen on the figure, where the more essential dimensions are noted.

Such a machine as that described, with motor and resistance, represents an outlay of about £5; its value has been very evident during three years' use in the laboratory.

In preparative work the separation of substances difficult to filter is effected more rapidly than by suction filtration. Separation of crystals from solvent and subsequent washing can be rapidly and easily carried out. In almost all cases the solid settles in the tube in the form of a cake which is not disturbed when the liquid is poured off and yet is sufficiently soft to be broken up by vigorous shaking; this is the case not only with crystals but with precipitates also, and thus it is possible to abbreviate a qualitative analysis very considerably.

In gravimetric analysis the curtailing of the usual processes should be very marked. It should be possible in a great number of cases to carry out the whole of an estimation in one vessel, thus avoiding not only the errors of transference but also the use of filter paper and the after-treatment of the paper.

A number of test analyses which have been carried out have indicated the utility of the method. The procedure in general was as follows:—A glass or quartz tube was thoroughly steamed out, dried, and weighed. The temperature at which the tube is dried should approximate to the final temperature of the analysis. The substance to be analysed is weighed into the tube; the amount should be less than that taken for ordinary analysis, particularly if the precipitate to be formed is strongly adsorptive, such as barium sulphate or aluminium hydroxide, when the weight taken should be about one quarter the usual. In such cases also large excess of reagent should be avoided. The substance is dissolved, precipitated, and centrifuged (if only one tube is being dealt with it should be balanced in the machine by a dummy), and the clear liquid is poured off, the precipitate broken up and washed by shaking or boiling, again centrifuged, and the process repeated until the washings are satisfactory, then as much as possible of the final washing is poured away, and the precipitate dried by heating first in an oven and finally over a burner to the required temperature, cooled, and weighed; or the tube may be first cautiously heated over a small flame and when dry receive its final heating.

In certain cases a fine scum appears on the top of the liquid in the tube and refuses to sink, but it may be conserved in one of two ways. If the filtrate be poured into a similar tube and this tube filled to the top with water the scum may be blown back into the original tube by a smart puff, or the formation of a scum may be prevented by the addition of a very small quantity of a solution of saponin, a procedure recommended by Nolte, who carried out a series of determinations of potassium,



sodium, and calcium in a centrifugal machine of larger type (Landw. Versuchs-Stat., 1915, 449.) It may be mentioned that the cost of Nolte's installation was some £150.

The value of the method of which a general outline has been given may be seen from a few results obtained mainly by students, thus making the test a fair comparison.

In a series of precipitations of silver chloride from silver nitrate (each of which occupied  $1\frac{1}{2}$  hrs.), the errors in weight obtained were -0.0007, -0.0006, -0.0009, -0.0005, -0.0006, -0.0006, and -0.0005 gm., the weight of precipitate being about 0.4 gm. Again, in precipitations of barium sulphate from barium nitrate the errors were +0.0001, -0.0015, -0.0023, -0.0010, -0.0006, -0.0007, and -0.0012 gm., the weights of precipitate varying from 0.2 to 0.75 gm.

A series of experiments in which barium sulphate was precipitated in presence of various salts gave interesting results, showing the method as lending itself to an examination of adsorption and emphasising the known adsorptive properties of this substance. In cases where a well-known adsorptive substance is present particular care should be taken to exercise restraint in the use of reagents and also of "original" material. If barium sulphate be precipitated by the addition of a large excess of potassium sulphate the results are uniformly high. Again, when barium sulphate is precipitated from magnesium sulphate by the addition of twice the theoretical amount of barium chloride, the errors are much higher in proportion when a larger weight of material is used. In dealing with small quantities it is advisable to weigh the tube used before and after the experiment as the weight loss during repeated boilings is with some types of glass quite appreciable.

A number of estimations of tin in solder were also made, the complete analysis being carried out in the tube. The percentage of tin found by the ordinary method was 24.50 (probably there is a constant method difference), the time taken for a single determination being three hours. The following results were obtained:—Sn 24.96, 24.92, 24.81, 24.89, 24.78, 24.87, 24.76%.

A number of other determinations have been carried out with equally satisfactory results. The method is one that offers in a great number of cases many advantages with a considerable saving of time and with little initial outlay, and it seems that almost all the approved gravimetric methods should be capable of suitable adaptation.

It is with pleasure that I thank the Colston Society for a grant towards the expenses of the work, also Mr. Alway, Mr. Tyler, and particularly Mr. Vincent for a large number of analyses carried out.

#### DISCUSSION.

Dr. H. W. BYWATERS said that a good deal of work had been carried out in America on the use of the centrifuge for the estimation of phos-

phates. He asked whether the results obtained in a centrifuge compared favourably with the ordinary gravimetric methods.

Mr. F. SOUTHERDEN asked whether the method was satisfactory in dealing with precipitates which did not settle satisfactorily, such as zinc sulphide. He suggested that where a large number of estimations had to be carried out, instead of weighing, the depth of the precipitate might be measured.

The PRESIDENT asked whether the author had used the method for such special work as the determination of phosphorus in iron and steel. There were several processes which measured the volume of the precipitate instead of weighing it for these determinations, and it appeared to him that the centrifuge ought to be specially applicable to these processes.

Dr. RIXON, in reply, said that he had satisfied himself that the machine could be used satisfactorily for a number of cases of gravimetric analysis. The application of the machine to methods in which the depth of the precipitate was obtained was quite possible, although he had not tested them. He saw no reason why the machine should not be used in practically any gravimetric analysis.

Dr. E. F. ARMSTRONG read a short paper entitled "Some Technical Applications of Catalysis," in which he dealt with recent developments in the catalytic synthesis of acetaldehyde and acetic acid. Under the present circumstances it is considered inadvisable to publish this paper.

On Wednesday evening the members were received at the Bristol Museum and Art Gallery by the Lord Mayor and Lady Mayoress, who were attended by the Sheriff.

The Annual Dinner was held at the Grand Hotel, Bristol, on Thursday evening, the President occupying the chair. Following the loyal toast, proposed by the President, the toast of the Society of Chemical Industry was proposed by Mr. Windus, and responded to by the President, and that of the Bristol and South Wales Section by Mr. Lloyd Howard, and responded to by Dr. T. H. Butler, who, in turn, proposed the toast of the University of Bristol, to which Prof. Francis replied.

The dinner was followed by an informal concert.

Friday afternoon was devoted to visits to the tobacco factory of Messrs. W. D. and H. O. Wills, Ltd., Messrs. Parker Brothers' tanneries, and the University Agricultural and Horticultural Station.

## Canadian Section.

Meeting held at Ottawa on May 22nd, 1918.

MR. T. H. WARDLEWORTH IN THE CHAIR.

## THE EXAMINATION OF COMMERCIAL DEXTRIN AND RELATED STARCH PRODUCTS.

BY F. W. BABINGTON, ALFRED TINGLE, AND C. E. WATSON.

The materials to be considered come under two heads, *viz.*, (1) starches that have been so treated as partly to hydrolyse a portion of the material, and (2) mixtures of untreated starches with starch that has been submitted to hydrolysis. Both classes of material may be treated analytically in the same way.

Starch furnishes many and ill-defined products when hydrolysed, but for commercial purposes it often suffices to distinguish between, and determine, "starch" and "dextrin gum."

Our increasing but still incomplete knowledge of this branch of carbohydrate chemistry has caused the name starch to have a wider and more vague meaning than formerly, so that it is necessary to define the sense in which it is here used. Analytically we consider as starch the carbohydrate or group of carbohydrates which, whether soluble in cold water or not, form a solution or gelatinise with hot water, give a blue colour on treatment with iodine, and are precipitated by semi-saturation of the cold solution with barium hydroxide.

By dextrin gum is meant the mixture which results from the hydrolysis of starch when the change has not been carried so far as a complete conversion into sugars, though the latter may be present in the gum. "Dextrin gum" gives no blue colour with iodine and is soluble both in cold water and in a cold, half-saturated solution of barium hydroxide.

The method of Lamb and Harvey (*J. Soc. Dyers and Col.*, 1918, 34, 10; this *J.*, 1918, 133) for the analysis of these mixtures is not satisfactory. It includes as "starch" only insoluble starch, while soluble starch is determined as part of the "dextrin." We have found that when the starch and dextrin are mixed in certain proportions it is very difficult to wash the former free from the latter with water; filtration is very slow and the starch passes into solution during the process to a serious extent. With reference to our first objection, we admit that for many commercial purposes it may be desirable to determine soluble starch among the dextrins. To adopt such a practice in our own laboratory, however, would not be satisfactory, as importers would thereby be penalised.

A method of examination which is both short enough for practical purposes and scientifically accurate is much to be desired, but for the present we have aimed at simplicity of procedure and a probable error of less than 5%.

**Determination of dextrin gum.**—The sample (1 gm.) is warmed in a 100 c.c. graduated flask, with 30 c.c. of water until just gelatinised, and cooled quickly; 50 c.c. of a cold saturated barium hydroxide solution is next added (the flask being meanwhile shaken), followed by enough water to bring the total volume to 100 c.c. The solution is filtered through a dry 15 cm. paper and an aliquot portion (50 c.c.) of the filtrate is pipetted into a platinum dish. After the addition of 2 drops of a

1% phenolphthalein solution, *N/1* hydrochloric acid is added cautiously till the neutral point is just passed. A faint pink colour is then restored by the addition of two or three drops of the barium hydroxide solution. A weighed quantity (about 10 grms.) of sand is added, and the dish heated on a water-bath. The sand should be stirred when almost dry, to expose the maximum surface to the air, after which the dish is transferred to a well-regulated oven and dried to constant weight at 120° C., below which temperature crystallised barium chloride is dehydrated. Dextrin gum being hygroscopic, precautions must be taken accordingly. The dish is now heated strongly, but not beyond the temperature necessary for the complete combustion of the organic matter. During ignition the sand should be well stirred at intervals to allow full exposure to the air. The dish and contents are then cooled and weighed. The difference in weight before and after ignition represents the dextrin gum in 50 c.c. of filtrate, *i.e.*, in half the weight of sample taken.

**Limit of accuracy.**—The phenolphthalein and the barium hydroxide added in excess of neutrality are obvious sources of irregularity, but if the amounts laid down above are not exceeded this is unimportant. A blank determination showed that the total resultant error from this source was 11 milligrams or 0.22% on the basis of a 1-grm. sample.

The precipitation of starch by barium hydroxide is not quite complete, and that portion which remains in solution must ultimately be weighed as dextrin gum. The error likely to arise from this cause was found by making blank determinations of dextrin gum in two samples of starch. No. 1 was arrowroot and No. 2 a corn starch.

No.	Weight of sample.	Weight of dextrin gum found.	Dextrin gum found, %.
1	grms. 1.00	grms. 0.0053	% 1.06
2	1.00	0.0087	1.74

Varying weights of arrowroot starch were added to measured volumes of a well standardised dextrin solution and these mixtures were examined by this method, with results shown in the following table:—

Mixture No.	Weight of starch taken.	Weight of dextrin in solution.	Dextrin.	Total weight of dextrin gum found.	Dextrin gum found.
	grms.	grms.	%	grms.	%
1	1.50	2.4879	62.3	2.4430	61.2
2	1.00	2.4879	71.3	2.3700	67.9
3	1.00	2.4879	71.3	2.5044	71.8
4	0.50	2.4879	83.2	2.4858	83.1

The error is thus within the limit aimed at, though much larger than would be tolerable for purposes of careful scientific enquiry.

Mixtures of unknown composition were next examined, all being ordinary articles of commerce. They were:—(A) "Yellow dextrin"; (B) "Soluble starch"; (C) "Soluble gum" (a textile dressing); (D) "Treated starch" (believed to be a starch which had been washed with dilute acid). Each sample was examined independently by two analysts, so that differences in the result of duplicate analyses display all the error which can arise from "personal equation." The results obtained were as follows:—Dextrin gum % in (A) 99.90, 99.74; (B) 11.60, 11.86; (C) 24.00, 25.00; (D) 17.00, 15.72.



If dextrin gum is determined by the above method and determinations are also made of ash and moisture, the starch may then be estimated by difference. Such a procedure is not always desirable, and we have commenced work on a method for estimating the total starch directly, by means of the polarimeter, after hydrolysis. The pressure of departmental routine has prevented the completion of our experiments. At present it is enough to say that we have found that starch which has been precipitated by barium hydroxide is somewhat more rapidly hydrolysed by hydrochloric acid than when not so treated. Whether the hydrolysis is complete we are not prepared to say, but we find that under our experimental conditions ( $100^{\circ}\text{C}$ . at atmospheric pressure) no further reduction in rotatory power can be observed after  $1\frac{1}{2}$  hours. Pending the completion of this work we suggest that the following determinations on such starch products as we have been considering will meet most commercial requirements:—(a) Ash. (b) Moisture. (c) Dextrin gum by the method described here. (d) Insoluble starch and (e) reducing sugars by some such method as that described by Lamb and Harvey (*loc. cit.*). (f) Total starch by difference  $[100 - (a + b + c)]$ . (g) Soluble starch by difference  $[f - d]$ . (h) Non-reducing dextrin gum, by difference  $[c - e]$ .

Our method may also be applied to mixtures of starch and gum arabic, such as are sometimes met with commercially.

Analytical Laboratory,  
Department of Customs,

Received, July 11, 1918. Ottawa.

Meeting held at Montreal on December 14th, 1917.

MR. T. H. WARDLEWORTH IN THE CHAIR.

## THE UTILISATION OF THE PEAT RESOURCES OF CANADA.

BY B. F. HAANEL.

### *Canadian peat deposits.*

The total area of Canada covered by peat bogs has been estimated to be 37,000 square miles, and the average depth of the peat deposits is put conservatively at 6 feet. A large portion of this area is in the North-west Territories, and the Yukon, and is not at present of economic importance, but over 12,000 square miles are situated in the central provinces of Canada.

Assuming that all the peat in this 12,000 square miles is suitable for the manufacture of fuel, it represents a quantity of standard peat fuel (containing 25% moisture) equivalent to over nine billion tons, which, on a basis of actual heating value, is equivalent to over five billion tons of good anthracite coal.

During the past ten years the Mines Branch, Department of Mines, has completely investigated and mapped 58 Canadian bogs, all of which are situated conveniently with respect to inhabited and industrial communities, railway and other transportation facilities. The investigations are conducted to determine the principal and controlling characteristics of a bog, *viz.*, its area, depth, quality at different depths, quantity in tons, and, in general, its suitability for any particular purpose. The area examined in detail comprises 170,000 acres, and represents a quantity of standard peat fuel estimated at 120,000,000 tons. Seven bogs

conveniently situated with respect to Toronto could supply that city with 26,500,000 tons of fuel, and seven bogs within easy reach of Montreal could supply 23,500,000 tons of fuel. Excellent bogs are, likewise, conveniently situated with respect to thickly inhabited communities in Nova Scotia, New Brunswick, and other parts of Canada.

### *Past efforts to create a peat industry.*

Whether or not a particular natural substance shall be exploited, has usually been decided from a "profit" point of view. Peat, not holding forth great prospects for fabulous profits, failed to attract the attention of large capitalists and industrial men, and therefore the creation of a peat industry was left in the hands of a few earnest and honest men with insufficient capital to succeed in an undertaking of this kind, and a few dishonest promoters. Several attempts have been made and as many failures with loss of capital involved have been recorded. But the larger portion of the capital lost could have been saved and a flourishing peat industry long ago established if the promoters had been advised by accredited engineers who understood their business. Instead, however, of profiting by the experiences of European investigators, money was expended in developing and testing ideas which had long ago been discarded as impracticable and, in many cases, impossible in the peat-using countries of Europe. Not until the results of the investigations conducted by the Mines Branch of the Department of Mines concerning the economic methods employed for the manufacture of peat fuel in European countries were placed at the disposal of the public, were men with unpractical ideas dissuaded from interesting people in their schemes.

I believe that the time is at hand when necessity will decide that Canadians utilise their peat resources, and in the most efficient manner. The exploitation of natural resources, by the individual, for the sole purpose of creating huge profits appears to be a thing of the past—at least it is to be hoped so.

### *Description of peat.*

Peat is a substance formed by the incomplete decomposition of vegetable matter either in, or in the presence of, water. The deposition of peat-forming matter may not be continuous, but may occur at irregular intervals; moreover, the vegetable matter composing the *detritus* laid down in the different periods may be of many different species. Consequently, a bog of considerable depth will have represented in it a series of strata or layers which may comprise many different forms of plant life. The stages of decomposition to which the various layers have proceeded may also be quite different. Material composing a peat bog may consequently be far from homogeneous.

The layers composing an old and well-humified peat bog are often so altered that scarcely a trace of the original plant structure remains. In such cases the peat mass is composed of such minute particles that in its natural state (associated with water) it possesses many of the properties of a colloid. The chemical alteration of the plant substances entering into the formation of all peat bogs gives rise to hydrocellulose, a gelatinous substance possessing the property of absorbing many times its own volume of water. It is a true colloid, and has properties which are of the greatest importance in the manufacture of peat fuel. This substance is distributed throughout the peat bog, but the quantity present in any particular section or layer is dependent on the nature of the plants composing such portions or sections and the degree to which the humification, or chemical alteration, has proceeded. It is, therefore, seldom, if ever,

found uniformly distributed throughout an entire bog.

Peat, in its natural state, is generally associated with about nine times its weight of water, and, moreover, the solid matter represents not only combustible substance, but also ash and other mineral matter. The separation of this large quantity of water, and the handling of so large a quantity of raw peat substance, in order to obtain a comparatively small quantity of combustible matter, represent the real problems in converting peat into fuel, on a commercial basis, and in a thoroughly economic manner.

Ignorance of the physical and chemical properties of peat, and a complete lack of knowledge concerning the results of the efforts made by European investigators, distributed over more than a century, are directly responsible for the lamentable failures with which the attempts in Canada have met.

#### *Manufacture of peat fuel.*

In order to remove the water content of peat, and to convert the substance into a convenient form of fuel, many people have proposed to employ presses of various designs, and to briquette the residue. All such attempts were doomed to failure, since the very nature of peat prohibits the employment of pressure for the removal of the water. The water content of well-humified peat cannot be reduced to below 80–85% by pressure alone, and, even in the case of very fibrous peats, it has been only possible to reduce the water content to a trifle below 80% by pressure. The bulk of the water content, therefore, must be removed by other means, that employed usually having been artificial heat. Such processes, employing pressure and artificial heat, are absolutely uneconomical, and, under present conditions, any process devised for the manufacture of peat fuel which depends on the employment of pressure or artificial heat, or both, for the removal of the water content, will inevitably result in failure.

M. Ekenberg, a Swedish chemist, who devoted a large part of his life to the investigation of problems connected with the manufacture of peat fuel, considered that the great difficulty with which peat parted with its water content was due to the hydrocellulose. He, therefore, attempted so to alter this substance chemically that the residue resulting would more readily part with its water by pressure alone, and he devised a process according to which the raw peat, containing upwards of 90% of water, was submitted to high temperature and pressure in specially designed iron tubes. The material was carbonised, the hydrocellulose was decomposed, and a material, considerably richer in carbon and of an appreciably higher calorific energy, was produced. But this altered substance still refused to part with the major portion of its moisture through pressure. The bulk of the water still had to be removed by artificial heat. Although several million dollars have been expended on this process by an English syndicate, and about forty thousand dollars more by the Swedish Government, it is to-day not a commercial success.

Peat fuel, however, is being manufactured economically and on a commercial scale in many of the European countries, and this industry has been expanding and growing more secure.

The only economic process in existence to-day is that which employs the sun and the wind for removing the moisture. This process is called the "wet process," and the product obtained is termed "machine peat." This is the process which the Mines Branch, Department of Mines, demonstrated at the Government peat plant at Alfred, Ont.

The "wet process" consists of the following steps:—(1) Excavating the raw peat by hand or

by specially designed mechanical excavators. (2) Transferring the excavated material to the hopper of a specially designed macerator. (3) Transferring and spreading this pulped peat on the portion of the bog prepared for drying purposes. (4) Cutting the spread peat, when sufficiently set, transversely and longitudinally into blocks, which, when dried to about 35% moisture, have the dimensions of ordinary building bricks.

The macerator, which performs, perhaps, the most important function of the entire process, consists of a cylindrical shell, inside which is a set of spiral rotating knives and a set of knives fixed to the inside of the cylinder. The material fed to the hopper is forced by the spiral knives through and against the fixed knives. The fibres, stems, and pieces of wood, which are often found scattered throughout a bog, are cut, torn or ground into small fragments, and the peat composing the different layers of the bog and the hydrocellulose are thoroughly intermixed. The degree to which pulping is carried is determined by regulating the opening of the spout through which the pulped material has to pass.

During the drying stage, the hydrocellulose content of the peat performs a very important function. It serves to bind the particles of the peat together, forming a concrete whole, and prevents the peat blocks from re-absorbing moisture when the drying period is interrupted by wet weather. This waterproofing of the peat block is due to the fact that the colloidal substance forms a skin covering the outside of the block, which always assumes a state of complete saturation. Hence it acts as a vehicle for conveying the moisture from the inside of the brick to the atmosphere. The moisture evaporated from the outside of the block by the sun's heat is immediately balanced by moisture drawn from within. This equilibrium is always maintained until drying is complete. During rainy weather the rain brings the skin to its maximum saturation, thus preventing moisture from reaching the interior of the block. The blocks, as the drying proceeds, contract and become denser and more resistant to breakage.

The Department of Mines manufactured, during the two seasons the Government plant was in operation, about 3000 tons of standard peat fuel. A large part of this was sold at a nominal price to householders in Ottawa and in the vicinity of the plant: 150 favourable opinions regarding the value of this fuel, from those who tried it, were collected by the Canadian Peat Society, and published in their Journal.

#### *Cost of manufacture.*

The results of the manufacturing operations conducted at Alfred indicate that, with strict business management, peat can be manufactured for \$1.70 per ton on the field. This figure includes all overhead charges, such as interest on investment, amortisation, etc. The machine employed depended on hand-dug peat, and, for an output of 30 tons per day, 14 men were required.

The newer plant which was erected on the Government bog, after its sale to private parties, was equipped with mechanical excavators, spreader, cable-way for transporting pulped peat to drying field, and many other innovations and improvements. Mr. E. V. Moore, who erected this plant, and operated it during the experimental stage, informed the author that the results obtained showed that peat could be manufactured at a cost considerably below that obtained with the Government plant. The conditions prevailing at the time war was declared unfortunately prevented the operation of this plant under thoroughly commercial conditions.



### *Properties of peat fuel.*

The following is a summary of the properties of peat manufactured as described:—

\* "Peat is a clean fuel to handle; has, as a rule, a very low ash content, and produces no soot or other deposit when burned in an ordinary cooking stove or open fireplace. The ash, moreover, is in a very finely divided condition, free from combustible matter, and can be easily removed from the stove or fireplace. Clinkers are not formed. On account of the ready manner in which peat fuel ignites, often a little paper or a few shavings are sufficient to start the fire. Peat fuel, on the other hand, is more bulky than coal and is of lower heating value per pound. The relation between anthracite coal and peat fuel as regards heating value per pound is 12,500: 7000 or 1.8: 1, that is, one pound of the average anthracite coal is equivalent in heating value to 1.8 pounds of peat fuel, containing 25% moisture. For a definite heating value, therefore, it is necessary to store 1.8 times the weight of the coal required in peat fuel. The volume occupied by the peat fuel, owing to its low specific gravity, will also be much larger than that of coal. One cubic foot of ordinary furnace anthracite coal weighs approximately 56 pounds, while one cubic foot of machine peat weighs about 27 pounds. The volume of peat required to equal coal of the above heating value will, therefore, be about 3.6 to 4 times that of the coal."

Peat is an excellent fuel for many of the domestic purposes for which coal is commonly used, and can, if certain changes are made in the design of house heating plants, be utilised in general for house heating. But peat possesses certain chemical characteristics which make it specially valuable as a source of nitrogen.

### *Nitrogen content of peat.*

The peat bogs so far examined in Canada all exhibit a high nitrogen content, and certain of them have an unusually high nitrogen content. Peat, therefore, becomes a most valuable source of nitrogen for the manufacture of ammonia and other nitrogen compounds.

In thirteen of the bogs so far examined in the province of Ontario, the nitrogen content of the peat reduced to a basis of 25% moisture is 1.3%. The total standard fuel content of these 13 bogs is 43,000,000 tons. The total nitrogen available is, consequently, 560,000 tons, and this quantity will give, with present day efficiency of recovery (as obtains with the by-product recovery producer) 1,800,000 tons of ammonium sulphate. This, of course, is simply one of the by-products. I shall refer to the process employed later.

### *Utilisation of peat fuel for industrial purposes.*

For power and metallurgical purposes it has been demonstrated that peat is an efficient and entirely satisfactory substitute for coal. Coal, of course, is much more convenient to handle, but the requirements of the one can be fulfilled by the other, provided the cost is the same.

The problems presented in the utilisation of peat fuel for the generation of a power gas, with or without by-product recovery, have been solved with entire satisfaction, and the use of peat for the generation of steam for power or other purposes is an assured economic fact. So far as the use of peat for metallurgical purposes is concerned, we can point to the by-product recovery peat coking plants in Germany and Russia.

It will be seen, therefore, that peat has been and can be used for very nearly all of the purposes for

which coal for over a century has been the only fuel used. But for certain reasons the plants erected at large expenditure to accomplish these ends are no longer in operation, although the products obtained have been eminently satisfactory. In regard to the large peat coke plants operated for some time in Germany, and which, for several years, have been closed down, the companies' statements show that the businesses were profitable, and the products met with a ready sale and at fairly high prices.

The entire trouble is to be found in the difficulty of supplying the requisite quantity of sufficiently dry peat to permit the plant to operate at full capacity throughout the year, and to supply a product (coke in this instance) which could be sold sufficiently cheaply on the open market to compete with coal coke. This is the great trouble with many of the larger undertakings of a more or less complex nature; the factors involved are not known, or else are not well enough understood, and, since the demand for the special products under consideration can or could be met by the products obtained from coal, the necessity for manufacturing the former product was not apparent. As coal becomes scarcer, and its price per ton rapidly increases, so will that of all the products derived from it, and then the above described peat-coke plants will come into their own. This time is not far off.

The treatment of peat fuel in gas producers, with or without by-product recovery, presents, however, a totally different aspect. In the case of a gas producer without by-product recovery, that fuel is used which experience shows is the cheapest, and the problem is simply one concerning the relative cost of peat fuel and other fuels obtainable.

In the case of by-product recovery plants, the situation is somewhat different. It is presumed that the principal aim of all by-product recovery producer-gas plants is the generation of a power or industrial gas with the maximum recovery of ammonia. Consequently, the nitrogen content of the fuel becomes one of the governing factors—in certain cases the governing factor.

This is a case where peat might successfully compete with coal, even though for a definite heating value peat be more expensive than coal. The profits resulting from the recovery of sulphate of ammonia increase rapidly with an increase in the nitrogen content of the fuel. It will at once be evident why certain of our peat bogs, in which the nitrogen content is over 2%, are so valuable for this kind of exploitation.

Plants of this description have been erected in Italy and Germany, for the purpose of generating electricity and recovering the nitrogen of the fuel as ammonia. One plant, in fact, near Venice, Italy, was established for the sole purpose of recovering the nitrogen content of the peat fuel. Two Italian plants are in operation to-day, even though at one of the plants the peat is of inferior quality as regards both its calorific energy and nitrogen content, and is exceedingly difficult to win.

The German plant, according to last reports, is out of commission, owing to the fact that the nitrogen content of the peat proved to be too low for profitable operation. Trouble was also experienced in the winning of the fuel. The tars obtained as a by-product at these plants were not considered to be of sufficient value for special treatment, so were burned under the boilers to help conserve fuel.

There are several bogs situated near certain of our industrial centres which are exceptionally high in nitrogen, of excellent heating value, and thoroughly satisfactory as regards quantity of fuel. If these bogs were exploited in the manner above outlined, and developed on a sound business basis, failure would be impossible. In fact, it has been

\* From address given by Dr. Eugene Haanel before Conservation Commission, Ottawa, November 27, 1917.

shown that, in special cases, a large profit on the investment could be made on the recovery and sale of the sulphate of ammonia alone, *i.e.*, assuming the power gas to be thrown away. Such installations do not, however, prove profitable when under a certain capacity, *e.g.*, 4000 horse power is the least capacity recommended.

#### *Generation of a power and industrial gas.*

The comparatively low heating value of peat fuel, together with its high water content, make it imperative that this class of fuel be utilised as close as possible to the source of its manufacture. Only when long hauls are obliterated and every possible economy is introduced in the manufacture and utilisation of the fuel, can peat for general purposes be employed in competition with coal. Since peat, as a rule, is so rich in nitrogen, every effort should be made so to utilise it that the highest possible recovery of the nitrogen could be realised. The profits derived from the by-products would appreciably decrease the cost of the power or power gas, if this were the principal object. A plant suitably situated with respect to a city or group of towns or villages could supply a gas which could be used for power, industrial, or domestic purposes, thus rendering those communities entirely independent of coal for heat for all purposes. The gas for various purposes could be transmitted through special mains to the towns or cities, and then supplied through the gas mains already in place, providing such arrangement could be made.

A large manufacturing and industrial area in South Staffordshire, England, is supplied with a power and industrial gas generated at a large Mond gas plant situated near a source of low grade coal. There is no reason why similar plants using peat should not be erected in this country, especially in those portions which are dependent on imported coal for heat (both industrial and domestic).

In regard to the utilisation of peat fuel for the generation of steam power, I shall only make the general statement that there are conditions and situations in this country which would be entirely favourable to the use of peat for this purpose.

#### *Peat as a source of oil and retort gas.*

The rapidly increasing demand for oils suitable for use in internal combustion engines, and the known scarcity of petroleum products in Canada, naturally prompt us to consider our own available sources of oils.

The principal source of oil other than the petroleum fields of Ontario exists in our oil shale deposits of New Brunswick, and, perhaps, those existing in other provinces of Canada. Certain of the lignites of the western provinces should in time become a valuable source of oils, and, to a certain extent, peat could be utilised for this purpose.

In the manufacture of peat coke, as conducted in Germany prior to the war, the following by-products were obtained from the distillation of the tar recovered:—Light oils, for either illuminating purposes or the manufacture of oil gas. Heavy oils, suitable for lubricating purposes. Paraffin, phenol, and asphalt. Tar water, containing ammonia, acetic acid, and methyl alcohol. When coked according to the Zeigler process 516 tons of peat containing C 35.3, H 3.4, N 0.7, S 0.1, O 28.4, ash 0.9, moisture 31.0%, and having a calorific value of 3792–3423 calories per kilo., or 7825–6161 B.Th.U. per lb., yielded 163.7 tons of peat coke, 25.8 tons of tar, and 269.0 tons of tar water. The 25.8 tons of tar yielded on distillation 11.6 tons of light oils, 3.9 tons of heavy oils, 1.8 tons of paraffin, 7.6 tons of phenol, and 0.8 ton of asphalt, a total of 4.5% of the weight of peat coked. The 269.0 tons of tar water yielded on further treatment 1.8 tons of methyl alcohol,

0.9 ton of ammonia, and 2.5 tons of acetic acid. The heating value of the gas produced was 322 B.Th.U. per cubic foot.

The above figures are taken from the Mines Branch Report: "Peat and Lignite: their Manufacture and Uses in Europe."

Attempts have also been made to retort peat for the sole purpose of generating illuminating gas, but they failed owing to the high cost of the peat used.

The utilisation of peat for the production of coke, illuminating gas, or for the recovery of oils alone, depends entirely on the quantity of peat fuel available, and the price at which it can be obtained. It is not a difficult matter, therefore, to decide whether or not the utilisation of peat for such purposes is feasible.

#### *The establishment of a peat industry.*

Many attempts have been made to start a peat industry in Canada, but for various reasons they have all resulted in failure. A flourishing peat industry must be established, despite past failures but we cannot afford to have any more such attempts terminate in this way.

The establishment of a peat industry along the lines suggested demands the support and sympathy of the various recognised engineering professions. In the first place, no attempt should be made to manufacture peat fuel without the aid of a well recognised peat engineer—one who is thoroughly familiar with the laying out of a bog for manufacturing purposes, and competent to design, erect, and operate the machinery required; and, secondly, no new processes or revolutionary improvements on well-known economic existing processes should be considered until the manufacture of peat fuel on a commercial scale according to a well-known and well-tried economic process has been securely established. Then, and only then, can attention be directed towards the improvement of existing processes, or the devising of new ones.

The erection of chemical, metallurgical, or power plants designed for the exclusive use of peat fuel, should never be attempted until specialists in the various lines involved are consulted, and the entire proposition considered from every possible angle. If such care is taken, failure will not result, and the erection of one successful plant will lead to the rapid erection of others.

Finally, the people of Canada should be educated to realise the necessity of utilising their own resources, even at some sacrifice in the beginning, instead of those obtained from some foreign source. In fact, this idea appears so important that I would even advocate Government legislation which would assist in, and insist upon, the utilisation of our own natural resources.

Received July 11, 1918.

## Newcastle Section.

### INFLUENCE OF A CORED DENDRITIC STRUCTURE IN ALLOYS.

BY O. SMALLEY.

(This J., July 15, 1918, 191–201 T.)

#### *Errata.*

Page 198 T, col. 1, line 27, for "nitric acid and cupric chloride" read "cupric chloride and nitric acid." Lines 30 and 31, for "picric acid and cupric chloride" read "cupric chloride and picric acid." Line 4, for "fig. 18" read "fig. 17."

Page 201 T, col. 1, line 6 from bottom, for "72 tons and 26 tons" read "26 and 72 ft.-lb respectively."



## Communications.

### APPLICATION OF THE POLENSKE METHOD TO THE ANALYSIS OF SOAPS AND FATTY ACIDS.

BY PERCIVAL J. FRYER, F.I.C.

No mention has apparently been made of the use of the Polenske method (measure of the insoluble volatile fatty acids) in the analysis of the insoluble mixed fatty acids from oils and fats. In the examination of soaps, as well as of commercial fatty acid mixtures, the original oils are not available; yet, in the former case especially, coconut and palm kernel oils have been very largely employed for the special properties which they confer on the soap.

The author has investigated the subject, and finds that, although the usual saponification is unnecessary (the volatile fatty acids distilling over on simply boiling with water), it is advisable to adopt the standard method, especially in view of the fact that the fatty acids from commercial soaps as well as industrial samples of fatty acids frequently contain proportions of neutral oils and fats.

In all cases examined of soaps made from coconut and palm kernel oils, the liberated fatty acids gave, on distillation, considerable proportions of soluble volatile acids, amounting to one-half to nine-tenths the normal Reichert-Meissl values. The figure was variable, according to the amount of water used to dissolve the soap and whether the fatty acids were subsequently washed.

It was found convenient to use the standard weight of material, *i.e.*, 5 grms., for the estimation, all the other details of the standard method being observed. (The weight of sodium hydroxide employed, *viz.*, 1 gm., was sufficient to neutralise the fatty acids and leave sufficient excess to ensure saponification of any neutral glycerides present.)

In the case of soap it is desirable—though not absolutely essential—that a uniform method of determining the fatty acids should be employed. The following is suggested: The percentage of fatty acids in the sample is ascertained, and a quantity of the soap taken equivalent to 10 grms. of the fatty acids (thus in case of a 60% soda soap 16.6 grms. is used; or with a 40% potash soap 25 grms.). Water is added to make the weight up to 200 grms., the mixture heated to complete solution, a few drops of methyl orange added, and then sufficient dilute (1 in 4) sulphuric acid to give a decided pink coloration. The solution is warmed till the fatty acids melt completely and

the aqueous liquid is quite clear, transferred to a separating funnel, the aqueous layer run off, the fatty acids shaken with four successive quantities of 100 c.c. of boiling water, and the clear acids filtered through a thick dry filter paper.

The standard Polenske method is carried out as follows: 5 grms. of the fatty acids is weighed into the standard (300 c.c.) flask, 2 c.c. of 50% caustic soda added, and the flask heated, with constant agitation, till the liquid suddenly clarifies. It is allowed to cool to about 100°, and 100 c.c. of boiling water is added and the soap dissolved. After addition of 0.1 gm. of powdered pumice the soap is decomposed with 40 c.c. of 6% sulphuric acid, and the flask connected with the standard condenser, heated gently till the fatty acids are completely melted, and 110 c.c. distilled in 19–21 minutes. The distillate is placed in water at 15° for 15 minutes, and is then filtered through a wet filter paper. The condenser and receiver are washed through with 18 c.c. of cold water, which is used to wash the fatty acids on the filter, and the condenser is then cleansed with four washings of 10 c.c. of neutral alcohol, which are run into receiver and poured over the filter and collected in a titrating flask, the mixed alcohol solutions being then titrated with N/10 alkali. A blank test is unnecessary, provided the alcohol used is carefully neutralised.

The author obtained the following average figures for the "insoluble" volatile fatty acids of coconut and palm kernel oils: Coconut 17.3%, palm kernel 10.5%.

The method of Burnett and Revis (Analyst, 1913, 38, 255; this J., 1913, 704) is also equally applicable for the discrimination of coconut and palm kernel fatty acids in mixtures of these with other acids. In this case N/10 baryta is used for the titration.

### THEORY OF THE COAGULATION OF HEVEA LATEX.

In a paper bearing the above title, by M. Barrow-cliff, published in this Journal, February 15, 1918, p. 48 T, credit was given to Whitby for the first suggestion (in 1912) that natural coagulation of latex is brought about by an enzyme. Mr. Barrow-cliff now writes that his attention has been drawn to the fact that the same suggestion had previously been made in 1908 by Dr. Spence, who, in a communication read before the Rubber Congress in London, entitled "The Practical Significance of Recent Advances in the Bio-Chemistry of the Latex of Rubber-Producing Plants," stated that "the spontaneous coagulation of most latices and the acidity which gradually develops in fresh latex on standing are produced, therefore, largely by the enzymes present in the latex."

## Canadian Section.

Meeting held at Ottawa, Ont., on May 22nd, 1918.

MR. T. H. WARDLEWORTH IN THE CHAIR.

## THE DETERMINATION OF MOISTURE IN WHEAT.

BY DR. F. T. BIRCHARD.

There is at present no standard method for the determination of moisture in wheat, and as a result considerable confusion exists. Our investigations prove conclusively that the ordinary methods described in the literature and generally adopted and practised are not adequate. This difficulty has been recognised by a number of workers. Thus Barnes and Grove\* invariably report the moisture in wheat under two headings, viz., that which is obtained by heating to 70° C., and that which is found by heating to 100° C. Leavitt, while noting that a variation of 1° C. makes a marked difference in the moisture results obtained on wheat, claims that all the moisture could be driven out by heating to 100° C. for 4½–5 hours in an open-air oven. He states that if the heating is prolonged beyond this time an increase in weight occurs, but our experiments show conclusively that the increase is apparent only, since if a sample is put back into the oven for a sufficient length of time and re-heated at the same temperature a further decrease is noted. Swanson† also finds that if the ground grains are used, seven hours' heating in a water oven is sufficient, and that 40 hours is required in the case of whole grains. This also is not in accordance with our results, since again we found that if the sample is heated for a further period and re-weighed an additional decrease in weight is obtained.

Budal‡ believed that constant weight could be obtained by heating in an open oven to 170° C. for about three-quarters of an hour, and that the loss obtained represented the true moisture content. Duvel§ also states that the true amount of moisture can be obtained by heating the whole grains to constant weight at 107° C.

Without referring further to the literature, it may be stated that we have not been able to find any completely satisfactory method for this operation, the difficulty being that the loss in weight on drying depends chiefly on the temperature employed, but also on the length of time the grain is heated. Thus, for example, when wheat is heated in an ordinary drying oven or in a vacuum chamber continuously for many months, we have been unable to determine the point at which no further loss in weight takes place. It is true that after a certain time the losses are very small, but when the time of heating is prolonged the total effect is considerable. Also by raising the temperature slightly more loss occurs, and although we have employed a large range of temperatures, no definite end point could be found, as reported by other observers. Occasionally we also noted a slight increase in weight, providing the heating was of too short duration, but if prolonged further an additional decrease in weight is always to be noted, so that it is probable that while the figure obtained by heating until a slight increase in weight is noted, approximates to the true value, this cannot be regarded as the absolute amount of moisture present, since further heating in our

experiments gave off more than an additional 1% when the heating was prolonged for a sufficient length of time. Some of the details of these experiments will be referred to later.

The method commonly employed in commercial practice is known as the Brown-Duvel method, and consists in heating 100 grams of wheat in a flask of oil to 180° C., measuring the volume of water distilled over in a graduated tube, and regarding this as the true amount of moisture in grain.

Although no account appears to have been published describing the manner of standardising the Brown-Duvel method, I was personally informed by Dr. Duvel that the wheat was simply heated in a water oven to constant weight, and then the arbitrary conditions necessary for obtaining the equivalent amount of water with the apparatus worked out. But since it is not possible to heat wheat at any temperature to constant weight, the Brown-Duvel apparatus cannot be regarded as giving absolute results. Since, however, we have shown that this apparatus can give comparable results which are sufficiently accurate for all commercial purposes, and since also this method has been officially adopted by the United States Department of Agriculture and is very largely in use both in the United States and in Canada, it would seem advisable that this method should be officially adopted in Canada.

It will only be necessary to consider here a few of the actual experimental results obtained.

Table I. shows the impossibility of obtaining constant weight by heating crushed wheat in the vacuum oven for 40 hours, as reported in the literature. Table II. illustrates the same results using whole grains instead of crushed wheat, the experiment being conducted for 60 days. Table III. shows the effect of heating whole grains of wheat in the Abderhalden apparatus at various temperatures, and emphasises the importance of these factors and the impossibility of obtaining constant weight no matter how long the heating be continued. Table IV. shows the impossibility of drying whole wheat grains to constant weight in the electric air oven at a temperature of 104° or at 109° C. Table V. shows the effect of heating flour and starch in a vacuum as compared with whole grains of wheat.

TABLE I

Crushed wheat heated in vacuum oven.

Temperature 98° C.

4 hrs.	5 hrs.	7 hrs.	10 hrs.	16 hrs.	23 hrs.	35 hrs.	49 hrs.
%	%	%	%	%	%	%	%
10.67	10.83	11.14	11.42	11.75	11.89	12.07	12.19
11.13	11.27	11.56	11.79	12.02	12.19	12.33	12.42
10.31	10.41	10.69	10.90	11.11	11.26	11.38	11.45
11.93	12.02	12.31	12.50	12.70	12.85	12.95	13.01
10.09	10.24	10.52	10.75	11.00	11.17	11.30	11.39
9.73	9.91	10.21	10.49	10.80	10.98	11.16	11.28

Temperature 99.7° C.

19 hrs.	43 hrs.	48 hrs.	74 hrs.
%	%	%	%
10.26	10.43	10.46	10.53
11.39	11.55	11.60	11.66

A number of samples of ground wheat were dried in a vacuum oven (26 in. vacuum). The first series of experiments was conducted at a temperature of 98° C., and it was found that the samples were still losing weight when the experiment was discontinued at the end of 49 hours. In the second series at 99.7°, samples were also losing weight when the experiment was discontinued at the end of 74 hours.

\* Agr. Research Inst., Pusa, 4 [6].

† Kansas State Agr. Bull. No. 177.

‡ Z. ges. Getreidewesen, Mar., 1914.

§ U.S. Dept. Agr. Bull. 99, 1907.



The following results were obtained using an ordinary double-walled water oven, the inner wall of which was completely surrounded by boiling water. The temperature of the interior was 98° C. The wheat was weighed into aluminium friction-top dishes.

TABLE II.

*Whole wheat heated in water oven.*

Time of drying, hours	Moisture, %
40	12·81
64	13·22
88	13·45
114	13·52
162	13·66
208	13·70
648	13·94
840	14·06
1032	14·07
1224	14·11
1416	14·21

Still losing weight when experiment discontinued.

TABLE III.

*Wheat grains heated in Abderhalden apparatus.*

Time of heating, hours	Source of heat, boiling	Moisture, %
24	CHCl <sub>3</sub> (61°)	9·44
24	CCl <sub>4</sub> (76°)	11·21
56	water	13·54
53	toluol (109°)	13·80
144	"	14·56
564	"	14·74
72	xylol (135°)	15·12
216	"	15·59
360	"	15·75

Still losing weight when experiment discontinued.

Before introducing toluol and xylol the sample had already been dried for 24 hours at the temperature of boiling chloroform, carbon tetrachloride, and water.

This demonstrates clearly the importance of the temperature factor, and shows that each temperature increase gives rise to a further loss in weight.

TABLE IV.

*Drying whole wheat grains in electric air oven.*

Temperature 103°—105° C.		Temperature 108°—110° C.	
Days	Moisture, %	Days	Moisture, %
8	14·24	8	13·61
12	14·27	12	13·94
15	14·28	15	13·91
18	14·34	18	14·08
25	14·44	25	14·18
33	14·54	33	14·39
43	14·68	43	14·52
51	14·79	51	14·61
59	14·80	59	14·71

*Comparison of drying of flour and starch by Abderhalden method.*

These experiments were made in a tightly fitting Abderhalden apparatus which held the vacuum exceedingly well. The apparatus was pumped out to about 24 in. vacuum each day. Flour and starch in weighing bottles fitted with ground stoppers to prevent loss, or absorption of water during weighing.

TABLE V.

Flour			Starch		
Time of heating	Source of heat	% Moisture	Time of heating	Source of heat	% Moisture
hrs.	Boiling	%	hrs.	Boiling	%
44	CHCl <sub>3</sub>	12·28	144	CHCl <sub>3</sub>	11·02
138	"	13·26	138	"	12·04
304	"	13·64	304	"	12·44
496	"	13·81	496	"	12·64
664	"	13·92	664	"	12·78
856	"	14·03	856	"	12·89
1160	"	14·07	1160	"	12·95
1544	"	14·07	1544	"	12·97
168	CCl <sub>4</sub>	14·09	168	CCl <sub>4</sub>	12·95
672	"	14·16	504	Water	13·05
1006	"	14·16	838	"	13·03
1396	"	14·16	1228	"	13·04
260	Water	14·22			

These results show that for flour and starch constant weight can be obtained for each temperature, and that while increasing temperature gives rise to slight decrease in weight, this decrease is exceedingly small and the weight may be considered as practically constant.

The following experiments were conducted in order to determine the degree of accuracy on the Brown-Duvel apparatus: 1000 grms. of wheat, which by the Duvel machine showed a moisture content of 16·6%, was dried in stages at a comparatively low temperature (about 80° C.) in the Freas electric oven. At the end of each period in the oven it was cooled in the desiccator, weighed, and a Duvel test applied to the dried wheat. This was continued until the wheat was quite dry. In the first series of tests the loss on drying at 80° was 45·5 grms., while the loss indicated by the Brown-Duvel method was 47·7 grms. In the next case a difference of 1·1 gm. of water was found between the methods; in the third a difference of 1 gm.; in the fourth a difference of 0·1 gm. In the second series of tests (where 500 grms. of wheat only was employed) the differences were smaller, namely, 0·02, 0·03, 0·05, and 0·03 gm. These differences when calculated as percentages of the moisture in the wheat are exceedingly small; thus in the case of the greatest difference found this would represent only 0·3 c.c. on a sample of wheat of normal moisture content (13%), and if the average of the results were taken a difference of only 0·03 c.c., quite a negligible quantity. It is also shown that the Brown-Duvel apparatus is equally accurate whether it be dealing with wheat of high moisture or with wheat of low moisture content.

In a third experiment a large sample of dry wheat was well mixed. From the sample two lots of 80 grms. were weighed quickly and each placed in a Duvel distillation flask. To each of these 80 gm. lots 20 c.c. of water was added from a burette and the flask sealed up, shaken from time to time, and allowed to stand for about 20 hours. The flasks were then unsealed, 150 c.c. of oil added to each, and a Duvel test made on each in the usual manner. The moisture on dry wheat according to Brown-Duvel was 1·9%, corresponding to 1·5 c.c. in the 80-gm. sample, or a total of 21·5 c.c. in the sample tested. The amounts actually obtained in duplicate experiments were 21·6 c.c. and 21·7 c.c. In a similar manner 85 grms. of wheat was employed with the addition of 15 c.c. of water. In the first case a total of 16·6 c.c. of water should be present according to the Brown-Duvel test, while 16·8 c.c. and 16·7 c.c. were obtained in duplicate experiments. These results would appear to indicate clearly that the Brown-Duvel apparatus applies equally to wheat of low or high moisture content, and that reliable comparative results may be obtained therewith.

For the convenience of those not wishing to use the Brown-Duvel apparatus, we have determined a

series of temperatures and the corresponding length of time which must be employed to give results identical with those obtained with the Brown-Duvel apparatus.

TABLE VI.

	Temperature, °C.	Time of heating necessary, hours approximately
Freas electric oven ...	100	54
	105	41
	110	28
	115	20½
	120	18
	125	10½
	130	8
	160	1½
Water oven ...	97	93
Abderhalden apparatus, vacuum 29 in....	99	52

All these experiments were repeated on wheats showing different characteristics, such as plump and thin grains, high and low moisture content, and in every case the results were the same. All the experiments, however, were confined to hard spring wheat, and whether any alteration would be necessary in the case of other varieties has not yet been determined.

Considerable variations occur in the temperature of the Freas electric oven at different points. This has also been observed by other workers. Thus not only do the different shelves show variations of several degrees, but different portions of the same shelf also show considerable differences. In the experiment described the samples were all grouped closely round the thermometers, and in addition a second thermometer was placed close to the sample on the shelf.

During the very prolonged heating of the wheat a gradual darkening in colour took place, which very slowly increased with the time of heating. At no point, however, can it be determined just where this discoloration first occurs; it is so very slight and so very gradual, some grains appearing to be more susceptible than others. A certain amount of chemical decomposition does occur even at the temperature of the water oven (approximately 98°), but as to the exact nature and extent of these changes little can be said at the present time.

In connection with the great difficulty experienced in drying wheat to constant weight, the remarks of Ostwald in "Foundations of Physical Chemistry" are not without interest. Speaking of colloidal precipitates, he says: "We have to remember that the vapour pressure of the exceedingly thin films of the moistening liquid is much less than that of the same liquid in the free state. The drying temperature must therefore be raised far beyond the boiling point of the liquid in order to get rid of the last traces of the latter, and the finer the powder the higher the temperature must be. Colloidal substances require the highest temperatures."

#### Summary.

It is not possible to determine the amount of moisture in grain by simply drying in an open oven or vacuum chamber at a definite temperature to constant weight or until an increase in weight takes place. A definite loss of weight occurs corresponding to the temperature employed and the length of time of heating, and increasing either of these factors leads to a still further loss of weight of the sample. Increase of temperature gives rise to an increased loss in weight that cannot be obtained at any lower temperature, no matter how long the heating be continued.

It is proposed that the Brown-Duvel method be adopted as the official method in commercial practice as the standard, and for those not employing this

apparatus other standard methods are proposed which give identical results. When the Brown-Duvel method is properly operated, the variation does not amount to more than 0.2%, and if the average of a number of determinations be taken, not more than 0.1%. For practical purposes this amount is negligible. Determinations made with other apparatus on the market, similar in principle, but not identical with the Brown-Duvel, give very different results, and experience shows that these are not reliable and bear no definite relation to those obtained with the Brown-Duvel apparatus.

## London Section.

Meeting held at Burlington House on June 3rd, 1918.

DR. CHARLES A KEANE IN THE CHAIR.

### THE ESTIMATION OF PHENOL IN CRESYLIC ACID.

BY J. J. FOX, D.SC., F.I.C., AND M. F. BARKER, B.SC., A.I.C.

A short time ago we published an account of a method for the determination of phenol in commercial cresylic acid (this J., 1917, 842) which had for its object the determination of the proportion of phenol left after stripping out the main quantity. For many purposes, however, it is sufficient to ascertain whether the cresylic acid contains more than a small amount of residual phenol, say 5%. While our process is comparatively short, it is perhaps not rapid enough to serve as a method of estimation for the class of cresylic acid referred to. We have therefore devised the short method described below.

We take this opportunity of making a few additions to the description of our original method (*loc. cit.*) in order to clear up one or two difficulties which may be found in practice. In the process as described it has not been made quite clear that a sufficient quantity of ether must be used to dissolve all the tar acids and so facilitate the subsequent washing with sodium sulphate solution. Moreover, benzene may be used in place of ether for getting rid of bases and tar oils, and this will no doubt be found more convenient in works; in addition to this it is rather easier to operate with benzene than with ether when strong soda is used to dissolve the cresylic acid. It should be noted, however, that benzene is obstinately retained by the solution of tar acids in 20% sodium hydroxide and therefore the soda solution must be heated sufficiently strongly to expel all the benzene before acidification. Three treatments with benzene are required to remove neutral oils and tar bases, the quantity of benzene used depending upon the degree of impurity of the cresylic acid. After acidifying the soda solution to liberate the tar acids, the aqueous layer may be extracted with benzene instead of with ether, using three portions of benzene of 30–50 c.c. each. The whole of the tar acids are dissolved in benzene, to which the benzene extract of the aqueous acid layer is added. The washing with sodium sulphate solution and drying with calcium chloride are carried out as described (*loc. cit.*). When benzene is employed in place of ether it is necessary to heat the tar acids to 140° C., or even higher, before all the benzene can be considered to be removed.

The neutral oils and bases can also be conveniently removed by treating the cresylic acid with a



good excess of 20% sodium hydroxide—3 volumes at least—and distilling with steam until no more oil passes over with the distillate. If more dilute soda (10%) is employed, a greater excess is desirable in order to prevent tar acids (notably *o*-cresol) being carried over with the steam.

It is to be noted that when phenols are dissolved in the theoretical quantity of sodium hydroxide and subjected to distillation with steam, some of the phenol is carried over with the steam, the amount depending upon the dilution of the soda and on the acidic strength of the particular phenol under consideration. Hence, it is necessary to dissolve the tar acids in a good excess of soda to press back the hydrolytic dissociation.

In the examination for phenol of disinfecting fluids of the so-called saponified cresol type, which contain resin or other soap, tar acids, and tar oils, the extraction of tar oils by benzene or ether is usually impracticable because the fluid frequently dissolves completely in the benzene or ether. Steam distillation is also out of the question because of the frothing. The procedure adopted by us is to take 200 to 400 c.c. of the disinfecting fluid, acidify it with sulphuric acid (1 in 1 or 1 in 2), and collect the mixture of fatty acid, resin, tar acid, and tar oil. Only traces of phenol are left in the acid layer if tar oils are present in appreciable quantity, as is generally the case with disinfecting fluids except those of the "lysol" type. The mixture of fatty and tar acids, etc., is transferred to a Würtz distilling flask and distilled up to 220° C., when all the phenol and most of the cresols distil over, together with some or all of the tar oils. It is very rarely that decomposition or volatilisation of fatty acids occurs at this temperature. The mixture of tar acids and oils is treated with at least twice the volume of 20% sodium hydroxide, and the neutral oils are removed either by distillation with steam, or by extraction with a suitable solvent (benzene). The soda solution is then acidified and the tar acids examined for phenol as usual.

An interesting paper dealing with phenol in cresylic acid has been published by Knight, Lincoln, Formanek, and Follett (J. Ind. Eng. Chem., 1918, 10, 9; this J., 1918, 85 A), in which a method is described whereby reference to graphs is claimed to be obviated. These workers also make use of *o*-cresol, and there is no doubt the method eliminates the possibility of estimating some of the *o*-cresol as phenol. Where large numbers of samples have to be dealt with, the method is too lengthy for general use. Our method of utilising *o*-cresol is rather different from that of Knight, Lincoln, Formanek, and Follett. We endeavour to procure a distillate up to 195° C. which shall contain *o*-cresol as the main constituent of the cresols which distil over up to this point. Having regard to the close agreement between the solidifying points of prepared mixtures of the pure cresols with those obtained with representative distillates from commercial cresylic acid (see table of solidifying points, this J., 1917, 842) it seems probable that the composition of the distillate up to 195° is, in these cases, as uniform as can be expected as regards the cresols. What the actual composition of this distillate may be is not known with any certainty, but two of the constituents can be determined, namely the phenol by our process and *m*-cresol by Raschig's nitration method. This leaves *o*- and *p*-cresol undetermined. From certain data in our possession it may be found possible to determine these two cresols, at least approximately, but in the present circumstances there is no opportunity of dealing with the matter adequately.

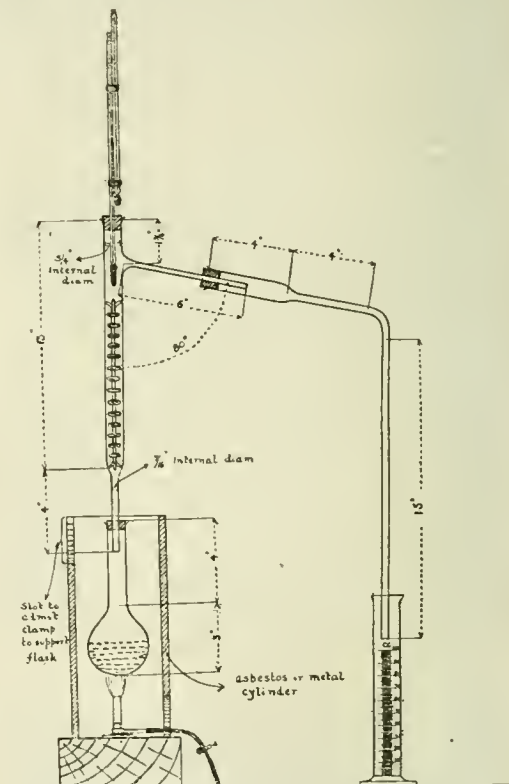
There is one point in connection with all methods requiring specific gravity and solidifying point determinations of phenols to which we desire to call particular attention. The phenols must be dried before the determinations are made. Distillation

alone is insufficient to remove all the water, and the influence of small quantities of water, particularly on the solidifying points, is very marked. It is not unlikely that the different melting and solidifying points of the cresols recorded by various workers are largely due to the presence of minute quantities of water.

While this paper was being written, a communication on the subject by G. H. Sharples appeared (this J., 1918, 109 T). This paper clearly brings out the effect of small quantities of water on the solidifying point and the specific gravity.\* Sharples recommends the addition of *o*-cresol at an early stage to ensure the presence of sufficient *o*-cresol and also to avoid one of the fractionations. This modification certainly shortens the process considerably.

*Short method for the examination of cresylic acid for phenol.* For this test the pure tar acids are required. Neutral tar oils, naphthalene, and bases, if present in more than traces, must be removed by steam distillation of the solution in sodium hydroxide, or by treatment of the solution by means of benzene or ether. The recovery of the tar acids from the soda solution is effected by acidification with sulphuric acid, care being taken to extract the acid layer so as to recover any phenol present in the sodium sulphate solution.

The tar acids are dried by means of ignited calcium chloride and distilled in the distillation apparatus shown in the figure.



This distillation apparatus has the dimensions shown in the figure and is the most convenient form to use. If long-necked flasks of the dimensions shown are not available, then a short-necked flask with a body of the same size may be used. The thermometer should be accurately calibrated

\* Paterno and Ampola (Gazz. Chim. Ital., 1897, 27, 523) have shown that the effect of 0.5% of water is to lower the solidifying point of phenol about 2° C. Similar results are found with the cresols.

between 90° and 250° C. and should have divisions of at least 0.5°.

The apparatus is best supported by three clamps, one attached to the flask and inserted through the slot in the cylinder for flame protection as indicated, a second about halfway up the fractionating column, and the third to support the condenser and fixed about midway between the top of the receiver (100 c.c. graduated cylinder) and the bend.

Rubber should not be used in the construction of the apparatus, as hot cresols gradually dissolve rubber. Ordinary cork stoppers are satisfactory but they must be renewed frequently.

100 c.c. of the thoroughly dried tar acids is distilled at the rate of 7 c.c. per minute, and the fraction (A) boiling up to 210° C. is collected. This fraction is redistilled at the same rate, and the portion (B) boiling up to 202° C. is collected. If this does not exceed 5%, the sample is satisfactory (*i.e.*, does not contain more than 5% of phenol) and no further test is required. When more than 5% is obtained, the fraction (B) is redistilled and the portion boiling up to and including 195° C. is collected separately and tested for phenol by taking its specific gravity at 15.5° C. and by means of bromine water.

**Specific gravity.** This is best taken in a pycnometer, or more expeditiously by means of a Westphal balance if enough distillate is available. The specific gravity may be taken at any convenient temperature and the necessary correction applied from the following table to bring the result to 15.5° C. The correction for specific gravity is given by the formula  $(t-15.5) \times 0.00055$ , where *t* is the temperature of the experiment. The correction factor is the mean of the actual values from large numbers of distillates. Very few distillates give results varying by more than 0.00002 from this mean factor.

Correction for specific gravity

°C.	Correction	°C.	Correction
10	-0.0030	20	+0.0025
12	-0.0019	22	+0.0036
14	-0.0008	24	+0.0047
15	-0.0003	26	+0.0058
16	+0.0003	28	+0.0069
18	+0.0014	30	+0.0080

**Bromine water test.** For this test a freshly prepared saturated aqueous solution of bromine is required. 0.1 to 0.2 c.c. of tar acid distillate is placed in a glass cylinder of about 100 c.c. capacity. About 10 c.c. of water is added, and then two or three drops of hydrochloric acid (sp. gr. 1.15). The mixture is well shaken and sufficient bromine water is added to bring the total volume to about 100 c.c. The cylinder is again vigorously shaken and the precipitate allowed to settle. If 5% of phenol or more was present in the original sample, a voluminous light precipitate is formed, which is better observed after the liquid has been allowed to settle for about 15 minutes. The cresols alone produce only a dark oil or a heavy granular precipitate which settles quickly. If the bromine precipitate is examined under the microscope (magnification 100) the phenol precipitate is seen to consist of small needles, and is quite distinct in appearance from the cresol compounds.

It is desirable to make up a few mixtures of phenol and cresols so as to become acquainted with the nature of the precipitate. With a little practice the phenol precipitate is unmistakable.\*

If the specific gravity at 15.5° C. exceeds 1.048 and a decided phenol precipitate is obtained with bromine water, then the proportion of phenol must be determined by the process previously described (this J., 1917, 843). In applying this process corrections for barometric pressure and emergent stem should be made as in the shorter method here described. If the specific gravity is 1.048 or less and the bromine precipitate is not characteristic of tribromophenol, the sample may in general be considered to contain less than 5% of phenol.

Corrections for the portion of the stem of the thermometer not immersed in the vapour and for barometric pressure are calculated from the following formulae.

TABLE I.

Correction for emergent stem.  $0.000143(T-t)N$ .

*T* is observed boiling point.

*t* is the temperature of the middle point of the portion of the stem outside the vapour.

*N* is the number of degrees of the stem outside the vapour.

Table of corrections to be added for values of (*T-t*) and *N*.

--N--

T-t	50	55	60	65	70	75	80	85	90	95	100
150	1.07	1.18	1.29	1.39	1.50	1.60	1.72	1.81	1.93	2.03	2.15
155	1.10	1.22	1.33	1.43	1.55	1.65	1.78	1.88	2.00	2.10	2.22
160	1.14	1.26	1.37	1.49	1.60	1.71	1.83	1.94	2.05	2.16	2.29
165	1.18	1.30	1.41	1.53	1.65	1.76	1.88	1.99	2.11	2.23	2.36
170	1.22	1.33	1.46	1.57	1.70	1.82	1.94	2.05	2.17	2.30	2.43
175	1.25	1.37	1.50	1.63	1.75	1.87	2.00	2.12	2.24	2.37	2.50
180	1.29	1.42	1.54	1.67	1.80	1.92	2.05	2.18	2.31	2.44	2.57
185	1.33	1.45	1.58	1.71	1.85	1.97	2.11	2.24	2.38	2.50	2.64
190	1.36	1.49	1.63	1.76	1.90	2.03	2.17	2.30	2.45	2.57	2.72
195	1.39	1.53	1.67	1.80	1.95	2.08	2.22	2.36	2.50	2.64	2.79
200	1.43	1.57	1.72	1.86	2.00	2.14	2.29	2.43	2.54	2.71	2.86

\* See Sharples, this J., 1918, 109 T.



*Table II. Correction for barometric pressure.* The corrections to be added or deducted for variations from the normal pressure of 760 mm. of mercury, are shown in the table. P is observed barometric pressure. This table is based on actual data obtained round about 760 mm. pressure.

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TABLE II.

P†	Correction	P	Correction	P	Correction	P	Correction
770	— 0.50	760	0.0	750	+ 0.50	740	+ 1.00
769	— 0.45	759	+ 0.05	749	+ 0.55	739	+ 1.05
768	— 0.40	758	+ 0.10	748	+ 0.60	738	+ 1.10
767	— 0.35	757	+ 0.15	747	+ 0.65	737	+ 1.15
766	— 0.30	756	+ 0.20	746	+ 0.70	736	+ 1.20
765	— 0.25	755	+ 0.25	745	+ 0.75	735	+ 1.25
764	— 0.20	754	+ 0.30	744	+ 0.80	734	+ 1.30
763	— 0.15	753	+ 0.35	743	+ 0.85	733	+ 1.35
762	— 0.10	752	+ 0.40	742	+ 0.90	732	+ 1.40
761	— 0.05	751	+ 0.45	741	+ 0.95	731	+ 1.45

† To convert inches into millimetres, multiply by 25.4.

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## THE SOLIDIFYING AND BOILING POINTS OF PHENOL AND THE CRESOLS.

BY J. J. FOX, D.SC., F.I.C., AND M. F. BARKER, B.SC., A.I.C.

Some time ago the authors were called upon to devise a method for the detection and estimation of small quantities of phenol in cresylic acid. One of the first things necessary in the investigation of mixtures of closely related substances such as phenol and the cresols, is to possess a complete record of the variation of the physical properties of such mixtures with the composition. The complete curves for mixtures of phenol and the cresols have not been recorded so far as the authors are aware. A large amount of work on mixtures of phenol and cresols has been done by Weiss (see this *J.*, 1913, 692, and also Weiss and Downs, this *J.*, 1917, 863), and tables are referred to in Lunge's "Coal Tar and Ammonia," but the information was insufficient for our purpose.

The results we have obtained with the purest cresols available in the large quantities needed for this class of work are important, whether they are considered from the point of view of the valuation of carbolic acid or on theoretical grounds.

In judging of the purity of the materials used, we have considered those samples as the best which furnished results for specific gravity and boiling point most nearly in agreement with the values recorded by W. H. Perkin (*J. Chem. Soc.*, 1896, 69, 1182), because Perkin's values can only be obtained after suitable purification and drying. With substances giving Perkin's constants the highest solidifying or melting points are obtained.

The constants that we have obtained for cresols and phenols have been given in an earlier paper

(this J., 1917, 842). Where necessary we have utilised Perkin's data for the specific gravity of phenol at various temperatures.

To obtain materials having the constants specified the following methods of purification were adopted.

*Phenol.* "Absolute" phenol of commerce was distilled, the first fractions containing a little water being rejected. At 182.5° C. the temperature remained constant and the middle fraction was collected for use. This fraction was further dried by keeping it in the liquid state over ignited calcium chloride for several hours. The resulting phenol had a freezing point of 40.5° C. when freshly prepared and dry.

*o-Cresol.* Commercially pure *o*-cresol was dissolved in excess of 20% sodium hydroxide, diluted with water, and the liquid distilled with steam to remove naphthalene, tar oils, and bases. The resulting soda solution was acidified with sulphuric acid, and the cresol layer removed and washed with water until free from sulphuric acid. The water was separated as completely as possible and the cresol was distilled. The first fraction distilling over was rejected, the portion boiling between 191° and 192° C. being collected. This fraction was dried over ignited calcium chloride and redistilled, the middle portion of constant boiling point being used. It had a freezing point of 30.0° C. and sp. gr. at 15.5° of 1.0512. By collecting the middle 50% alone, we had no difficulty in preparing *o*-cresol with the solidifying point mentioned.

*m-Cresol.* A supply of Kahlbaum's *m*-cresol was available and this was found satisfactory when redistilled. Larger quantities were obtained by Raschig's process (Ger. Pat. 114,975) from commercial *m*-cresol. The redistilled *m*-cresol was heated in a large flask with 4 times the weight of strong sulphuric acid (as described by Raschig) at 100° C. for about one hour. The acid was diluted with water, steam was passed in and the liquid was heated until a temperature of 125°–127° C. was reached. The heating of the flask was regulated to maintain this temperature, at which decomposition of the *m*-cresol sulphonic acid results, and the *m*-cresol passes over with the steam. It is sometimes coloured yellow by this treatment, but although the colour persists it does not in any way affect the physical properties of the *m*-cresol. The cresol was purified by redistillation after drying over calcium chloride, the fraction boiling between 202.0° and 202.5° C. being collected and redistilled. Our preparation, as well as Kahlbaum's (when dried and redistilled), solidified at +2.4° C. and had a sp. gr. of 1.0387 at 15.5° C. The success of this method of purifying *m*-cresol depends on preventing the temperature to which the sulphonic acid is heated with steam rising much above 125° C. If the *m*-cresol is not thoroughly dried it will not solidify. Slight variations in the boiling point were observed with different specimens.

*p-Cresol.* A large quantity of commercially pure *p*-cresol, freed from tar oils, etc., was repeatedly liquefied, frozen, and drained on gauze from adherent liquid. The solidified portion was then liquefied and distilled, the first portion of the distillate being rejected. The dried cresol had sp. gr. 1.0288 at 15.5° C. and froze at 36.0° C. The purification of the cresols leads to considerable waste of material, hence it is desirable to operate on several kilos.

The constants found by us differ a little from those given by Knight, Lincoln, Formanek, and Follett (J. Ind. Eng. Chem., 1918, 10, 10) the freezing points for *o*- and *p*-cresol being higher than those given by these workers, but we prefer them for the reasons mentioned above. It might be added that *o*-cresol prepared from pure *o*-toluidine was never in our experience so good as the *o*-cresol

obtained by working up several kilos. of the best commercial *o*-cresol available.

Specific gravities were taken in a capped silica Sprengel tube holding about 30 c.c. and also in a pycnometer holding 50 c.c.

The freezing points were determined in an apparatus of the Beckmann type as used for molecular weight determinations. Open-scale thermometers divided in tenths of a degree were used. The mercury thread was arranged so that one degree at most was visible above the top of the freezing point tube. This was secured by changing the thermometer according to the temperature to be read. The bulb of the thermometer was always well immersed in the liquid and the same quantity of liquid, as nearly as possible, was used for each determination. The freezing point tube was surrounded by an air bath, at the bottom of which some dry calcium chloride was placed to keep the air dry. This also stopped the formation of mist and so prevented the view of the contents of the vessel being obscured. The cresol mixture was stirred by means of a spiral wire stirrer, and moisture was prevented from gaining access to the freezing point tube by attaching a calcium chloride drying tube to the side tube of the apparatus. The air bath was surrounded by an outer jacket, containing water for the freezing point temperatures of 20° C. and higher, ice and water for temperatures of 10° C. and lower, and salt and ice for the lowest temperatures. The contents of the outer jacket were stirred with a stout wire stirrer. As excessive supercooling must be avoided, the temperature of the outer bath should be as near as possible to that of the freezing point expected, generally within 2° or 3° C. At the freezing point the temperature rises to a maximum and remains constant for some time. There is no difficulty in obtaining readings of the freezing point accurate to less than 0.1° C. It might of course be possible to obtain greater accuracy by the use of other means of recording the temperature, but the results actually obtained are sufficient for use. In the cases where considerable supercooling might occur, accurate freezing points could only be obtained by keeping the temperature of the outer jacket as close as possible to the freezing point, and also by adding a crystal of one of the constituents.

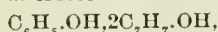
The apparatus used for the boiling point determinations consisted of a flask practically identical with that described by Northall-Laurie (Analyst, 1915, 40, 386) fitted to a condenser. By means of this device the condensed liquid flows back into the boiling liquid, so that by careful regulation of the heating to prevent violent ebullition, the composition of the boiling liquid remains practically unchanged. For determining boiling points above or below atmospheric pressure, the top of the condenser was attached to a mercury manometer and air reservoir, which contained about 20 litres of air. This acted as a "cushion," and connection with the condenser was made by a T-piece and suitable taps, so that any part of the apparatus could be isolated. Air was pumped out of or forced into the reservoir and the reservoir tap was then turned on slowly to equalise the pressure throughout the apparatus. When this had occurred the boiling was commenced. It was found that with a reservoir of the dimensions given, the manometer and temperature indications were readily kept constant for a considerable time, and for each pressure observations could easily be made over an interval of at least 20 minutes. This is sufficient time to permit the conditions to become uniform and to obtain equilibrium. In every case, drying tubes containing freshly ignited calcium chloride were interposed between the air reservoir and the T-piece.



It is convenient to consider the boiling point and freezing point curves together.

*Phenol and o-cresol* give a continuous freezing point curve (Fig. 3). There are no breaks, and a minimum freezing point results with nearly equimolecular proportions of the two constituents. There is no clear eutectic point and no evidence of the formation of compounds. The boiling point curve (Fig. 6) is almost a straight line lying between the boiling points of the separate constituents.

*Phenol and m-cresol.* This freezing point curve (Fig. 1) is the most complicated and at the same time is of the greatest interest. It exhibits two minima, one at  $-4^{\circ}\text{C}$ . with 10 mols. %, and one at  $+10.2^{\circ}\text{C}$ . with 50 mols. % of phenol. A well defined maximum point occurs at  $+17^{\circ}\text{C}$ . with 33.3 mols. % of phenol. The latter point is noteworthy as it corresponds with a compound of phenol and *m*-cresol represented by  $\text{C}_6\text{H}_5\text{OH} \cdot 2\text{C}_7\text{H}_7\text{OH}$ . The form of the curve is that of many other freezing point curves of binaries and it has two eutectic points. That at  $-4^{\circ}\text{C}$ . results from the freezing point curve of *m*-cresol and the compound



and that at  $+10.2^{\circ}\text{C}$ . from the freezing point curve of this compound and phenol. It is difficult to avoid the conclusion that the compound of *m*-cresol and phenol is stable under the conditions described. The boiling point curve of phenol and *m*-cresol is not a straight line, but deviates from a straight line in the well-known manner of other mixtures of completely miscible liquids.

*p-Cresol and phenol.* This curve (Fig. 1) has two branches meeting at 50 mols. % where the eutectic point occurs. Lunge obtained similar results. The

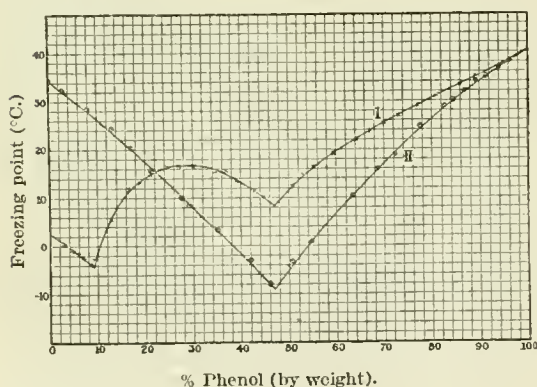


FIG. 1.

I. Phenol and *m*-cresol. II. Phenol and *p*-cresol.

boiling point curve of phenol and *p*-cresol is similar to that of phenol and *m*-cresol.

The freezing point curves for the cresols taken in pairs are shown in Figs. 2 and 3. Again *m*-cresol furnishes the most complicated curves. With *m*-cresol and *p*-cresol an undoubted but small maximum point results with about 20% of *p*-cresol and the eutectic occurs with the 50% mixture (Fig. 2). The form of this curve suggests the existence of a second eutectic very close to pure *m*-cresol, but we could not find the point with our apparatus.

The curve for *o*-cresol and *m*-cresol is a complex one, and recalls that of *m*-cresol and phenol. While there are two undoubted breaks in this curve with 40% and 80% of *m*-cresol, there is no well-defined

maximum between the breaks, and the existence of a compound of *o*-cresol and *m*-cresol cannot therefore be inferred.

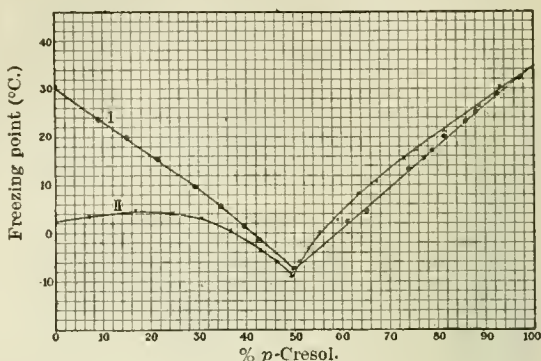


FIG. 2.

I. Ortho-cresol and para-cresol. II. Meta-cresol and para-cresol.

No very deep study of the freezing point curves is necessary to emphasise their great practical importance when considering methods for the valua-

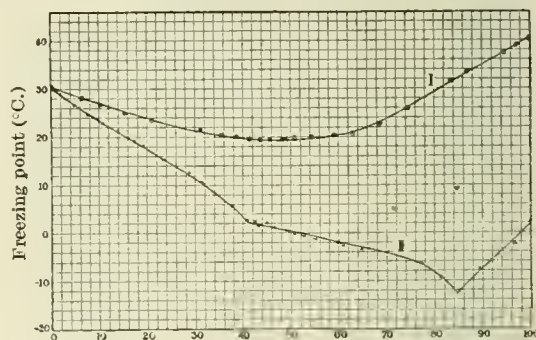


FIG. 3.

I. Phenol and *o*-cresol. % Phenol (by weight). II. *o*-Cresol and *m*-cresol. % *m*-Cresol.

tion of carbolic acid, such as Lowe's method. The outstanding fact is that many different mixtures of

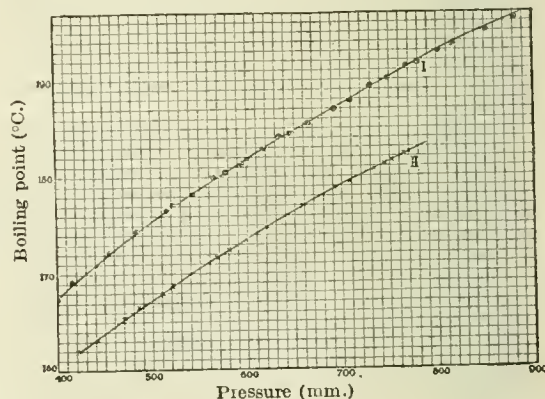


FIG. 4.

I. *o*-Cresol II. Phenol.

phenol and the cresols have the same solidifying point. Furthermore, mixtures containing large proportions of phenol may have very low melting points. This is clearly shown by drawing a horizontal line through the point  $10^{\circ}\text{C}.$  in Fig. 1. It is unsafe in practice to consider cresylic acid free from phenol simply because it yields a distillate which does not freeze when immersed in ice water. The bearing of these observations on the possibility of separating phenol from the cresols by freezing is evident.

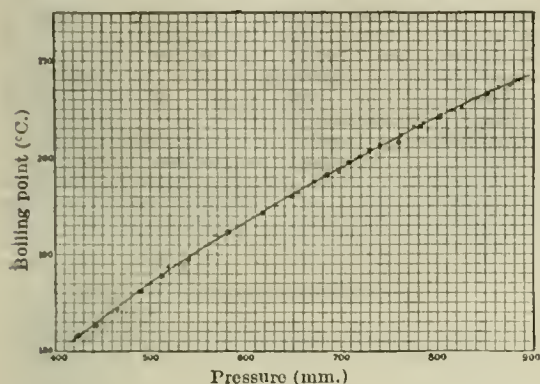


FIG. 5.

● *p*-Cresol.× *m*-Cresol.

Expressed in figures, the value of  $dt/dp$  for phenol and the cresols for the whole range of the curve is, phenol 0.059, *o*-cresol 0.065, *m*-cresol 0.059, *p*-cresol 0.061. *o*-Cresol gives the outstanding curve of these four, the other three being nearly parallel. The results may be put in another way and one of which the application in practice is important.

When it is desired to correct the boiling point of the cresols for pressure in the ordinary determination of the boiling point, the general formula

$$\theta = C(760 - p) \times (273 + t)$$

is used (Young's "Fractional Distillation," 1903, p. 14); where  $\theta$  is the correction to be added or deducted, and  $p$  and  $t$  are the observed pressure and temperature.  $C$  has the following values:—phenol 0.000101; *o*-cresol 0.000126; *m*-cresol 0.000103; *p*-cresol 0.000103. The high value of " $C$ " for *o*-cresol, which is quite outside the limits due to experimental errors, might be taken as some evidence that the substance possesses a simpler molecule than the other phenols, and in fact Hewitt and Winmill have shown that *o*-cresol is the least associated of the cresols and phenol (Chem. Soc. Trans., 1907, 91, 441).

Fig. 6 shows the boiling points of mixtures of phenol with the cresols and furnishes a good illustration of the general rule that with mixtures of closely related substances, the boiling point curve departs less from a straight line as the difference between the boiling points of the components diminishes. Following Young's nomenclature (*loc. cit.*), if the boiling point curve for any pair of substances is a straight line, the formula

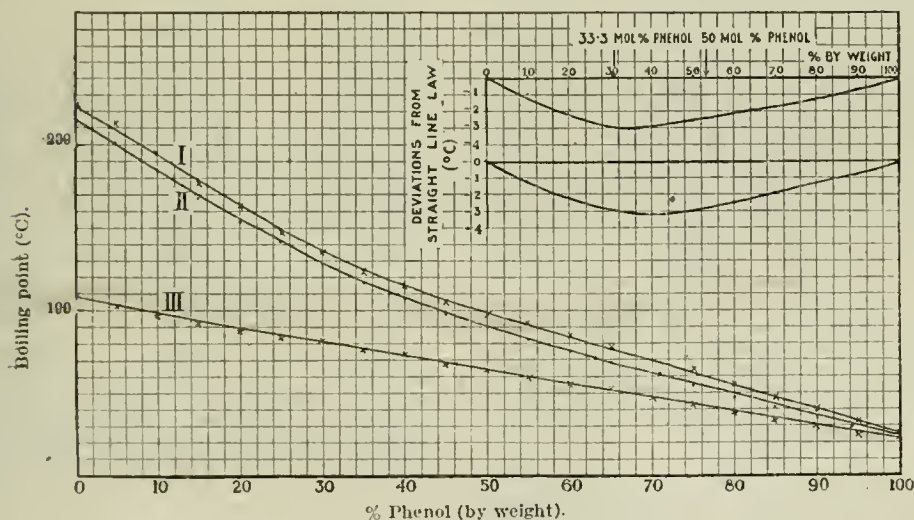


FIG. 6.

I. Phenol + *m*-cresol. II. Phenol + *p*-cresol. III. Phenol + *o*-cresol.

The boiling point curves showing the variation of boiling point with pressure (Figs. 4 and 5 and table) are almost straight lines. Those for *m*- and *p*-cresol almost coincide, as the boiling points of these two substances are only about  $1^{\circ}\text{C}.$  apart throughout the range of pressures examined.

$t' = t_b + 0.01m\Delta$  applies. In this case  $t_b$  is the lower boiling point,  $m$  is the molecular percentage of the higher constituent,  $\Delta$  the difference in boiling points between the constituents, and  $t'$  is the calculated boiling point of the particular mixture under consideration. If the difference between the calcu-

Boiling points at different pressures.

Pressure mm.	850	800	760	700	600	500	400
	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$	$^{\circ}\text{C}.$
Phenol ..	186.7	184.2	182.1	179.2	173.7	167.6	159.7
<i>o</i> -Cresol ..	194.7	192.5	190.5	187.5	181.8	175.7	168.2
<i>m</i> -Cresol ..	206.6	204.2	202.2	199.4	193.5	186.7	—
<i>p</i> -Cresol ..	206.6	204.1	202.1	199.2	193.4	187.0	179.3



lated boiling point and that observed is plotted against the molecular percentage composition of the mixture, the upper curves in Fig. 6 are obtained. These curves show in a convenient form the deviation of the boiling point of the phenol mixtures from the straight line law, followed by phenol and *o*-cresol.

We desire to express our indebtedness to Sir J. J. Dobbie, F.R.S., for permission to publish the results in this and the foregoing paper.

Government Laboratory.

May 13th, 1918.

## Sydney Section.

Meeting held on May 16th, 1918.

MR. H. O. SMITH IN THE CHAIR.

### THE USE OF THE REFRACTOMETER IN THE EXAMINATION OF EUCALYPTUS OILS.

BY HENRY G. SMITH, F.C.S.

It is now recognised that the determination of the physical properties of essential oils is of considerable value in their classification and investigation. The various oils obtainable from the eucalypts are, in most cases when true to species name, equally well controlled in this way, and of all the physical tests applicable for the purpose, that of refractive index is not the least valuable.

A relative constancy in the character of the oils of identical species of eucalyptus, growing under natural conditions, has been shown, and abundant evidence has accumulated on this point. Only in a very few instances does distinct local variation occur, and even with these species the reasons for this departure are fairly well understood, the selective peculiarities of the species themselves being perhaps the most important factor in this connection. No absolute constancy can, of course, be expected with plant products—essential oils particularly—as these are aggregates of constituents, each having its own physical constants. With the eucalypts this particular fact might suggest a difficulty in securing fairly concordant results on account of the diversity of constituents in the genus, but experience has shown that this is not so and the instances given below illustrate this clearly.

The methods of extraction of eucalyptus oils in Australia have improved considerably during recent years, and greater care is now taken in the selection of material from desired species; consequently the oil is more constant in character, and the steam-rectified product remarkably uniform in physical properties.

It is becoming customary to sell the oil from a particular eucalypt by its species name, as for instance, *Eucalyptus polybractea*, *E. Smithii*, *E. dives*, *E. Macarthuri*, and so on, and in this way it is possible to secure the desired uniformity in the product. When the advantages of this method become fully recognised and more extensively followed, the industry will be at a considerable advantage, and the practice of mixing oils from different species in order to meet a particular standard will become quite unnecessary.

Not only is the refractometer useful in controlling the oils themselves, but it is also the means of indicating the presence of previously undetected

constituents. It was in this way that butyl butyrate was found, first in the oil of *Eucalyptus Perriniana* (Proc. Roy. Soc., N.S.W., Dec., 1914). The abnormally low refractive index (1.4538 at 10° C.) of the lower boiling fraction suggested the presence of an unrecognised constituent in eucalyptus oils. This ester has since been shown to be a common constituent in the oils of allied species, not only from those common to Eastern Australia but from those of Western Australia also, localities thousands of miles apart.

The principal species now worked most extensively in Eastern Australia are, (1) *Eucalyptus polybractea*, the "Blue Mallee" of New South Wales, and the "Silver Leaf Mallee" of Victoria; (2) *Eucalyptus australiana*, the "Narrow Leaf Peppermint" or "Messmate" of New South Wales; (3) *Eucalyptus dives*, the "Broad Leaf Peppermint" of New South Wales and Victoria.

The figures tabulated below give the physical properties of the oils of these three species commercially distilled from material true to name, and during a considerable period of time:—

#### *Eucalyptus polybractea*.

Type of oil and date	Sp. gr. at 15° C.	Rotation $\alpha_D$	Refractive index at 20° C.
Crude oil, 1907 ..	0.9245	+0.8°	1.4631
Crude oil, 1909 ..	0.9269	+0.6°	1.4633
Steam rectified, 1911	0.9264	+0.5°	1.4586
Rectified, 1912 ..	0.9271	+0.4°	1.4596
Rectified, 1912 ..	0.9256	+0.5°	1.4598
*Rectified, 1913 ..	0.9273	-0.2°	1.4602
Rectified, 1913 ..	0.9270	+0.4°	1.4612
Crude oil, 1914 ..	0.9292	-0.3°	1.4624
Rectified, 1914 ..	0.9270	+0.3°	1.4598
Rectified, 1914 ..	0.9298	+0.2°	1.4608
Rectified, 1916 ..	0.9300	+0.5°	1.4596
Rectified, 1917 ..	0.9195	+0.3°	1.4598

\* This oil was from Bendigo, Victoria; all the others were from Wyalong, N.S.W.

It will be noticed that two of these samples were levorotatory. This is due to the influence of a small quantity of the optically active aromatic aldehyde aromadendral. The optical rotation of the oil of this species is but small at any time, and although mostly dextrorotatory, occasionally shows levo-rotation. This result in no way interferes with the quality of the oil, and is not due to phellandrene. The American objection to a levorotatory eucalyptus oil is, in this case, not warranted, and should not be persisted in. Considerable quantities of the oil of this species are now being sent to America, and I would suggest that the refractive index is a more satisfactory means of control than that of optical rotation, particularly as the oil is usually recently distilled when it leaves Australia.

The oil of this species is one of the richest in cineol content, and this is indicated by the low refractive index. The solubility in alcohol (79% by weight) of the steam-rectified oil is from 1.05 to 1.1 volumes and is remarkably constant.

#### *Eucalyptus australiana*.

This species was, prior to the investigation of the Tasmanian eucalypts by Mr. Baker and myself

(Roy. Soc. Tasmania, Oct., 1912), considered by botanists to be *E. amygdalina*, but the Australian tree has now been raised to specific rank. During the last three or four years considerable quantities of a high-class eucalyptus oil have been distilled from it in the Nerrigundah and Yowrie districts of New South Wales. In the "Research of the Eucalypts," Baker and Smith, 1902, p. 170, it was shown that by fractional separation at stated times during the primary distillation, the oil which came over during the first hour was much the richest in cineol. The yield of oil is also greater from this species than from other eucalypts. It is thus possible to adopt this mode of procedure. The species appears to have broken away from its natural habitat on the highlands and established itself at lower altitudes, where the phellandrene has practically disappeared, the cineol increasing to a corresponding extent (see Baker and Smith, Proc. Roy. Soc. N.S.W., Dec., 1915). In the above-mentioned districts the oil is now commercially separated at the end of the first hour, and contains about 70% of cineol or over; it is sold for pharmaceutical purposes. The remainder, which comes over during the later distillation, is utilised for the separation of minerals by flotation or for other purposes.

The constancy of the physical characters of the first-hour oil can be judged from the following figures, those of refractive index being in remarkable agreement.

*Eucalyptus australiana* of New South Wales.

First-hour oil.

Place and date	Sp. gr. at 15° C.	Rotation $\alpha_D$	Refractive index at 20° C.
Nerrigundah, 1913 ..	0.9188	+0.3°	1.4621
Nerrigundah, 1913 ..	0.9193	+0.9°	1.4628
Yowrie, 1913 .. ..	0.9186	+0.5°	1.4624
Yowrie, 1913 .. ..	0.9195	+0.4°	1.4622
Nerrigundah, 1914 ..	0.9193	+1.5°	1.4631
Yowrie, 1914 .. ..	0.9199	+0.1°	1.4622
Yowrie, 1914 .. ..	0.9202	-1.2°	1.4636
Nerrigundah, 1914 ..	0.9195	+1.4°	1.4631
Reedy Creek, 1915 ..	0.9196	+1.2°	1.4625
Yowrie, 1916 .. ..	0.9193	+1.3°	1.4626
Bellimbria, 1917 ..	0.9186	+1.7°	1.4617
Yowrie, 1917 .. ..	0.9205	+1.6°	1.4635
Wyndham, 1917 ..	0.9220	+0.7°	1.4635

Second-hour oil.

Yowrie, 1917 ..	0.9096	+2.0°	1.4710
Nerrigundah, 1917 ..	0.9160	+3.6°	1.4711

In the majority of cases the optical rotation is slightly to the right but occasionally the oil is levorotatory. This is due to the presence of a small proportion of phellandrene, but this is not detrimental to the quality of the oil. The value is indicated by the refractive index, which only shows a very slight increase over the dextrorotatory samples.

With the second-hour oil the refractive index is higher, and in this the cineol does not usually exceed a fourth of the amount present in the first-hour oil.

As the monetary value of the first-hour oil is about double that of the remainder, it may be necessary to adopt a somewhat rigid standard in order to maintain the quality of the first-hour oil.

The ready solubility in alcohol of the oil of *E. australiana* is particularly noticeable, even if distilled at high altitudes when phellandrene will be present and the rotation -12° to -15°. With the first-hour oil from the Nerrigundah and Yowrie districts the solubility is equal to that of *E. polybractea*, and is remarkably constant.

*Eucalyptus dives*.

The oil of this species, when freshly distilled, shows a corresponding relative constancy in constituents and physical properties, as is noticeable with other species of eucalyptus. The oil of *E. dives* consists principally of levorotatory phellandrene, has a high optical rotation, and contains, as a rule, less than 5% of cineol. Much of the oil of this species has reached Europe as the product of *E. amygdalina*. This was to a certain extent excusable, because *E. dives* was formerly classified as a variety of *E. amygdalina*, and varietal names seem soon to lose their significance.

The oil of *E. dives* is considered to be the best of all essential oils for mineral separation by flotation, and considerable quantities are now being used in Australia for that purpose. The species has a most extensive range in Eastern Australia and gives about a 3% yield of oil from freshly cut material.

When freshly distilled the levo-rotation is very high, as is also the refractive index. The tendency among distillers is now to distil the leaves of this species for several hours in order to obtain all the available oil from the leaf. The oil from this extended distillation has naturally a higher specific gravity than that of the oil from, say, a four hours' distillation, but for the purposes for which the oil is employed both are of equal value.

The following figures were obtained with the crude oils from the various localities, which are in some instances wide apart. The oils were all commercially distilled.

*Eucalyptus dives*, New South Wales.

Place and date	Sp. gr. at 15° C.	Rotation $\alpha_D$	Refractive index at 20° C.
Cooma, 1913 .. ....	0.8928	-61.3°	1.4803
Colombo, 1914 .. ..	0.8976	-62.3°	1.4803
4 hours' distillation, Mongarlowe, 1916 ..	0.8892	-69.8°	1.4798
8 hours' distillation, Mongarlowe, 1916 ..	0.9004	-63.9°	1.4811
Colombo, 1916 .. ..	0.9020	-55.4°	1.4808
Mongarlowe, 1916 ..	0.9011	-59.3°	1.4806
Braidwood, 1916 ..	0.9001	-56.7°	1.4809
Orange, 1916 .. ..	0.8950	-61.2°	1.4805

It is apparent that the determination of the refractive index of the oil of this species is of considerable value in its control.

The refractometer employed was a Zeiss-Abbé instrument with water-jacketed prisms, and for correction the figure 0.00047 was employed. For correcting the specific gravity the figure 0.00075 was used.



## NOTES ON CINEOL.

BY J. C. EARLE, F.I.C.

*Solubility in water.* The solubility of pure cineol at various temperatures was determined in an apparatus of the type devised by Farrington for the determination of fat in skim milk by the Babcock test (see Snyder, "Dairy Chemistry," p. 26). The following results were obtained:—

Temperature, °C.	Solubility, grms. of cineol in 100 grms. water.
15	0.64
17.5	0.57
19	0.54
21	0.35
40	0.21
50	0.19

*Detection of cineol by the iodol method.* A few tests were made to determine the sensitiveness of the iodol test for cineol. Various mixtures were made of pure cineol with a phellandrene oil containing no cineol. One c.c. of the oil under examination was warmed in a nearly boiling water-bath, the warm oil was saturated with iodol and filtered through a small paper into a watch-glass. Crystals of the double compound of cineol and iodol developed in the filtered oil immediately when one vol. of cineol was mixed with 3 vols. of phellandrene oil; on standing for a short time with 10 vols. of phellandrene oil, and after standing for half an hour with 20 or 30 vols.

## Communications.

## RESIDUAL AND EXTINGUISHING ATMOSPHERES OF FLAMES.\*

BY THOMAS FRED ERIC RHEAD.

When a jet of combustible gas or vapour is allowed to burn in air in a closed vessel, the flame gradually alters in character as the oxygen diminishes, and finally goes out. The atmosphere remaining after the flame has been extinguished is usually termed the "residual atmosphere"; such an atmosphere contains, besides oxygen and nitrogen, the products of combustion of the combustible gas.

On the other hand, a mixture of oxygen and some "inert" gas, such as nitrogen or carbon dioxide, can be prepared so that it is just unable to support the combustion of a combustible gas introduced into it. Such a mixture is termed an "extinguishing atmosphere."

The research described in this communication had as its main object the determination of the oxygen contents of the residual and extinguishing atmospheres for flames of various gases, the inert gas employed for the extinguishing atmosphere being nitrogen.

Several investigators have approached the problem from the point of view that the oxygen content of the residual and extinguishing atmospheres of a flame should have the same value, and that this value represents a physical constant for that flame.

Their results for methane, hydrogen, and carbon monoxide are as follows:—

TABLE A.

Investigator	Oxygen content of residual atmosphere			Oxygen content of extinguishing atmosphere		
	Me- thane %	Hy- drogen %	Car- bon mon- oxide %	Me- thane %	Hy- drogen %	Car- bon mon- oxide %
Clowes and Feil- mann* ..	15.6	5.5	13.35	17.4	6.3	15.1
W. P. Jerissen and N. H. Sie- wertz von Reesemat† ..	—	4.0	11.2	—	3.5	10.0
J. Harger‡ ..	17.5	7.5	—	—	—	—

\* This J., 1894, 1155.

† Chem. Weekblad, 1909, 6, 1053; see this J., 1910, 478.

‡ "Coal Mine Explosions" (1913).

The lack of agreement in the above results is probably due to differences in experimental conditions.

In order that a flame may be maintained when a stream of combustible gas issues into an atmosphere of oxygen and an inert gas (nitrogen) at ordinary temperature and pressure, a mixture of the gas with the atmosphere must be produced in sufficient quantity, which, when ignited, will self-propagate flame at a rate equal to the rate of flow of the mixture. For any particular combustible gas there exists a range of such mixtures in which flame can be propagated at rates rising from zero to a maximum and again falling to zero, as the mixtures used contain increasing percentages of combustible gas. As the oxygen content of the mixture is decreased the range is considerably reduced, until finally only one particular stationary mixture at ordinary temperature and pressure will just propagate flame.

Burgess and Wheeler (Chem. Soc. Trans., 1914, 105, 2591—2605; this J., 1914, 1195) found that for methane the mixtures with air which would propagate flame ranged from 5.6 to 14.8% methane, and Parker (Chem. Soc. Trans., 1915, 107, 328—337; this J., 1915, 482) obtained the following figures for the initial rate of propagation of flame in the intermediate mixtures (i.e., the rate of the "uniform movement") :—

Percentage of methane in air	Rate of propagation of flame, cm. per sec.
6	53
7	69
8	85
9	102
10	107
11	198
11.7	76

On reducing the oxygen content of the mixtures, Burgess and Wheeler (Chem. Soc. Trans., 1914, 105, 2598) reached the limit of 13.25 to 13.45% of oxygen, below which no variation in the proportions of methane and nitrogen would give stationary mixtures capable of self-propagating flame.

If a combustible mixture is caused to flow along a tube at a rate equal to the initial rate of propagation of flame in that mixture the flame will appear to be stationary, but if the gaseous flow be increased the flame will travel in the direction of that flow until it is projected into the air, where it will be extinguished, because the mixture is destroyed by dilution with air.

Immediately a stream of combustible gas emerges into an atmosphere of oxygen and nitrogen, intermediate mixture begins, and all ranges of mixtures are produced between the limits of 100% gas on the

\* Publication of this paper on the results of work carried out prior to the war, has been withheld until now because the author hoped to complete it, but has been continually prevented by war conditions.

one hand and 100% atmosphere on the other hand. One particular mixture will propagate flame at a maximum rate, and consequently it will be possible to maintain a flame with increasing velocity of gas stream, until the rate of travel of this mixture is just greater than the maximum rate of flame propagation. If, however, the oxygen content of the atmosphere be reduced, the range of mixtures of combustible gas and atmosphere which will propagate flame against the flow of the mixtures is also reduced, and a point is reached at which no mixture of gas and atmosphere is formed, in which the rate of flame propagation balances the flow of the mixture, consequently the flame is extinguished and the atmosphere represents the so-called extinctive atmosphere of that flame under the conditions existing.

The mixture which will just maintain flame against its own flow must of necessity be richer in oxygen than the stationary mixture which contains the lowest percentage of oxygen just to propagate flame. The same observation holds in the case of a gas burning to extinction in an enclosed volume of air at ordinary temperature and pressure, except that in this case the "residual" atmosphere contains products of combustion—usually carbon dioxide, which affects the flames rather differently than nitrogen.

A stream of combustible gas, issuing into an atmosphere of oxygen and nitrogen, mixes with that atmosphere through two main causes: (1) The interdiffusion between the gas and atmosphere, and (2) the mechanical mixing effected by the orifice or jet supplying the gas.

The maintenance of a flame in the mixtures so produced depends upon:—

(1) The rate of propagation of flame in the mixtures, involving (a) the number of molecules of oxygen concerned in the combustion of one molecule of gas. (b) The calorific value of the gas. (c) The conductivity and specific heats of the combustible gas, oxygen, nitrogen, and products of combustion. (d) The rate of radiation of heat

Under the most favourable experimental conditions the oxygen content of the residual or extinctive atmosphere left by flames of different combustible gases burning in air will depend primarily upon the rate of diffusion of the gas, and the effect of this will be increased or decreased according as the reaction is simple or complex, and according as the initial rate of flame propagation in the mixtures is great or small. For example, in the case of hydrogen (the residual atmosphere of the flame of which contains about 5.6% oxygen) there is a high rate of diffusion, a simple reaction, and a high initial rate of flame propagation in its mixtures with air, but in the case of methane (which gives a residual atmosphere containing about 16.0% oxygen) the rate of diffusion is much slower, the reaction more complex, and the rate of flame propagation smaller.

TABLE B.

	Hydrogen	Methane
Rate of diffusion .. ..	1.0	0.351
Number of mols. of oxygen required for the combustion of one molecule of gas .. ..	$\frac{1}{2}$ mol.	2 mols.
Initial rates of inflammation in combustible mixtures with air .. ..	20    280 30    430 40    490 50    385 60    190	6    53 7    69 8    85 9    102 10    107 11    198 11.7    76
Oxygen content of residual atmosphere .. ..	About 5.6%	About 16.0%

\* Haward and Ottagawa, Chem. Soc. Trans., 1906, 109, 83-89; this J., 1916, 244.

† Parker, *loc. cit.*

#### EXPERIMENTAL.

##### Residual atmospheres.

The chief difficulty met with in determining the residual atmosphere of a flame is that caused by the hot products of combustion heating the contents of the vessel and so enabling the flame to burn longer than it would do if the contents remained at the room temperature. For a flame of constant size this error is likely to be less in a large vessel than in a small one, because the products have a longer time to cool before they come into contact with the flame again. Even with a large vessel this difficulty is experienced if the flame is sufficiently large, because when nearing extinction the flames of some gases rise above the jet and burn in the heated atmosphere at the top of the vessel.

Experiments were carried out in two apparatus, one of 1½ litres capacity and one of 12 litres. Most of the results in the smaller apparatus were vitiated by the heating of the contents by the flames.

The 12 litre apparatus (Fig. 1) consisted of an inverted aspirator bottle with a jet placed axially 3 in. from the bottom and fitted with electrical ignition. Samples were taken simultaneously from the top and bottom of the apparatus, and, although no mechanical method of circulation was resorted to, these samples showed very little difference in oxygen content. The top sample contained approximately 0.25% less oxygen than the bottom sample, unless flames over 1½ in. high were used, in which case wider differences occurred between the two samples. All analyses were carried out in a Bone and Wheeler gas analysis machine. The bottom sample, taken 2 in. above the jet, was recorded as the residual atmosphere. Temperatures taken by means of thermometer B at the side of the jet 1 in. away showed a rise in temperature of only one or two degrees during the experiment,

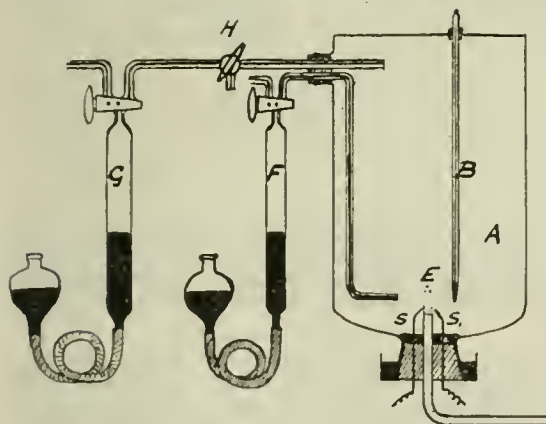


FIG. 1.

from the flame. (e) The dilathermancy of the gases. (f) The ignition temperature of the gas in the atmosphere concerned. (g) The rate of diffusion of the gases. (h) The effect of temperature on the rates of the chemical reactions involved.

(2) The rate of flow of the combustible mixtures away from the orifice, involving the rate of flow of the stream of combustible gas.

(3) The size of the flame, involving the area over which combustion is taking place and the amount of combustible mixture produced in unit time.

(4) The cooling effect (and possible catalytic effect) of the orifice when the flame remains at that point.



therefore unless the flames rose to the top of the vessel they would not be appreciably sustained by any heating of the contents of the vessel.

The pressure was maintained at that of the atmosphere by allowing some gas to escape (1 or 2% by volume of the enclosed atmosphere) at the beginning of the experiment through H dipping into a water seal. Preliminary experiments using a pressure gauge had shown that expansion of the atmosphere only occurred at the commencement of an experiment, and that from then, right to extinction very little variation in pressure took place, until the flame went out, when a rapid fall in pressure set in.

The influence of the size of the flame was first determined, employing pure methane. Using a glass tube jet 4 mm. internal diameter, and varying the rate of the gas stream to give flames of different sizes, the following results were obtained:—

Initial height of flame, cm.	Oxygen in residual atmosphere, %.
0.6	16.8
3.0	15.6
6.0	14.9

The 3 and 6 cm. flames, when near extinction, travelled to the top of the vessel, where they continued burning for some time in the heated atmosphere, and hence the samples taken near the jet in

the increasing proportion of carbon dioxide produced per volume of oxygen consumed, but this requires confirmation. No carbon monoxide or unburnt gas was detected in the residual atmosphere of methane; the methane had completely burned to carbon dioxide and steam. The methane flame enlarged considerably during combustion and gradually rose as a non-luminous blue cone about 8 in. above the jet, where it finally went out.

In the case of propane the flame increased to about three times its initial size, became non-luminous, rose above the jet, and flickered violently before extinction. The butane flame increased from 2 inches to 6 inches in height, became smoky, and flickered violently towards the end. Owing to its high density some of the gas fell down the sides of the jet and a very slight flame flashed through it. The pentane flame (from a small lamp) decreased considerably from an initial height of 1 inch to about  $\frac{1}{4}$  inch, owing presumably to the decreased evaporation consequent on the flame becoming cooler when nearing extinction. The cyanogen flame enlarged from 1 to 3 inches and had a red inner cone surrounded by a blue zone, which was again surrounded at the top by a green cone. It flickered violently before extinction. The blue carbon monoxide flame burned steadily, rose a little

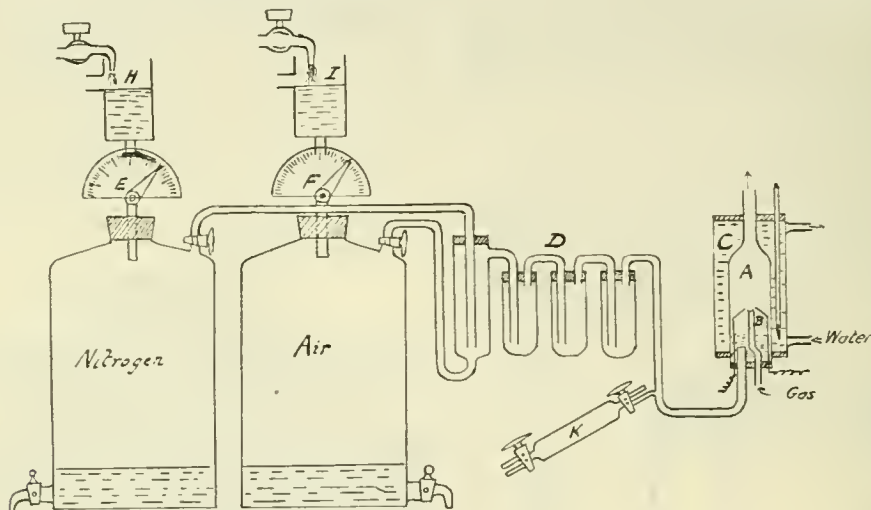


FIG. 2.

these cases do not accurately represent the atmosphere round the flame, but the results seem to indicate that a large flame has a lower oxygen content in its residual atmosphere than a small flame, quite apart from the heating of the atmosphere.

The relative values of the oxygen contents of the residual atmospheres left by the flames of different gases were determined, using a silica open-tube jet of 4 mm. internal diameter, and supplying the gases at the same rate in each case. The initial size of the flame varied from about  $\frac{1}{4}$  in. in height in the case of hydrogen to about 2 in. in the case of butane.

Gas used.	Oxygen in residual atmosphere.	
	%	%
Methane ... ..	15.6	15.4
Propane ... ..	15.9	15.8
Butane ... ..	16.0	—
Pentane ... ..	16.4	—
Cyanogen ... ..	15.3	15.0
Hydrogen ... ..	5.7	5.6
Carbon monoxide ... ..	10.2	—

The slight increase noticeable on ascending the methane series of hydrocarbons is possibly due to

above the jet, and increased in size before extinction. The hydrogen flame increased very considerably in size and became almost invisible, and rose high above the jet before extinction.

#### Extingtive atmospheres.

The method usually adopted for ascertaining these (plunging the flame into ready-made mixtures and obtaining the correct mixture by the method of trial and error) was considered unsuitable, and an apparatus was devised to study the effect of feeding a flame with mixtures of oxygen and nitrogen. By gradually increasing the nitrogen in the mixture a point was reached at which the flame went out. The mixture of oxygen and nitrogen which just extinguished the flame was recorded as the extinctive atmosphere for that flame under the conditions of the experiment. The method allows of fine adjustment and control, and the flame can be studied in all its phases under varied conditions.

The apparatus used (Fig. 2) consists essentially of two parts: one for making and supplying the atmosphere of desired composition, and the other for observing the effect of that atmosphere on the flame in question. Two large gas holders were filled, one

with air and the other with nitrogen, and connected to the mixer, D. When the graduated taps, E and F, were opened, water flowed into the gas holders from the constant-level reservoirs, H and J, and displaced the air and nitrogen, causing them to pass forward into D. By regulating E and F, a steady stream of air and nitrogen of constant composition passes from the mixer to the combustion chamber, A. With well-made cleanly-drilled water taps, E and F, graduated to pass known quantities of water under constant head, the volumes of air and nitrogen could be very easily adjusted to give any desired mixture which, moreover, could be repeated at will. The connections in the mixer were made of glass tubing 9 mm. internal bore. This mixer was found very efficient for delivering up to four litres of a mixture of oxygen and nitrogen per minute.

By allowing mercury to run out of K, a sample of the atmosphere was obtained for analysis. The glass cylindrical combustion chamber, A, was 2.4 cm. internal diameter and 12 cm. high, and was surrounded by the water jacket, C. During an experiment a steady stream of cold water flowed through C, and the temperature was noted by the thermometer. The combustible gas was supplied at any desired constant rate to the jet, B, from a gas-holder of the same type as those used for supplying the air and nitrogen, and was ignited electrically. A little glass wool packed loosely at the bottom of the chamber, A, served to arrest any upward draught likely to be caused by the tube supplying the atmosphere. In this apparatus the heating effect of the products of combustion on the atmosphere was eliminated.

The experiments were conducted in the following manner: A steady stream of cold water was sent through the water jacket and a stream of air from the air-holder through the mixer and combustion chamber at a rate just sufficient to keep the flame steady at the open-tube jet, B. The nitrogen was then gradually turned on (time being allowed between each increase in nitrogen to wash out the mixer with the new mixture) until the flame just went out, and then, before altering E and F, a sample was taken in K and analysed as representing the extinctive atmosphere.

The velocities of the gas stream and atmosphere were calculated from the diameter of the gas jet and combustion chamber, and the volume of gas and atmosphere passing through per minute.

The following observations were made on the limited number of experiments carried out:—

(1) For a constant speed of combustible gas stream and constant speed of atmosphere the extinctive atmospheres of methane, ethane, propane, and butane were found to be identical. This possibly points to a similar primary reaction in each case.

TABLE C.

Com- bustible	Internal diameter of jet, mm.	Speed of atmosphere, cm. per min.	Speed of gas, cm. per min.	% O <sub>2</sub> in extinctive atmosphere
Methane ..	4	684	288*	16.6
Propane ..	4	715	288	16.6
Propane ..	3	362	230†	16.3
Butane ..	3	362	230	16.3

\* = 36 c.c. per minute.

† = 16 c.c. per minute.

(2) The oxygen content of the extinctive atmospheres of the above gases decreases (within limits) with the increased speed of atmosphere.

It appears that, within certain limits (yet to be determined), if the extinctive atmosphere supplying the flame is to have a minimum oxygen content, it must be supplied at a speed greater than that

of the combustible gas. The minimum speed will depend upon the rate of propagation of flame in the mixture produced.

TABLE D.

Com- bustible	Internal diameter of jet	Speed of atmosphere, cm. per min.	Speed of gas, cm. per min.	% O <sub>2</sub> in extinctive atmosphere
	mm.			
Propane ..	4	715	288	16.6
Propane ..	4	937	288	15.0
Methane ..	4	684	288	16.6
Ethane ..	4	790	288	15.0
Butane ..	4	822	288	15.3
Propane ..	4	937	288	15.0
Methane ..	3	195	230	18.2
Propane ..	3	362	230	16.3

With a slow speed of atmosphere the oxygen is burnt out too quickly, or what amounts to the same thing, the combustible mixture is formed too slowly, but by supplying the oxygen faster, i.e., by increasing the speed of the atmosphere, the combustible mixture is formed quickly enough to maintain flame. The limit to which the speed of the atmosphere can be increased will be reached when the rate of inflammation in the mixture just balances the upward movement of the mixture.

It is thus seen how an atmosphere, too poor in oxygen to maintain a flame when fed to the latter at a slow speed, may be able to maintain that flame when fed at an increased speed. This tends to invalidate Harger's suggestion of employing in coal mines an atmosphere containing 17% oxygen as a preventative of explosions.

An experiment in the apparatus with ethylene delivered through a 4 mm. jet at a rate of 36 c.c. per min., i.e., 288 cm. per sec. and the atmosphere at 872 cm. per sec., gave an extinctive atmosphere containing 13.3% oxygen.

(3) A coal gas flame behaves quite differently from the paraffin hydrocarbon flames. The oxygen content of the extinctive atmosphere of coal gas appears to be almost unaffected by the speed of the atmosphere, but decreases with the increased speed of the gas stream:—

TABLE E.

Speed of atmosphere, cm. per min.	Internal diameter of jet, mm.	Speed of gas, cm. per min.	O <sub>2</sub> content of extinctive atmosphere
500	3	514	13.2
291	3	514	13.1
253	3	514	13.5
500	3	514	13.2
500	3	1028	12.3

This curious behaviour of coal gas will be more easily understood from the following observations and experiments.

It is always noticed that a coal gas flame near extinction behaves differently from the flame of a pure gas such as methane. When the flame of the latter almost reaches extinction it leaves the jet, and can be made to burn at any distance above the jet (within limits) as a blue cone. On the other hand, a coal gas flame near extinction becomes broken, the top portion disappears, leaving a hollow cup-shaped blue flame on the jet, and some gas escapes unburned.

This phenomenon is probably due to coal gas consisting chiefly of hydrogen and methane, two gases possessing widely different oxygen contents in their extinctive atmospheres, and though there might be sufficient oxygen present to support a hydrogen flame, the methane cannot burn, or at least only partially. By sampling the centre of a cup-shaped flame produced in a large form of the apparatus, it was found to consist of inflammable gas containing a much higher percentage of methane than the



original coal gas. The hydrogen burned preferentially.

The following result was obtained using a mixture of 50% of pure methane and 50% of pure hydrogen:—

Original mixture		Sample from centre of cup-shaped flame
Hydrogen	...	10.3
Methane	...	36.1
Carbon dioxide	—	0.8
Carbon monoxide	—	0.5
Oxygen	...	2.1
Nitrogen	...	50.2

The observation that increased speed of coal gas stream gives a lower value for oxygen in its extinctive atmosphere may be explained as follows: When the atmosphere supplying the flame contains less than about 15% of oxygen the methane in the gas begins to act as a diluent. In order that it may burn it must absorb heat from the hydrogen flame. Below 15% oxygen, however, the combustible mixture of oxygen and hydrogen is still formed. With slower gas streams hydrogen is burnt too quickly, but by increasing the hydrogen supply, that is by increasing the speed of the gas stream, the combustible mixture of oxygen and hydrogen is produced quickly enough to maintain a flame. The limit to which the increased speed of gas stream can be carried will be reached when the rate of propagation of flame in the mixture is just able to balance the upward movement of the gas.

#### Preparation of gases.

**Methane** was prepared by the action of water on a comparatively pure form of aluminium carbide, the gas being washed with ammoniacal cuprous chloride solution to remove acetylene. Hydrogen was removed by passing the gas through a U tube containing oxidised palladium sponge, heated to 100° C. in a water bath. The methane used was analysed by explosion analysis and found to contain 98% of CH<sub>4</sub>.

**Ethane**.—Prepared from zinc ethyl and water. The gas was liquefied by liquid air and fractionated three times, and gave a product with a purity of 98%.

**Propane**.—Prepared by the action of dilute hydrochloric acid on a paste of zinc dust and propyl iodide. The gas was passed through a cooled worm, and then through concentrated sulphuric acid and strong potash solution; it was then liquefied by solid carbon dioxide and ether, and fractionated four times. The propane had a purity of 99%.

**Butane**.—Prepared from normal butyl iodide in the same manner as the propane. Purity 99%.

**Hydrogen**.—Prepared by the action of sulphuric acid on zinc, passed through boiling alkaline potassium permanganate, and then over copper gauze at a red heat, giving a gas of 99% purity.

**Carbon monoxide**.—Prepared by the action of strong sulphuric acid on a strong solution of formic acid (sp. gr. 1.2); the gas, after passing through strong caustic potash solution, had a purity of 99%.

**Cyanogen**.—Prepared by heating pure recrystallised mercuric cyanide, the gas being liquefied by carbon dioxide and ether and fractionated several times. It had a purity of 99.5%.

**Ethylene**.—Prepared by the action of sulphuric acid on alcohol, the gas being washed through water and concentrated caustic potash.

**Nitrogen**.—This was supplied in cylinders, and was free from oxygen and carbon dioxide.

In conclusion the author wishes to thank Dr. R. V. Wheeler (who suggested the problem as having an important bearing on the composition of coal mine atmospheres) and the Home Office Explosions in Mines Committee for permission to publish the results of the work.

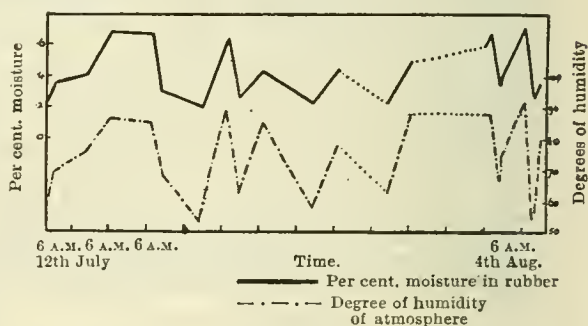
## MOISTURE IN RAW RUBBER.

BY O. STAFFORD WHITBY, M.Sc., A.R.C.S.

The amount of moisture absorbed by a sample of Hevea crêpe rubber and by a sample of Hevea sheet rubber (both cut into small pieces) when kept (in desiccators) at 16° and at 30° C. in air of different degrees of humidity, has been determined by van de Leur.\* The present paper records observations made† under actual tropical conditions on the variation of the water-content of raw rubber and the relation of this quantity to the humidity of the atmosphere, the form of the rubber, and the presence of serum solids. The observations are confined to rubber produced by acetic acid coagulation from the latex of *Hevea brasiliensis* (the common plantation rubber tree).

The moisture-content of both crêpe rubber and sheet rubber was observed to exhibit a diurnal change, being, on a normal day in the Eastern tropics, very markedly higher in the early morning than in the late afternoon. Three samples of latex crêpe (Nos. 1, 2, and 3), taken at random from a day's crop, showed, after they had become "dry" according to the usual criterion (*i.e.*, the absence of white, opaque spots) and were ready for shipment, the variations of moisture-content recorded in Table I. In this table the temperature and the degree of humidity of the atmosphere at the various times are also recorded. Unless otherwise stated, the samples hung in a small drying shed. Sample No. 4, included in the table, is a sample of sheet rubber and will be referred to later. The samples were of the same size (approx. 30 grms. each), and their thickness was that usual for thin latex crêpe—say 100 grms. per 1000 sq. cm.

The general parallelism between the water-content of the above samples of crêpe and the degree of humidity of the atmosphere is well shown in the following figure. It is also clear from this figure that in general the percentage of moisture present is high in the early morning and low in the late afternoon.



The course of drying of samples of latex crêpe from a time shortly after their preparation to the time when they were dry (*i.e.* free from opaque spots) was followed. It was found that after the percentage of water had fallen to (approximately) 1% the samples showed a negative rate of drying during the most humid part of the 24 hours—say, from sunset to sunrise; and that, in general, the rate of drying was influenced by variations in the degree of humidity of the atmosphere. The figures for the three samples of crêpe quoted in Table I. are given in Table II.

Sheet rubber was found to be less sensitive than crêpe rubber, as regards its moisture-content, to

\* Mededeelingen van den Rijksvoortlichtingsdienst ten behoeve den rubberhandel en de rubbernijheid te Delft, 2e. Serie, 1916, 473-9; this Journal, 1917, 1104.

† In the Federated Malay States; mostly in 1913.

TABLE I.  
Variation of moisture-content of "dry" crêpe.

Time	Temperature, °C.	Humidity	Moisture %* Sample No. 1	Moisture % Sample No. 2	Moisture % Sample No. 3	Moisture % Sample No. 4
July 10, 4 p.m. .. ..	30.5	69	0.19			
" 11, 7.40 a.m. .. ..	26.4	82	0.10			
" 11, 2.30 p.m. .. ..	31.7	61	0.13		6.23	
" 11, 6.30 p.m. .. ..	30.0	69	0.25		0.35	
" 12, 3.15 p.m. .. ..	29.7	76	0.28	0.29	0.41	
" 13, 7.10 a.m. .. ..	24.7	86.5	0.53	0.64	0.68	
" 11, 9 a.m. .. ..	27.2	85	0.53	0.64	0.68	
" 14, 4.30 p.m. .. ..	30.5	69	0.18	0.25	0.29	
" 22, 3.15 p.m. .. ..	32.0	54	0.11	0.17	0.21	
" 22, 5.30 p.m. .. ..	30.8	58	0.10	0.15	0.19	0.25
" 23, 8.15 a.m. .. ..	26.1	89.5	0.51	0.60	0.63	0.36
" 23, 5 p.m. .. ..	31.4	61	0.16	0.23	0.26	0.27
" 24, 8.20 a.m. .. ..	27.8	85	0.36	0.15	0.42	0.32
" 25, 4.15 p.m. .. ..	31.7	58.5	0.14	0.20	0.23	0.25
" 26, 8.30 a.m. .. ..	27.0	78	0.35	0.42	0.44	0.29
Aug. 1, 6.30 a.m. .. ..	—	—	0.49	0.57	0.58	0.36
" 1, 4.20 p.m. .. ..	32.0	63	0.16	0.21	0.23	0.25
" 2, 6.50 a.m.† .. ..	25.0	88	0.42	0.50	0.50	0.31
" 4, 6.50 a.m. .. ..	24.7	88	0.35‡	0.42‡	0.60	
" 4, 9.40 a.m. .. ..	25.0	88	0.53	0.60	0.69	
" 4, 10.50 a.m. .. ..	27.0	82.5	0.51	0.64	—	
" 4, 11.50 a.m. .. ..	—	—	0.43	0.54	0.50	
" 4, 2.50 p.m. .. ..	30.3	67	0.32	0.38	0.39	
" 4, 3.50 p.m. .. ..	30.0	67	0.28	0.35	0.35	
" 4, 4.50 p.m. .. ..	29.2	75	0.33§	0.32§	0.40	
" 5, 6.50 a.m. .. ..	23.9	92	0.39	0.47‡	0.71	
" 5, 8.45 a.m. .. ..	26.1	80	0.40‡	0.64	0.53	
" 5, 9.55 a.m. .. ..	—	—	0.36	0.54	0.41	
" 5, 10.56 a.m. .. ..	—	—	0.29	0.42	0.36	
" 5, 11.55 a.m. .. ..	30.0	55	0.25	0.35	0.31	
" 5, 1.55 p.m. .. ..	31.4	58	0.22	0.27	0.27	
" 5, 2.55 p.m. .. ..	—	—	0.22	0.25	0.27	
" 5, 5.30 p.m. .. ..	29.6	79	0.33	0.34	0.37	

\* Percentages in this and subsequent tables refer to the bone-dry sample.

† The crêpe samples were now dried in a desiccator, and the dried material was exposed to the air again on the following day. Nos. 1 and 2 were kept in a cupboard over-night.

‡ Sample now hung in the open again.

§ Sample now placed in a cupboard over-night.

TABLE II.  
Variation in rate of drying crêpe.

Time	Temperature, °C.	Humidity	Per cent. moisture	Rate of drying in interval*	Per cent. moisture	Rate of drying in interval*	Per cent. moisture	Rate of drying in interval*
			SAMPLE No. 1		SAMPLE No. 2		SAMPLE No. 3	
June 28, 8.30 p.m. .. ..	27.0	80	10.83		11.86		14.88	
" 28, 9.30 p.m. .. ..	26.4	84.5	10.48	3.27	11.48	3.15	14.39	3.43
" 29, 8.50 a.m. .. ..	25.8	87	8.19	1.02	9.13	2.07	11.20	1.95
" 29, 12 noon .. ..	30.0	70	7.20	3.66	8.13	3.30	9.94	3.67
" 29, 3.30 p.m. .. ..	28.6	80	6.41	3.11	7.37	2.64	8.92	2.94
" 29, 6.30 p.m. .. ..	28.1	82	5.88	2.62	6.86	2.22	8.27	2.28
" 29, 9.15 p.m. .. ..	26.7	85	5.62	1.69	6.59	1.59	7.87	1.90
" 30, 8.30 a.m. .. ..	26.7	85	4.65	1.53	5.65	1.21	6.58	1.45
" 30, 2.45 p.m. .. ..	26.4	85	3.79	2.06	4.80	2.41	5.50	2.63
" 30, 7.10 p.m. .. ..	24.4	89.5	3.60	1.15	4.65	0.70	—	—
July 2, 9.10 a.m. .. ..	25.6	87	1.86	1.27	2.84	1.04	2.97	1.08
" 2, 5 p.m. .. ..	25.6	84.5	1.37	3.35	2.27	2.48	2.36	2.63
" 2, 6.30 p.m. .. ..	25.7	83	1.38	-0.29	2.27	-0.14	—	—
" 3, 7 a.m. .. ..	23.3	91	1.34	0.26	2.22	0.21	2.28	0.34
" 3, 11 a.m.† .. ..	25.3	84	1.24	1.79	2.02	1.41	2.10	1.58
" 3, 8.45 p.m. .. ..	26.1	91	0.91	2.77	1.67	2.06	1.65	2.20
" 4, 7 a.m. .. ..	24.2	89.5	0.95	-0.48	1.68	-0.08	1.65	0.00
" 4, 9.30 a.m. .. ..	25.6	87	0.91	1.68	1.64	1.16	—	—
" 4, 11.45 a.m. .. ..	28.3	76	0.75	8.17	1.46	5.17	1.39	3.30
" 4, 2.30 p.m. .. ..	30.0	69	0.54	10.09	1.20	6.21	1.14	6.48
" 4, 5.15 p.m. .. ..	30.7	70	0.45	6.25	—	—	—	—
" 4, 7 p.m. .. ..	27.8	78	0.50	-7.02	1.09	2.03	1.03	1.93
" 5, 7 a.m. .. ..	24.7	90	0.72	-3.70	1.28	-1.50	1.14	-1.68
" 5, 12 noon .. ..	28.9	72	0.47	6.92	1.00	4.43	0.92	5.22
" 6, 7.15 a.m. .. ..	25.0	89.5	0.62	-2.26	1.08	-0.41	1.03	-0.59
" 6, 6.30 p.m. .. ..	27.8	85	0.28	5.25	0.60	3.93	0.58	3.89
" 8, 7.20 a.m. .. ..	25.0	89	0.36	-0.99	0.58	-0.10	0.56	-0.06
" 8, 2.30 p.m. .. ..	29.2	74	0.26	4.17	0.45	3.22	0.44	3.04
" 9, 7.15 a.m.† .. ..	22.8	89	0.48	-5.24	0.68	-3.05	0.67	-2.97
" 9, 12 noon .. ..	29.2	70	0.29	8.29	0.47	6.43	0.46	6.45
" 9, 5.30 p.m. .. ..	30.6	71	—	—	0.30	6.56	0.31	5.85
" 10, 7.30 a.m. .. ..	25.6	84.5	0.36	-1.23	0.49	-4.49	0.52	-4.70
" 10, 4 p.m. .. ..	30.6	69	0.19	5.69	0.28	4.95	0.31	4.76
" 11, 7.40 a.m. .. ..	26.4	82	(dry)	—	0.50	-5.77	0.52	-5.10
" 11, 2.30 p.m. .. ..	31.7	61	—	—	0.21	8.51	0.23	8.05
" 11, 6.30 p.m. .. ..	30.0	69	—	—	0.29	-9.35	(dry)	—
" 12, 3.45 p.m. .. ..	29.7	76	—	—	0.29	0.00	—	—

\* Expressed as percentage of the water present at the time of the last observation which has been lost (or gained) per hour in the interval.

† Dull morning.

‡ Heavy rain during interval.



changes in the degree of humidity of the surrounding atmosphere. It exhibited smaller variations than crêpe in the percentage of water present at different times: on the one hand it retained a higher percentage of water in the middle of a hot, dry day, and on the other hand it absorbed less moisture over-night. Observations on the variations in moisture-content of a considerable number of samples of sheet were made. The figures for the sample of sheet (no. 4, weight 35 grms.) included in Table I, were typical.

It is probable that the higher moisture-retaining capacity of sheet, and, thus, the higher percentage of moisture which sheet samples taken under the usual commercial conditions show, depends upon the fact that the serum solids are removed from the coagulum less completely in the preparation of sheet than in the preparation of crêpe, the coagulum being only rolled and washed in the preparation of sheet, whereas it is masticated in a strong current of water in the preparation of crêpe.

TABLE III.

*Variation in moisture-content of rubber containing serum solids.*

Time	Temperature, °C.	Per cent. moisture
Jan. 15 .. .. .		Dry
" 17 .. .. .		3.11
" 18 .. .. .		3.22
" 19, 9.15 a.m. ..	27.0	2.77
" 19, 3.30 p.m. ..	33.0	1.94
" 20, 3 p.m. .. .	33.0	2.00
" 20, 5.45 p.m. ..	31.0	2.00
" 21, 9 a.m. .. .	28.5	2.71
" 21, 10 a.m. .. .	29.6	2.61
" 21, 3 p.m. .. .	32.3	2.05
" 21, 8.30 p.m. ..	28.1	2.19
" 23, 9 a.m. .. .	27.9	2.79
" 24, 8.30 a.m. ..	26.7	2.81
" 25, 7.20 a.m. ..	23.7	2.27
" 25, 6.10 p.m. ..	29.7	1.66
" 26, 8.55 a.m. ..	25.0	2.31
" 28, 4 p.m. .. .	33.0	1.44
" 29, 9.15 a.m. ..	27.5	2.17
" 30, 7.15 a.m. ..	24.0	2.37
" 30, 3.50 p.m. ..	31.0	1.76
" 31, 9.30 a.m. ..	27.5	2.02

It is to be expected that, owing to differences in the degree of dilution of the latex employed, to differences between different samples of latex in regard to the actual ratio of serum solids to rubber,\* and to differences in the amount of rolling to which the coagulum is subjected, commercial samples of sheet will differ somewhat in the amount of serum solids which they contain, and, thus, on the view that the moisture-retaining capacity of raw rubber is associated with its content of serum solids, will vary in the percentage of moisture which they retain under identical conditions. It was, in fact, observed that samples of sheet do exhibit differences in the percentage of moisture which under given conditions they contain.†

The greater readiness with which crêpe, as compared with sheet, increases its water-content in a humid atmosphere may probably be justly regarded as due to the much greater surface which this form presents. As results mentioned later suggest, it would seem that the greater the area of a rubber sample in relation to the weight, the greater is the sensitiveness, as regards water-content, to changes in the humidity of the surrounding atmosphere.

That the presence of serum solids in the dry coagulum is closely associated with the moisture-content of the latter was shown as follows. It

was found that rubber prepared in such a way as to contain the whole or the greater part of the solids of the latex serum had a much higher water-content than ordinary sheet or crêpe and exhibited a greater absolute variation of water-content in response to changes in the degree of humidity of the atmosphere. Table III. shows the percentage of water present at various times in a sample of rubber (in sheet form) containing the whole of the serum solids. A considerable number of other samples of a similar character were examined and found to show variations in water-content of a similar magnitude.

Table IV. shows variations in water-content of three portions of different size from the above sheet, and indicates that the larger the piece in question, the smaller is the extent to which the percentage of water present changes with variations in the humidity. No. 1 represents a portion of the sheet weighing when dry 1.2435 grms.; No. 2 the portion quoted in Table III., weighing 2.5000 grms. In this table are also included for comparison figures for a sample of ordinary sheet (unsmoked; weight, 5 grms.), which are typical of those for four samples of ordinary sheet under observation over the period in question.

TABLE IV.

Time	Temperature, °C.	Per cent. moisture No. 1	Per cent. moisture No. 2	Sheet sample
Jan. 23, 9 a.m. ..	27.9	3.79	2.79	
" 24, 8.30 a.m. ..	26.7	3.87	2.81	0.26
" 24, 4.30 p.m. ..	31.5	—	—	0.23
" 25, 7.30 a.m. ..	23.7	—	2.27	0.24
" 24, 6 p.m. .. .	29.7	2.23	1.66	0.17
" 24, 4 p.m. .. .	33.0	2.11	1.44	0.17
" 29, 9.15 a.m. ..	27.5	—	2.17	0.22
" 30, 7.15 a.m. ..	24.0	—	2.37	
" 30, 3.50 p.m. ..	31.0	—	1.76	

Dry samples of the serum solids were prepared and were found to be extremely hygroscopic. Thus 0.315 gm. exposed in an evaporating basin absorbed 36.8% of its weight of moisture within 35 minutes.

*Conclusions.* The chief conclusions indicated are as follows:—

1. The percentage of moisture in raw rubber in the form of sheet or crêpe varies considerably with the degree of humidity of the surrounding atmosphere. In rubber-producing climates it shows a diurnal variation.

2. Sheet rubber tends to retain a higher percentage of moisture than crêpe.

3. The moisture-retaining capacity of raw rubber is closely associated with the presence of serum solids. The latter are very hygroscopic.

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## COMPARATIVE METHODS FOR DETERMINING THE STATE OF CURE OF RUBBER.

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The state of cure or degree of vulcanisation of rubber may be formulated in reference to (1) the percentage of combined sulphur calculated on the rubber present, that is the coefficient of vulcanisation, (2) the physical properties of the vulcanisate, particularly the load supported per unit cross-sectional area at a given elongation or *vice versa*. The former method is independent of the age and external conditions of the vulcanisate, while the latter is dependent on these conditions. It is therefore necessary to make a careful comparative study of the coefficients and the corresponding physical properties under varying conditions before the latter can be taken as a measure of the condition or state of cure. I have already shown that

\* On this point, see O. de Vries, *Archief voor de Rubber-cultuur in Ned.-Indië*, Sept., 1917.

† Thus, for example, three samples of sheet (two air-dried, one smoked) hanging alongside one another contained the following percentages of moisture, 9 a.m. (temp. 27.5°): 0.208, 0.217, 0.273.

the physical properties are dependent on the age of the vulcanised specimen,\* so that comparable results are only obtainable when the specimens are tested at a fixed period subsequent to vulcanisation. The present results show that the temperature† also has a considerable influence on the physical properties, and uniform conditions must be observed in order to obtain comparable figures.

Three typical specimens of plantation rubber were taken—namely, (1) ordinary pale crêpe, (2) ordinary smoked sheet, (3) unrolled coagulum matured for a few days and then crêped.‡ The same mixing as in previous experiments was used, consisting of 90 parts of rubber and 10 parts of sulphur. Although the results obtained with this mixing are not directly applicable to the mixings employed commercially, it has the advantage of simplicity and the large proportion of sulphur brings about considerable variations in the coefficient and physical properties with relatively small variations in the conditions of vulcanisation. Separate portions of the "mixings" were vulcanised for varying periods, as in the ageing experiments, but with shorter "intervals," viz., 15 minutes in the case of the ordinary crêpe rubber, 13 minutes for the smoked sheet, and 10 minutes for the crêpe from matured coagulum. These intervals were chosen to correspond approximately with the rate of cure of the various rubbers.

The stress-strain curves for a vulcanised rubber may be obtained either directly (by an autographic attachment to the testing machine) or by plotting the values for the loads at the corresponding

ture. The other half of the rings were placed in an insulated box in a cold place, the average temperature being approximately 10° C. which may be regarded as a cold room temperature. Some of the rings were withdrawn from the incubator and insulated box the day following vulcanisation and tested. The others were left in the incubator and insulated box for one week and then tested.

The following tables give the results obtained in the physical tests (loads in grms. per sq. mm. cross-sectional area, corresponding to final lengths of nine and ten times the original length). They also give the coefficient of vulcanisation of each vulcanised specimen calculated from the combined sulphur figure. It is known that the coefficient of vulcanisation is not affected by the age of the specimen, provided that the latter is kept at a temperature not exceeding 60°–70° C.\* and that the specimen is not overcured.† This is one of the advantages possessed by the coefficient as an index of the state of cure. The drawback to its use lies chiefly in the laborious nature of the determination and the delay in obtaining results, as the vulcanised rubber is preferably extracted during one whole week to make certain that no appreciable quantity of free sulphur remains in the rubber.

A glance at these figures shows that a difference in temperature of 18° C. produces a marked difference in the results of the physical tests. Taking the crêpe rubber (Table I.), it appears that the temperature has a greater influence on the results than the period of ageing. This is more easily seen if the results be plotted in the form of curves

TABLE I.  
*Ordinary crêpe.*

Ref. no.	Time of cure (minutes)	Final length (original = 1)	Load in grms. per sq. mm. after an ageing period of:—				Coefficient of vulcanisation
			1 day.		7 days.		
			28° C.	11·5° C.	28° C.	11·5° C.	
5685	90	10	520	504	496	488	1·85
		9	265	270	265	260	
5686	105	10	672	576	660	563	2·15
		9	368	303	340	291	
5687	120	10	883	715	824	720	2·43
		9	471	367	429	368	
5688	135	10	1100	846	1060	887	2·76
		9	572	437	543	423	
5689	150	10	1290	1060	1380	1120	3·18
		9	715	541	735	585	
5690	165	10	1520	1340	1630	1290	3·42
		9	878	636	890	598	
5691	180	10	Broke	1540	Broke	1700	3·82
		9	1060	830	—	784	

elongation or stretch of the sample. As these curves are very similar for all vulcanised rubber and sulphur mixings and, when taken for any one sample, do not intersect, their position is most conveniently indicated by the load required to produce a given elongation, or *vice versa*. For the purpose of comparing the stress-strain curves I have taken the loads when the specimens are elongated to 9 and 10 times the original length. Although a fully cured or slightly overcured rubber may not in all cases stretch to ten times the original length, most of the present specimens stretch to this extent, and they all allow of stretching to nine times the original length.

The method adopted in the present experiments consisted in vulcanising the rubber and cutting rings from the vulcanised product on the same day. Half of these rings were placed in an incubator, where they were maintained at a temperature of 28° C. or thereabouts, that is, a summer tempera-

(Fig. 1). The four upper curves represent the loads corresponding to the coefficients of vulcanisation at a final length of 900% and the lower for one of 1000%. The curves lie in pairs. The individuals of each pair approximate closely to one another, and represent the tests made with samples kept at the same temperature for one and seven days after vulcanising. In the case of the smoked sheet, SS, and crêpe from matured coagulum, MC (Fig. 2), the curves given are the averages of the two ageing periods, and show similar differences due to the difference in temperature between vulcanising and testing. In the case of crêpe from matured coagulum, the curves lie rather more closely than with ordinary crêpe. As an illustration of the considerable effect produced by moderate variations in temperature, we note that the curve for smoked sheet, SS, elongated to nine times the original length and aged at 11° C., cuts the curve for crêpe from matured coagulum, MC, aged at 28° C. (Fig. 2, the dotted line, SS, cuts the continuous line, MC). From this it follows that, over

\* This J., 1916, 872.

† See also P. L. Wormeley, "The Rubber Industry," 1914, 246 (this J., 1915, 724).

‡ Eaton and Grantham, this J., 1915, 989, etc.

\* Kolloid Zeits., 1912, 10, 299.

† This will be shown in a later publication.



a series of vulcanisations, corresponding to coefficients varying from 2 to 3, smoked sheet elongated to nine times its original length will support a load as great as or greater than crêpe from matured coagulum elongated to ten times the original length (no account being taken of temperature variations). Fig. 3 corresponds to Fig. 2 except that the results at different temperatures have been averaged so as to eliminate the effect of temperature. It will be seen that whereas the curves for crêpe, C, and matured coagulum, MC, lie close together, both are some distance from the curve for smoked sheet, SS. Figs. 1 and 2 illustrate particularly the influence of temperature on the ageing factor, while Fig. 3 illustrates the

they run approximately parallel for sheet and ordinary crêpe rubber, but show appreciable variation in the case of crêpe from matured coagulum.

A word may be said with regard to the relationship between the coefficient and time taken to vulcanise the rubber at a constant temperature. Fig. 4 corresponds to Fig. 1 and gives the loads plotted against the time of cure instead of against the coefficients. It will be noted that the curves in the two figures correspond almost exactly. If the coefficient be plotted against the time of cure, as is shown in Fig. 5 (continuous lines) for the ordinary crêpe (C) and smoked sheet (SS), the curves obtained are straight lines or as nearly straight as the limited data and relatively short

TABLE II.  
*Smoked sheet.*

Ref. no.	Time of cure (minutes)	Final length (original = 1)	Load in grms. per sq. mm. after ageing period				Coefficient of vulcanisa- tion
			1 day.		7 days.		
			28° C.	10.5° C.	28° C.	10.5° C.	
5692	80	10	623	536	782	668	1.83
		9	345	303	442	449	
5693	93	10	787	645	984	918	2.18
		9	425	355	592	567	
5694	106	10	878	762	1160	1070	2.40
		9	512	465	663	598	
5695	119	10	Broke	Broke	1390	1280	2.74
		9	616	523	788	713	
5696	132	10	1410	1185	1480	1490	2.98
		9	815	620	878	843	
5697	145	10	Broke	1320	1630	1410	3.31
		9	896	685	1040	859	
5698	158	10	Broke	1400	Broke	1520	3.58
		9	975	908	1190	940	

TABLE III.  
*Crêpe from matured coagulum.*

Ref. no.	Time of cure (minutes)	Final length (original = 1)	Load in grms. per sq. mm. after ageing period				Coefficient of vulcanisation
			1 day.		7 days.		
			28° C.	13° C.	28° C.	9° C.	
5699	30	10	265	258	485	451	1.50
		9	155	142	289	313	
5700	40	10	334	329	570	518	1.89
		9	180	167	349	278	
5701	50	10	398	358	678	582	2.15
		9	209	185	400	329	
5702	60	10	487	444	826	674	2.48
		9	241	240	472	380	
5703	70	10	653	624	958	857	2.87
		9	323	288	567	516	
5704	80	10	772	702	1110	1050	2.93
		9	366	355	639	545	
5705	90	10	995	947	1270	1180	3.13
		9	492	425	—	650	

differences shown by the three different types of rubber when stretched under varying loads at constant temperatures. It will be noted that the curves for smoked sheet approach most closely to a straight line, while those for crêpe from matured coagulum show the greatest curvature (Fig. 3). This is characteristic of the curves at all ageing periods and temperatures. The curve for smoked sheet at highest loads suddenly takes a horizontal course, but this requires confirmation. These results are not in agreement with statements that have been made as regards the comparative figures for the coefficient and the stress-strain curves. Thus, de Vries and Hellendoorn, who express the curve by means of the length at a load of 1.30 kilos. per sq. mm., state that these figures run exactly parallel to the coefficients.\* My results show that

range of vulcanisations can show. In the case of the crêpe from matured coagulum (MC) there appears to be a distinct but slight curvature convex to the abscissa (and not concave, as in the load curves, see dotted lines). This is probably due to the influence of the accelerator, which alters the character of the chemical reaction. The curve marked XX represents the load plotted against the time of cure for ordinary crêpe rubber but when aged at 11° C. and tested the day after vulcanising. Under these conditions the curvature is more marked.

The degree of error introduced by a difference of temperature corresponding to the change from a warm to a cool room, say 18° C., is brought out in Fig. 4. Ordinary crêpe under a load of, say 800 grms., corresponds to a cure of about 180 minutes when the sample is tested at 11.5° C. and 158 minutes at 28° C. If the specimens be kept for a few days before testing, the results are similar.

\* This J., 1917, 1258.

FIG. 1.

Relationship of load and coefft. for ordinary crêpe rubber. Continuous lines give loads when elongated to 10 times the original length. Dotted lines 9 times.

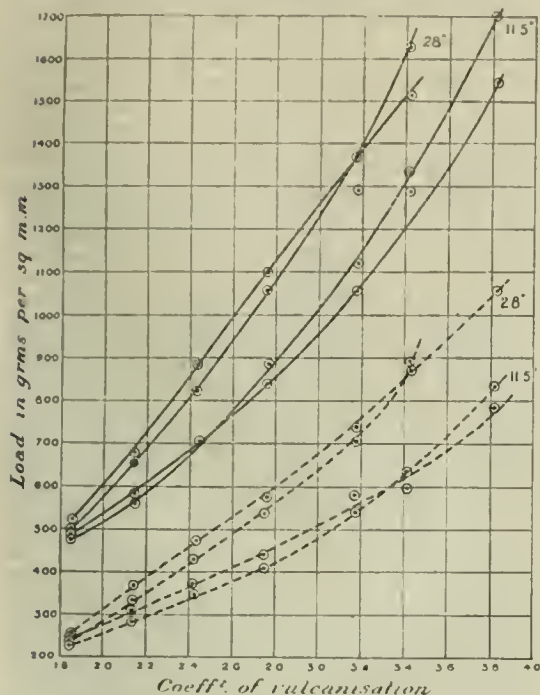


FIG. 2.

Relationship of load and coefft. for smoked sheet (S.S.) and crêpe from matured coagulum (M.C.) elongated to 10 times the original length (continuous lines) and 9 times the original length (dotted lines). All samples 7 days old when tested.

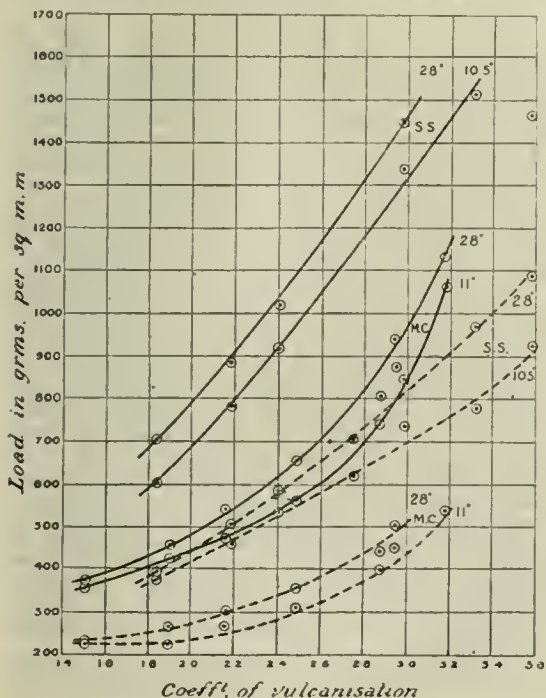


FIG. 3.

Relationship of load and coefft. for ordinary crêpe (C.), smoked sheet (S.S.), and crêpe from matured Coagulum (M.C.) aged 7 days. Average of both temperatures. continuous lines give load when elongated to 10 times the original length. Dotted lines to 9 times.

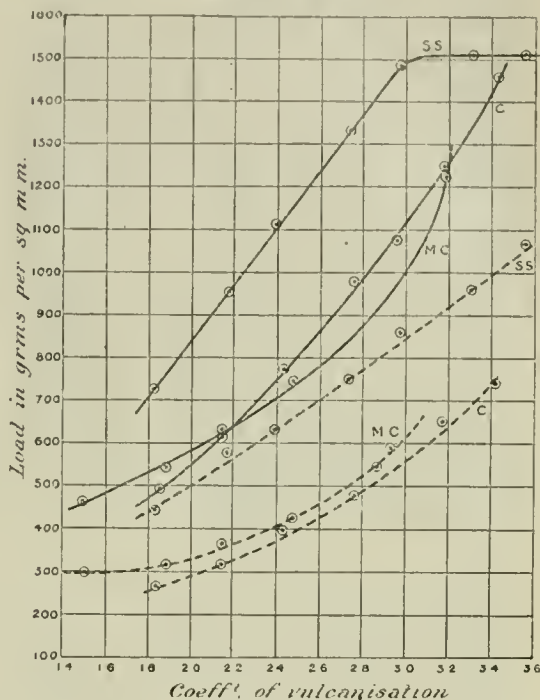
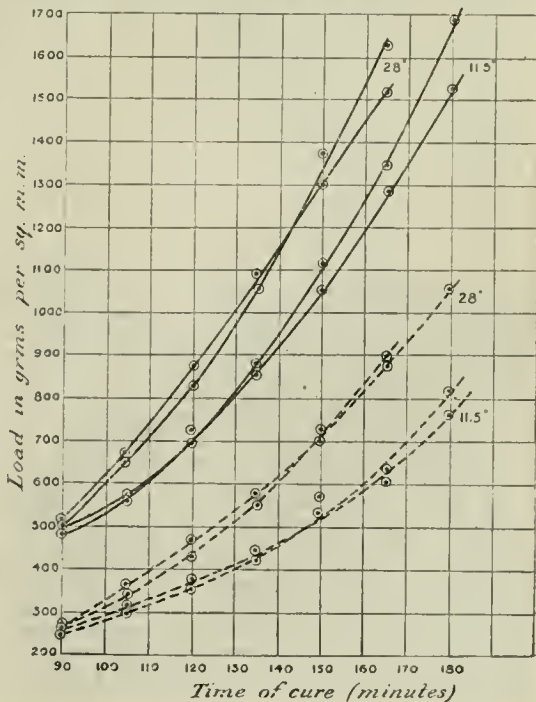


FIG. 4.

Relationship of load and time of cure for ordinary crêpe rubber, elongated to 10 times the original length (continuous lines) and 9 times the original length (dotted lines).



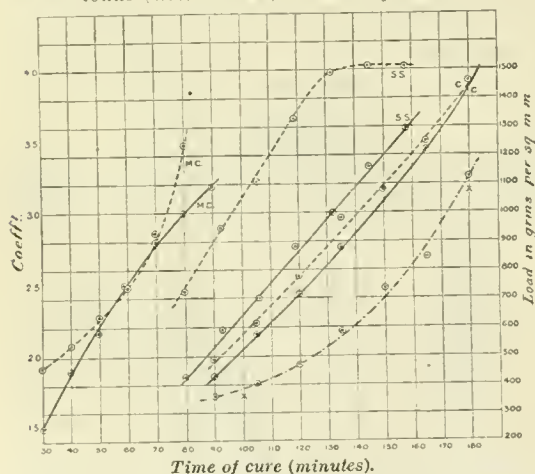


How far the figures in the tables are typical of the three types of rubber remains to be seen, but as each of the three samples consists of a mixture of several specimens of raw rubber they should be fairly representative. There are some irregularities in the experimental results, particularly at the higher cures, but the figures as illustrated by the curves in Figs. 1, 3, and 4 leave no doubt as to the trend of the results and illustrate the very considerable variations produced at short ageing periods by moderate fluctuations in temperature. They also show, as was foreshadowed in a previous paper, that the stress-strain curve corresponding to any particular coefficient exhibits appreciable variations between different types of rubber, even when the conditions of vulcanising and testing are the same (Fig. 3). It is probable that a close connection will be found to exist between the curve and the rate of cure when samples are all of the same type, *e.g.*, crêpe containing about the same percentage of caoutchouc. Where the non-caoutchouc ingredients are greater there is an effect similar to that produced by compounding the rubber with foreign ingredients. This will result in a reduced final length corresponding with the capacity to support a greater load at a given elongation. Thus, crêpe rubbers containing more caoutchouc than sheet, should give curves coming lower down on the diagram, which is actually the case as shown in Fig. 3.

Fig. 5 gives both the coefficients and the loads plotted against the time of cure. As in the case

FIG. 5.

Relationship of coefft. (continuous lines) and loads (dotted lines) to time of cure.



of the coefficients, the load curves for ordinary crêpe (C) and smoked sheet (SS) are almost straight lines, but the curve for crêpe from matured coagulum (MC) shows an appreciable curvature but in the reverse direction. It is also interesting to note that whereas the curves for the ordinary crêpe (C) and crêpe from matured coagulum (MC) lie close to one another, those for the smoked sheet (SS) are a considerable distance apart. The reason for this has already been suggested, namely, the larger proportion of non-caoutchouc constituents present in the sheet. The loads supported at a given elongation are all greater in the case of sheet rubber than would be expected from the time of cure and the coefficients. The discrepancy thus introduced is easily calculated. Take the three types of rubber and note the time of cure required for the rubber to support a given load, say 750 grms. per sq. mm. At this point the crêpe from matured coagulum has a coefficient of 2.5. In Fig. 5 (dotted lines) we have MC 60 minutes, SS 81 minutes, C

117 minutes. The rates of cure are therefore as  $\frac{60}{2.5} : \frac{81}{3.0} : \frac{117}{4.0} = 100 : 74 : 51$ . Now calculate the relative rate of cure of the three samples based on the coefficients (continuous lines, Fig. 5). Take the minutes required to produce a coefficient of, say, 2.5 in the crêpe from matured coagulum which corresponds to a load of 750; the figures are MC 60 mins., SS 109 mins., C 123 mins. The rates of cure are therefore  $\frac{60}{2.5} : \frac{109}{3.0} : \frac{123}{4.0} = 100 : 55 : 49$ —an appreciably different relationship from that based on the coefficient. If other points on the stress-strain curve be taken other ratios will be obtained, because the curves for crêpe from matured coagulum diverge appreciably from straight lines.

A few figures may also be given based on the graphs (Fig. 3), showing the differences which may result from the exclusive use of stress-strain curves for the purpose of estimating the state of cure without reference to the type of rubber under examination. Taking, as before, the crêpe from matured coagulum as a standard for comparison, this gives a coefficient of 3 at a load of 1000 grms. At this same load the smoked sheet gives a coefficient of approximately 2.22 and the ordinary crêpe a coefficient of 2.82. That is to say, as judged by the stress-strain curves, the three rubbers are cured to the same degree when, according to the coefficients, the smoked sheet is only 74% vulcanised and the crêpe 94% vulcanised taking the crêpe from matured coagulum as 100% vulcanised. If we take the load at 750 grms., we obtain the corresponding figures 75 and 96 for the sheet and crêpe, which are in fair agreement with the foregoing. At a load of 500 grms., however, the positions of the graphs for C and MC are reversed and the figures become 88 for smoked sheet and 112 for the crêpe, based on MC=100 as before. At higher loads, again, as for instance 1250 grms., the curves for C and MC give smoked sheet 82 and crêpe 100. It will therefore be seen that considerable differences arise when comparing different types of rubber for rate of cure, according to whether the coefficient or stress-strain curves be taken as the basis of the calculation, and this particularly applies to smoked sheet when contrasted with milled, that is ordinary crêpe rubber.

#### Summary.

(1) The position of the stress-strain curves is appreciably influenced by the period elapsing between vulcanising and testing the rubber and by the temperature. Hence the results are only comparable when these conditions are kept constant. Results obtained in the summer are not comparable with those obtained in winter, nor those obtained in the Tropics with those obtained in Europe.

(2) The position of the stress-strain curves is influenced by the type of the rubber, *i.e.*, whether crêpe or sheet, probably owing to a variation in the proportion of non-caoutchouc ingredients.

(3) The coefficient of vulcanisation is independent of the above and other conditions and is therefore a more reliable index of the rate of cure. In any case, if the stress-strain curves are to be taken as an index of the state of cure, it is essential that these curves be obtained under standard conditions.

(4) Of particular interest is the greater curvature of the graphs for crêpe from matured coagulum than of those for ordinary crêpe and sheet. This may be attributed to the larger proportion of accelerating base contained in the former.

(5) To ensure as great a degree of uniformity as possible, all specimens should be tested as soon as can be after vulcanisation—say the next day—and between vulcanising and testing the specimens should be kept as nearly as possible at say 30° C., a temperature easily maintained either in Europe or the Tropics.

I am indebted to the Rubber Growers' Association for permission to publish these results.

## London Section.

Meeting held at Burlington House on June 3rd, 1918.

DR. CHARLES A. KEANE IN THE CHAIR.

# THE DETERMINATION OF TIN IN HIGH-GRADE WOLFRAM ORES AND THE USE OF LEAD AS A REDUCING AGENT IN PEARCE'S ASSAY.

BY A. R. POWELL.

The specifications for wolfram used in the manufacture of ferro-tungsten require that the ore shall contain not more than 1% of tin; sometimes the stipulated maximum is lower still, e.g., 0.75%. A rapid and reliable method for determining tin in wolfram concentrate is, therefore, of the greatest importance for the purpose of controlling the work of the electro-magnetic separator and for testing the adaptability of the magnetic separation process to new parcels of tin-wolfram ore, as some varieties of cassiterite show marked magnetic properties.

In the volumetric determination of tin by means of iodine, even a moderate quantity of tungstic acid masks the end-point completely by the intense blue colour of the lower oxides of tungsten formed in the reduction. Hence the necessity for removing the tungstic acid before titration. Several methods to this effect have been proposed and used, but they are, on the whole, too tedious for rapid work, and not always reliable. Of the four methods described below the first three, though slower, were found to give good results, and are useful as a check. The fourth method is rapid and has proved very satisfactory. A preliminary account is also given of the use of lead as a substitute for iron or nickel in the Pearce method.

**Determination of tin in wolfram ores.** Tin occurs both as cassiterite and stannine in wolfram ores; the former is insoluble in acids and in fused bisulphate, the latter dissolves in *aqua regia*, and fusion with bisulphate renders part of the tin soluble. In case cassiterite alone is present, attack with acids or fusion with bisulphate will dissolve the wolfram and leave the tin oxide unattacked; with stannine, however, the solution must also be examined for tin. In the author's experience cassiterite is quite insoluble in *aqua regia*, but, on fusion with bisulphate, a small amount goes into solution when the melt is extracted with dilute sulphuric acid. This rarely exceeds 1 mgrm. and is often negligible.

**Method 1.** The ordinary *aqua regia* attack of the slimed wolfram ore leaves the cassiterite with the tungstic acid, and, when the latter is dissolved out with ammonia, the small residue left may be fused with sodium peroxide and the tin determined as in the well-known Pearce assay. This is the usual method for removing the wolfram, but its great drawbacks are: (a) the ore must be very finely powdered before attack; (b) the complete solution of the wolfram is a lengthy and tedious operation; (c) the extraction of the tungstic acid with ammonia or soda introduces another titration; (d) if stannine is present the acid filtrate must be treated with hydrogen sulphide, the precipitate extracted with sodium sulphide to remove copper, and the tin precipitated with acid in the filtrate from the copper sulphide, the washed tin sulphide being ignited with the residue from the ammonia extraction.

**Method 2.** Fusion of the ore with potassium cyanide was also used, but, though a much more rapid method, it was not free from objections. Thus, when many assays are being conducted at the same time, the fumes from the hot, strong cyanide solutions are apt to be dangerous. Again, the wear on the porcelain crucibles is very great, one crucible rarely surviving more than three fusions. The analysis is conducted as follows: 5 grms. of cyanide is gently fused in a porcelain crucible and allowed to cool again until a crust forms, on to which 1 gm. of ore is gently transferred. The crucible is covered and the temperature raised slowly until vigorous effervescence ceases, after which the contents of the crucible are stirred gently and heated for five minutes longer. After cooling, the melt is extracted with hot water and the insoluble residue, consisting mainly of metallic iron and tin, filtered off, washed free from cyanide with hot water, and dissolved in hydrochloric acid and a little potassium chlorate. The resulting solution is ready for reduction and titration. If the ore contains arsenic, copper, or bismuth, it must be treated with 20 c.c. of nitric acid (sp. gr. 1.2) prior to fusion. The insoluble matter, which contains all the tin, is filtered off, well washed with hot water, ignited, and fused with cyanide. Although the method gave, on the whole, reliable results, inexplicably low figures were obtained at times, so that, while useful as a check, it did not answer well as a control method.

**Method 3.** The possibility of separating the two metals after getting them into solution as sodium tungstate and stannate was next investigated. Auger (Z. angew. Chem., 1906, 19, 140) has described a method in which the tungsten is precipitated as blue oxide by means of zinc and hydrochloric acid, but this method was found to be slow and the separation imperfect. As in the separation of molybdenum from tungsten, it appeared possible to separate the tin from a tartaric acid solution of the sodium salts by precipitation with hydrogen sulphide. It also seemed probable that boiling the alkaline solution of the two metals with ammonium nitrate would precipitate the tin as metastannic acid. Both the above procedures were found workable, as the following experiment shows:—0.5 gm. of pure tin oxide was fused with 5 grms. of sodium peroxide in an iron crucible, the cold melt extracted with boiling water, the solution cooled, made up to 500 c.c. and filtered through a dry filter. A number of 20 c.c. trials (=0.0158 gm. Sn) were pipetted into small flasks and treated as follows:—(a) The solution was acidified with hydrochloric acid and reduced direct. (b) The solution was acidified with tartaric acid and saturated with hydrogen sulphide; the precipitate was filtered off, washed, dissolved in hydrochloric acid and reduced. (c) The solution was treated with 5 grms. of ammonium nitrate, boiled till free from ammonia, the precipitate filtered off, washed, dissolved in hydrochloric acid and reduced. (d) The solution was treated as under (c) but the precipitate was ignited and fused with peroxide as in Pearce's assay. The amounts of tin found were (a) 0.0157, (b) 0.0154, (c) 0.0153, (d) 0.0158 gm.

The precipitation with hydrogen sulphide generally gave slightly low results, ascribed to loss of colloidal sulphide in washing. The method as applied to ores is as follows: 2.5 grms. of ore is fused with 10–12 grms. of sodium peroxide in an iron crucible in the usual manner. The cold melt is extracted with water and a little alcohol, the liquid diluted to 250 c.c. in a graduated flask and 200 c.c. filtered through a dry filter. The solution is transferred to a beaker and 5 grms. of tartaric acid added, followed by hydrochloric acid to produce a distinctly acid reaction. The hot solution is saturated with hydrogen sulphide, the



precipitate filtered off and washed with warm 2% sodium acetate saturated with hydrogen sulphide. If arsenic is absent the precipitate is dissolved directly in hydrochloric acid, but, if present, the precipitate is ignited in an iron crucible at a very low temperature and the residue fused with peroxide.

Precipitation with ammonium nitrate is carried out by boiling the alkaline filtrate, obtained as above, with 20 grms. of ammonium nitrate and a little filter pulp until the smell of ammonia is barely perceptible. The precipitate is allowed to settle, filtered off, and well washed with hot 2% ammonium sulphate; it may either be extracted with hydrochloric acid and the filtered solution reduced as usual, or ignited and fused with peroxide. Arsenic, if present, is carried down by the metastannic acid and may cause trouble in the reduction and titration. It should, preferably, be removed by a preliminary digestion of the ore with nitric acid. The methods given under (3) work equally well when the tin is present in the form of stannine as when present as cassiterite.

4. *Author's method.* After removal of the wolfram by fusion of the ore with bisulphate, as usually carried out, the melt is heated with dilute sulphuric acid, which dissolves iron, manganese, and titanium sulphates but precipitates the tungstic acid in a white flocculent form which is difficult to filter and wash free from iron salts. Consequently, on leaching the residue with ammonia, ferric hydroxide is formed, which retains tungstic acid, and re-precipitation is impracticable when time is an important factor. Caustic soda readily dissolves tungstic acid; it also precipitates iron, but in the presence of sufficient tartaric acid, both metals remain in solution. It was thus found possible to obtain a clean residue in one operation; further work showed that addition of caustic soda was unnecessary, since hot 5% tartaric acid readily dissolved the fused cake without separation of tungstic acid.

The exact details of this method, which has proved its superiority, after nearly three years' continual use, are as follows: 1 gm. of the sample, crushed to pass a 90-mesh sieve, is mixed with 5 grms. of previously fused bisulphate in a capacious silica crucible, and the mixture heated, gently at first, finally to a red heat. The crucible

is cooled by standing it in a shallow basin of cold water (taking care that no water enters the crucible); when nearly cold, it is covered with a watch-glass to avoid loss by the splintering of the melt. The crucible is half filled with hot 5% tartaric acid and gently warmed over a small flame, whereby the melt is readily detached. It is then transferred to a beaker, together with 70–80 c.c. of tartaric acid solution, and the whole boiled until the cake has completely disintegrated and all soluble matter dissolved. Another method of removing the melt from the crucible consists in pouring it on to a cold iron or nickel plate. When cold the mass is boiled with 5% tartaric acid in a beaker and what remains in the crucible rinsed out with the same liquor into the main solution. The insoluble material is filtered off on a Whatman No. 1 or 2 filter, well washed with hot water, ignited wet in an iron crucible, and fused with 2 grms. of sodium peroxide. In washing out the crucible and beaker care should be taken that all the tinstone, which has a great tendency to hang back, is washed out.

It was soon found that some ores give slightly low results by this method, and this was traced to solution of a little tin in the tartaric acid liquor; it may be recovered by a hydrogen sulphide treatment, the tin being precipitated as sulphide and filtered off, together with the insoluble matter. Gentle ignition converts the sulphide to oxide without loss.

Ilmenite, columbite, and monazite, if present, are dissolved by the bisulphate fusion and subsequently dissolve in the tartaric acid liquor. Scheelite dissolves completely and no separation of calcium sulphate occurs. If much copper, bismuth, or molybdenum is present, the ore should first be treated with 20 c.c. of nitric acid (sp. gr. 1.2), the residue filtered off, washed with hot water, ignited wet, and fused with bisulphate. Arsenic is volatilised on igniting the residue from the fusion, but large quantities of mispickel or iron pyrites cause much effervescence and consequent danger of loss during the fusion, so that it is better, in these cases, also, to give the ore a preliminary treatment with nitric acid.

Table 1 gives a comparison of the results obtained by the above methods. After the removal of the wolfram, the stannic chloride solution ob-

TABLE 1.

Ore	Composition %	Method 1	Method 2	Method 3		4. Author's method		
		<i>Aqua regia</i> % Sn	Fusion with NaCN % Sn	Fusion with Na <sub>2</sub> O <sub>2</sub>		Total Sn %	Bisulphate fusion Separate determination of Sn	
				Tin pptd. by H <sub>2</sub> S % Sn	Tin pptd. by NH <sub>4</sub> NO <sub>3</sub> % Sn		in insoluble residue	recovered by H <sub>2</sub> S
A	WO <sub>3</sub> , 70.4; Cu, 0.25; As, 0.16; con- tained stannine ... ..	0.35	0.37*	0.33	0.37	0.37*	0.24	0.16
		0.36	0.34*	0.30	0.37	0.35*	0.24	0.11
B	WO <sub>3</sub> , 72.9; As, 0.08; Bi, 0.07 ...	0.61	0.59	0.53	0.55	0.60	0.59	nil
		0.59	0.55	0.57	0.51	0.59	0.59	nil
C	WO <sub>3</sub> , 65.4; As, 1.19; Cu, 0.24 ...	1.97	1.90*	1.93	1.95*	2.00*	1.83	0.14
		1.97	1.95*	1.92	1.79*	1.96*	1.89	0.06
D	WO <sub>3</sub> , 63.8; As, 0.22; Bi, 0.96 ...	7.10	6.97*	7.02	6.95	7.05*	6.78	0.29
		7.08	7.04*	7.07	7.09	7.07*	6.82	0.20
E	WO <sub>3</sub> , 74.3; As, nil; Cu, Bi, traces	0.12	0.08	0.10	0.12	0.14	0.12	nil
		0.12	0.10	—	—	0.12	0.11	nil
F	Synthetic mixture ... ..	5.48	5.40	5.42	5.45	5.46	5.44	nil
		5.45	5.47	5.44	5.36	5.46	5.41	trace

\* The ore was given a preliminary treatment with nitric acid.

tained either by fusion or solution was reduced in a round flask with three iron nails for 30 minutes after the colour of the ferric chloride had disappeared. The reduced solution was cooled in running water after the addition of a piece of clean marble, the nails removed, washed with air-free water, and the solution titrated with a weak solution of iodine (1 c.c.=0.002 gm. Sn approx.). The iodine was standardised against pure tin, and due allowance made for the amount of iodine consumed by the nails (this correction never amounted to more than 0.3 c.c. of the iodine solution).

The synthetic mixture F was made by slining together 20 grms. of a pure crystal of wolfram and 1.6 gm. of a pure crystal of tinstone.

*Note on the use of lead as a reducing agent in the tin assay.*

The objections to the use of iron in the reduction of tin solutions are: (a) the blank; (b) the greenish tint of the reduced solution which tends to obscure the delicate end-point obtained with weak iodine, which is, therefore, sometimes difficult to discern within one drop. Search was therefore made for a reducing agent which would give colourless solutions, and this was found in lead, the separation of the chloride being almost entirely obviated by the addition of common salt to the solution and by increasing the strength of the acid so as to give the most rapid reduction possible. The procedure is as follows: The peroxide melt is leached with hot water into a 250 c.c. round flask, using as small a quantity of water as possible, and the solution made just acid with hydrochloric acid. After diluting to 100 c.c., 20 grms. of common salt, 40 c.c. of strong hydrochloric acid, and 10 grms. of granulated lead are added, a funnel is inserted in the neck of the flask and the solution boiled vigorously for 20 minutes after the disappearance of the ferric chloride colour. A piece of clean marble is then dropped in, the solution cooled in running water, diluted with 50 c.c. of cold, boiled water containing a pinch of sodium bicarbonate and a slight excess of hydrochloric acid, and rapidly titrated with iodine without removing the lead. The end-point appears suddenly and may be accurately ascertained to one drop. A few crystals of lead chloride may separate, but they settle readily and do not in any way obscure the end-reaction. In order to ascertain whether the presence of lead during titration would lead to high results, several of the titrated solutions were gently shaken for half an hour and again titrated. In no case was more than 1 c.c. required, hence it may be assumed that during the original titration which usually took less than a minute owing to the small quantities of tin present, the back-reduction, due to the presence of lead, was equivalent to less than one drop of iodine and, therefore, negligible. With regard to the time of reduction, 20 minutes' vigorous boiling is ample for quantities less than 0.05 gm. of tin; for quantities up to 0.1 gm. 30 minutes' is quite sufficient. A certain amount of time is, of course, consumed in reducing the ferric chloride formed from the iron crucible: this generally occupies between 10 and 20 minutes and varies inversely with the quantity of tin in solution.

To reduce further the time taken in the reduction a preliminary treatment with zinc was tried (*cf.* Hutchin's zinc-nickel reduction, *Bull.* 149 *Inst. Min. Met.*, Feb., 1917; *this J.*, 1917, 293) with excellent results. The best method of effecting this was found to be as follows: The cold peroxide melt is leached with hot water, and hydrochloric acid slowly added until all the ferric hydroxide has dissolved giving a clear solution, excess of acid being avoided.

2–3 grms. of zinc dust is sprinkled into the warm solution which is vigorously agitated for a short time. The iron is almost immediately reduced, while the tin is precipitated as metal. The solution, which should occupy a bulk of about 100 c.c., is treated with 20 grms. of common salt, 34 c.c. of pure hydrochloric acid, and 5 grms. of granulated lead, heated to boiling, boiled ten minutes, a small piece of clean marble dropped in, and the solution cooled, diluted, and titrated as before. During the final boiling the precipitated tin dissolves almost immediately; the boiling with lead ensures that no oxidation occurs.

For comparison, reduction with zinc followed by boiling with iron nails instead of lead was tried, the nails being removed before titration. Good results were obtained and the smaller amount of iron in solution enabled the end-point to be determined very sharply. It was found that small amounts of arsenic did not interfere in the zinc-iron reduction, but the effect with lead has not yet been definitely ascertained.

The following table gives results obtained by the different methods of reduction. All the ores were given a preliminary bisulphate fusion to remove the wolfram and the residual tin oxide was fused with sodium peroxide. The standard was reduced in each case in exactly the same manner as the assay.

TABLE 2.

Solution reduced with	Iron	Lead	Zinc-iron	Zinc-lead
	Sn %	Sn %	Sn %	Sn %
Ore E ... ..	0.14	0.11	0.12	0.12
	0.12	0.11	0.10	0.14
Ore F ... ..	5.46	5.41	5.45	5.4
	5.46	5.45	5.48	5.47
Ore G ... ..	0.66	0.66	0.68	0.66
	0.64	0.66	0.64	0.63
Ore H ... ..	1.68	1.63	1.70	1.67
	1.68	1.65	1.66	1.67

#### DISCUSSION.

Dr. W. R. SCHOELLER said that he had found the bisulphate-tartaric acid method to work splendidly; the fusion and leaching could be carried out in 10–15 minutes. He had not tried lead as a reducing agent in the tin assay, but it was desirable to use a metal which gave a colourless solution. Iron gave a fairly pale solution, but was not always sufficiently pure; the same objection held good for nickel, which was certainly not to be recommended for small quantities of tin on account of the dark green colour of the solution. Antimony, which was also used as a reducing agent, gave no coloured compound, but its purity was often doubtful, besides which the metal obtained by the precipitation process contained tin, derived from the tinned iron used. If it was proved that lead was a good reducing agent for large quantities of tin, its use would become more general, as it was one of the purest metals obtainable in commerce.

Mr. C. E. BARRS asked whether reduction with lead had been tried on solutions containing larger amounts of tin than those mentioned. There might be one drawback to the use of lead, and that was the formation of lead iodide when titrating. Perhaps this could be overcome quite as easily by cooling with ice water as by the use of sodium chloride, provided that the vessels would stand the sudden change of temperature. He thought reduction with iron was much to be preferred to the use of nickel; it was necessary, however, to



see how much iodine was consumed in a blank determination, and if the amount was large he always rejected such iron.

Mr. G. NEVILL HUSTIE said that another metal which could be used for the reduction in Pearce's assay and which gave a colourless solution was aluminium. On quantities of 0.1 gram. of tin, reduction with aluminium and with antimony (Kahlbaum's purest) gave the same figures. He agreed that the colour produced with nickel was an objection.

Prof. PEARCE said that in estimating small quantities of tin by the Pearce method and using both iron and nickel as reducing agents he had obtained erratic results varying with the acidity of the solution. The amount of tin found appeared to diminish with the acidity of the solution. Had the author observed whether the results obtained were to any extent dependent upon the amount of hydrochloric acid present?

Mr. GAMMIE suggested that magnesium might satisfactorily be used for reduction.

Mr. CHAPMAN said that he had obtained very good results with certain batches of nickel, but, as had been mentioned by Mr. BARRS in regard to iron, it was necessary to test the material used. The last batch of nickel which he had used proved unsatisfactory and he believed this was entirely due to the presence of cobalt.

Mr. POWELL, in reply, said he was at present engaged in trying lead reduction on larger quantities of tin; he had obtained some good results in solutions containing up to 0.25 gram. of tin, and he hoped to be able to communicate some of the results later. As to lead chloride separating, the addition of sodium chloride prevented its deposition in any quantity. He thought that aluminium and magnesium would be objectionable as reducing agents on the ground that they would precipitate the metal, which would then have to be dissolved again, without any reducing agent being present, to prevent oxidation. With antimony he always found that he got higher results than with other agents, although the results were concordant amongst themselves. This point was at present under investigation. His experiments so far seemed to show that, in the case of lead, an excess acidity of the solution had no effect. He usually worked with one-third of the bulk of the solution in hydrochloric acid and never found any difficulty with any variety of ore. Many of them which were sent as wolfram ores had been found to contain only 1 or 2% of wolfram and the rest all sorts of minerals (e.g., ilmenite, tourmaline, columbite, etc.), but it did not make any difference as the bisulphate-tartaric acid method cleaned out all objectionable impurities.

## Communications.

### THE MANUFACTURE OF CHLORAMINE T.

BY J. K. H. INGLIS.

Although two years have elapsed since Dakin showed the value of chloramine-T as an antiseptic, no detailed account of the method of manufacture has yet been published. The drug has shown itself of great value in cases of cerebrospinal meningitis, diphtheria, etc., and after a trial of a small specimen which I had made, I was asked to supervise the manufacture of a regular supply for the military camps in New Zealand, it having been found impossible to procure it outside the Dominion.

In Dakin's work the starting point for the preparation was *p*-toluenesulphonic chloride, obtained as a by-product in the manufacture of saccharin. No such material being available in New Zealand, details had to be worked out for the manufacture from toluene itself, and these details form the material for this paper.

#### Sulphonation.

The sulphonation of toluene with concentrated sulphuric acid leads to the formation of the three possible monosulphonic acids; but the higher the temperature the larger the proportion of the *p*-acid (Ber., 1911, p. 2504), the highest proportion being 70 to 80%. The price of sulphuric acid being relatively high in New Zealand, I found it best to use two parts by weight of sulphuric acid (sp. gr. 1.84) to one part of toluene (approximately equal volumes) and to carry out the sulphonation at the boiling point of toluene. 250 c.c. of sulphuric acid (1.84) is heated to 110° C. by immersing the reaction flask in a heated paraffin bath, and then 250 c.c. of hot toluene is added, the whole being very vigorously stirred with a glass circulating stirrer. The efficiency of the stirring is very important, as upon it depends the time necessary to complete the sulphonation. This reaction was carried out in a flask fitted with a reflux condenser, and only a small loss of toluene vapour took place, sulphonation being complete in about thirty minutes with very little charring. Under these conditions about 75% of the toluene is converted into the *p*-sulphonic acid, the remainder being a mixture of *o*- and *m*-acids. If this mixture of acids was isolated, converted into dry sodium salts, and treated with phosphorus pentachloride, the *p*-chloride could be obtained; but as the other chlorides would be useless there would be a considerable waste of material. We therefore made use of Lange's discovery (Ger. Pat. 57,391) that if water is added so that the mother liquid consists of only 65% sulphuric acid, then on cooling, the crystals that separate consist only of the *para*-acid which separates almost completely—the mother liquid retaining the *o*- and *m*-isomers in solution. In order to carry this out, 45 c.c. of water is added to the reaction mixture after sulphonation as above and the liquid poured out and cooled. The liquid rapidly crystallises and sets to a crystalline cake, and on filtering over an asbestos mat (as in a Gooch crucible) nearly the whole of the mother liquid can be drained away, leaving a white crystalline mass which proves to contain only small amounts of *o*- and *m*-impurities. The crystals that separate appear to be the monohydrate,  $C_6H_4SO_3H.H_2O$ , and correspond to about 75% of the toluene taken. Hence about 25% of the toluene is either lost as vapour or is contained in the mother liquor in the form of the *o*- and *m*-acids. This separation by crystallisation is, however, not very sharp, and the mother liquid on standing continually deposits further crops, which, however, are nearly free from the *para*-acid.

The *o*- and *m*-acids being useless to us, experiments were made by Mr. C. L. CARTER at my suggestion, to see how completely the toluene could be recovered by the action of superheated steam. It was found that if the liquor were heated to about 170° and then a brisk current of steam at 200° were blown through it the bulk of the toluene was rapidly evolved, the distillate containing at first as much as 20% of toluene. When the percentage fell below 5% the operation was stopped, as further continuance was hardly profitable (cf. Armstrong, J. Chem. Soc., 1884, 45, 138). The total amount thus recovered was about 14%, leaving 11% not accounted for. Part of this is lost by evaporation during mixing and subsequent sulphonation, and a small part is left in the sulphuric acid. Probably most of the remainder is lost by evaporation *in vacuo* during the filtration of the crude acid.

The cake of crude *p*-acid is dissolved in water, slightly more than enough calcium carbonate is added to precipitate the sulphuric acid as calcium sulphate, and the still acid liquid is filtered. It is then nearly neutralised with caustic soda, made distinctly alkaline with sodium carbonate, boiled, and again filtered. It is important to remove the calcium as completely as possible as it may become a troublesome impurity in the later stages. This method gives a solution containing only sodium sulphamate and sodium carbonate, and on acidifying with hydrochloric acid and evaporating to dryness there remains a pure white salt consisting of the required sodium sulphamate with a trace of sodium chloride. The sulphamate is so soluble in water that this seems to be the best method of preparation.

#### *Preparation of the sulphonic chloride.*

This can easily be prepared by heating together on the water bath approximately equal weights of the sodium salt (dried at 130°C.) (191 grms.) and phosphorus pentachloride (288.5 grms.). In this case the reaction takes place very rapidly and easily and a large amount of phosphorus oxychloride is obtained. Mr. C. S. Hicks, working in my laboratory, found, however, that the oxychloride itself can be used according to the equation,  $2\text{XSO}_2\text{Na} + \text{POCl}_3 = 2\text{XSO}_2\text{Cl} + \text{Na}_2\text{PO}_3 + \text{NaCl}$ , and he obtained a 76% yield. Hence if less than half the weight of pentachloride be taken and the mixture be heated some hours on the water-bath under reflux condenser the following reaction takes place:— $3\text{XSO}_2\text{Na} + \text{P}^{\text{V}}\text{Cl}_5 = 3\text{XSO}_2\text{Cl} + 2\text{NaCl} + \text{NaPO}_3$ . The proportions of salt and pentachloride according to this equation are 573 to 298.5, or 2 $\frac{1}{2}$  to 1; but the reaction is very slow towards the end as the mass becomes too solid and the yield is apt to decrease. This trouble might be avoided by using the theoretical proportion of pentachloride and sufficient oxychloride, which should then be recovered unchanged. An experimental trial of this gave 92% yield but the oxychloride was not recoverable. We therefore use 2 parts of sodium salt for each part of pentachloride and the action then completes itself more easily and only small amounts of oxychloride are left over—the reaction being allowed to go on over-night under reflux condenser on a water-bath. The oxychloride is then distilled off *in vacuo* and the residue put into cold water. The sulphonic chloride is the only part insoluble and is separated by filtration: a small amount of oily impurity (*o*- and *m*-sulphonic chlorides) can be pressed out from the solid cake. The yield is nearly quantitative, 96–99%. The solid sulphonic chloride still contains small amounts of *o*- and *m*-impurities, but these are gradually eliminated in the later stages.

#### *Preparation of sulphonamide.*

This operation, though quite simple in theory, causes a certain amount of trouble without special apparatus. The alternative methods are treatment with ammonium carbonate on the water-bath and treatment with strong ammonia solution. The latter method, if properly controlled, is decidedly the more satisfactory. If the sulphonic chloride is ground to a fine powder and concentrated ammonia is poured upon it, a violent reaction begins almost at once and the liquid boils. In this way considerable amounts of ammonia are driven off and the action is apt to be incomplete. We had some difficulty in procuring vessels in which the reaction could be conveniently carried out under pressure, and owing to the impossibility of getting special vessels made which would stand the action of a hot mixture of ammonia and ammonium chloride, we used stoneware ginger-beer bottles which had a screw stopper with a rubber

sealing. 100 grms. of sulphonic chloride could be placed in each, 100 c.c. of 0.880 ammonia quickly added and the stopper screwed in firmly. On shaking vigorously the action began, and as soon as the bottle became too hot to hold it was placed in cold water. No mishaps occurred and the yield was excellent, the liquid only requiring filtering and the residue washing with cold water. The mother liquors on evaporation give small amounts of the *m*-amide but very little else besides the ammonium chloride. Under these conditions about half the ammonia taken is actually used and on a large scale the unchanged ammonia could be recovered by boiling the liquors. The yield of *para*-sulphonamide is 94–97%.

#### *Preparation of chloramine-T.*

The conditions for the conversion of amide to chloramine were given in detail by Dakin, one mol. of amide being dissolved in 1.2 mols. of sodium hypochlorite of definite concentration and the chloramine then precipitated by the addition of strong brine. At first his actual proportions were used, but it was found to be unnecessary to use brine. If the amide is dissolved in a warm, strongly alkaline solution of sodium hypochlorite of concentration 1.3 to 2.0 N, a large crop of chloramine is formed on cooling and the remainder can easily be obtained on evaporation. It is very important to have a decided excess of caustic soda, and for each mol. of amide we usually take 1.05 to 1.1 mols. of hypochlorite and 1 mol. of sodium hydroxide. The preparation of the hypochlorite is of course straightforward. Owing, however, to the cost of acids in New Zealand and to there being no electrolytic source of chlorine, we found it most convenient and cheapest to use bleaching powder as the starting point. The crude chloramine-T obtained in this way is contaminated with sodium chloride; but even the crude crystals on drying contain 92–95% of chloramine. The substance can be easily purified by recrystallisation from water. If dissolved in twice its weight of hot water, a large proportion can be obtained in the pure form (over 98%) on cooling. The final filtration is apt to be troublesome; the crystals do not pack well on the filter and it is therefore difficult to remove the mother liquor completely. If, however, the cooling is carried out rapidly without stirring a different shape of crystal is obtained which filters much more readily. It is thus possible, working with large quantities, to get a product of over 99% purity.

#### *Utilisation of by-products.*

In the sulphonation as described most of the toluene is used, but half the sulphuric acid taken is contained in the mother liquors after removal of the toluene by superheated steam. At first it was hoped that it would be possible to recover the acid in the pure state by distillation and use it over again; but there is always some charring and the impurities remaining make it comparatively useless. It contains 34% of water. In the remaining stages of preparing the sulphamate the by-products are small in quantity and comparatively valueless. Some lime or calcium carbonate is used and converted into sulphate, but the quantity is very small. In the preparation of the sulphonic chloride the by-products are sodium chloride and sodium *ortho*- and *meta*-phosphate. It would probably be worth while to utilise the latter on the large scale but our experiments are incomplete. In the preparation of the sulphonamide the chief by-products are ammonium chloride and excess of ammonia. The former can be easily recovered and the ammonia could be made use of with suitable apparatus.

In the preparation of hypochlorite and of chloramine, the by-products are calcium carbonate—a



nearly pure precipitate—and a liquor consisting of sodium chloride and hydroxide. No doubt use could be made of this liquor but we have made no attempt in this direction. The above results have been obtained under laboratory conditions making some pounds of chloramine per week. Owing to lack of filtering apparatus and other labour-saving devices we have not made experiments on a still larger scale; but our experiments have always been made with a view to their application to large-scale operations and we have every reason to believe that they would not fall in commercial practice.

*University of Otago,*

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# A SIMPLE AND RAPID METHOD FOR THE ESTIMATION OF ALCOHOL IN SPIRITUOUS LIQUORS.

BY NAOENDRA CHANDRA NAO, M.A., F.I.C., AND  
PANNA LAL, M.SC.

The method for the estimation of alcohol described below is the result of an investigation to devise a simple method for its estimation with a fair degree of accuracy, avoiding distillation.

The method consists in treating a known quantity of spirituous liquor in a glass tube graduated in tenths of a c.c. (or finer graduation if procurable) with an excess of anhydrous potassium carbonate, adding about 5 to 10% of water in case the percentage of alcohol is above 90. The mixture is then thoroughly shaken and allowed to settle (or preferably centrifuged), when it will separate into a lower layer of solid potassium carbonate, a middle layer of saturated solution of potassium carbonate, and an upper layer of alcohol hydrate corresponding with the formula  $4C_2H_5OH, H_2O$ , as will appear from the experimental results given below. The aqueous potassium carbonate solution (middle layer) contains 0.00275 vol. of alcohol hydrate per c.c.

The composition of the alcohol hydrate was determined by density determinations by weighing, as well as by the glass hydrometer at 15.6° C. The results for specific gravity were 0.81961 and 0.8200 respectively, corresponding with 94.04 and 93.92% of alcohol by volume. The formula  $4C_2H_5OH, H_2O$  assigned to the hydrate requires 94.061% of absolute alcohol by vol., 91.089% by weight. The alcohol hydrate does not leave any solid residue on evaporation, showing that it does not dissolve potassium carbonate. The coefficient of expansion of the alcohol hydrate as determined by a weight thermometer was 0.001076; calculated theoretically by extrapolation from Tralles' Table II. the value 0.001068 was obtained.

The formula obtained for calculating the per-

centage of alcohol as the results of experiment and on theoretical grounds is as follows: Percentage of alcohol =  $(V + v \times 0.00275)[1 - 0.001068(t - 15.6)] \times 0.7936 \times 94.06 \div W$ , where  $V$  = volume of alcohol hydrate directly read off in the graduated tube in c.c.,  $v$  = volume of the saturated potassium carbonate solution (middle layer),  $t$  = temperature observed during the experiment in °C.,  $W$  = weight of the sample taken in grms., 0.00275 is the solubility (in c.c.) of the alcohol hydrate per c.c. of the saturated potassium carbonate solution, as actually found by experiment, 0.001068 is the apparent coefficient of expansion of the alcohol hydrate, 0.7936 is the specific gravity of absolute alcohol (15.6°/15.6°; it is assumed that the graduation of the glass apparatus used had been carried out at 15.6° C.), and 94.06 is the percentage by volume of absolute alcohol present in the alcohol hydrate liberated (upper layer). (This corresponds to percentage composition by weight, alcohol 91.089, water 8.911, the corresponding density being 0.819514.)

If the volume of saturated potassium carbonate solution is less than 2 c.c. the correction for the solubility of alcohol hydrate in potassium carbonate solution may be dispensed with as it does not appreciably affect the result.

In order further to verify the formula given above, 5 c.c. of Merck's alcohol (marked "absolute alcohol, sp. gr. 0.795") was mixed with saturated potassium carbonate solution, some solid carbonate also being added. The strength of the alcohol as calculated by our formula was 99.46%; the specific gravity observed by means of a gravity bottle was 0.79536, corresponding to 99.44% by weight of alcohol.

The percentage of alcohol found in a sample of whisky was 39.45 using our method, and 39.47 by distillation (using ice).

Numerous estimations have been made in alcohol solutions and spirituous liquors by this simple method and in many cases the results have been tested and confirmed by hydrometer readings. The results obtained were almost identical by the two methods. The point at which there is some slight disagreement between our experimental figures and those of published tables (Tralles' tables), is where the apparent coefficient of expansion of the alcohol hydrate is 0.001076. This might be urged as additional evidence for the existence of this hydrate.

In conclusion it may be mentioned that the method is quite accurate even though not more than 5 c.c. of the liquor under examination be used. Solids in solution do not affect the result. Loss by evaporation is prevented, as distillation is avoided and readings are taken in closed tubes. Ice is not required even if the temperature be high. This method is equally applicable to methyl alcohol.

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*Benares, India.*

Received Aug. 26, 1918.

## London Section.

*Meeting held at the Rooms of the Royal Society of Arts on November 4th, 1918.*

DR. CHARLES A. KEANE IN THE CHAIR.

### THE ALSACE POTASH DEPOSITS AND THEIR ECONOMIC SIGNIFICANCE IN RELATION TO TERMS OF PEACE.

BY PAUL KESTNER, PRESIDENT OF THE SOCIÉTÉ DE CHIMIE INDUSTRIELLE.

#### INTRODUCTION.

No greater pleasure could be accorded to me than that of bringing you the cordial greeting of the chemists and manufacturers of France.

In addressing you there is added to the natural satisfaction caused me by the honour of being the interpreter of my French colleagues an impersonal and loftier feeling of joy and pride, which arises from the character of this assembly.

Is not this meeting, which has a certain solemnity due to the circumstances, an outward and visible sign of the Entente Cordiale? Is it not also the manifestation of a community of effort and of aspirations the main principle of which has inspired the action of the British and the French peoples and will inspire their economic policy?

With admirable unanimity they have affirmed their intentions. During four years their friendship has been sealed by the heroism of sacrifice on the battlefields of France and the East. To-day it shines in the splendour of a decisive victory. Without doubt, even before this frightful conflict which is convulsing the world and will surely transform it, the men of science and the manufacturers of the two countries were interested in each other's work and followed each other's researches and discoveries attentively. Henceforth, whilst respecting the free expansion of the genius of either nation, they will unite their experimental activities in a co-operation which the sincerity that springs from a common ideal will consecrate. They will set on record their resolve to contribute by scientific and technical labours to the magnificent and historic work of inaugurating the reign of justice and peace. They will pursue this object with the noble ambition of rendering some effective aid to the building up and development of a happier and better civilisation.

It has been said that science knows no frontiers. This would be true as an absolute proposition, leaving contingencies out of account, and it would have been true if the lust of military autocracy, of conquest, and of the absorption and subjection of other nations had not made of science an instrument of abuse, of despotism, and of violence in the hands of a predatory race. The central empires have unnaturally distorted the spirit and the mission of true science, the mother of progress. Under the name of "Kultur" they have associated it with unbridled barbarism, making it an accomplice of the worst excesses.

A close agreement of conscience, an impulse of sympathy, and a confidence which the brotherhood in arms and reciprocal services will render permanent, have co-ordinated the scientific efforts of England and France.

Far from the war having stopped or weakened scientific life, it has on the contrary stimulated it. The zeal of seekers after truth has been galvanised. The progress of science during these

years of strife and the value of the results obtained are without parallel, above all in the domain of applied chemistry. In the future the prodigious efforts of our chemists and the ceaseless help that they have given on both sides of the Channel to the common defence, to augmenting the strength of the Allies, to the continuity, progress, and intensification of the production of the two countries, will be looked upon as a marvel. Thanks to them, to their zeal and their ingenuity, the chemistry of the allied countries which has powerfully contributed to the winning of the war, will, after peace is declared, spread abroad its benefits in all branches of industry.

#### THE POTASH DEPOSITS OF ALSACE.

I have chosen this as the subject of my address not only because I am a native of Mulhouse and the subject is accordingly of particular interest to me, but also because no other subject is of more interest and importance at the present time.

It is in fact these deposits which form the principal reason for Germany's desperate resolve to keep Alsace-Lorraine at all cost. There are also evidently sentimental reasons for this resolve. Alsace, as has been said, is the symbol of German unity built up on our defeat, and this is the truth.

But for Germany sentiment is only an outward show; it is only self-interest which counts, which directs all the collective activities of the nation. Germany is a business house, whose Emperor prided himself on being the commercial traveller. In this war which they declared on us, the Germans saw merely a gigantic commercial operation, of which the end would justify the means, and which was destined to enrich the firm and ruin their competitors.

The potash deposits of Alsace form the principal reason for Germany's desperate struggle to keep Alsace-Lorraine at all cost. The stake that Alsace-Lorraine represents for Germany, from the economic point of view, is a formidable one; Alsace-Lorraine, outwardly the symbol of German unity, is in reality the key to the industrial and economic supremacy of Germany.

Alsace-Lorraine holds two treasures, the loss of which would be, for our enemies, the downfall of all their dreams of economic hegemony. First it contains the iron-ore deposit of Lorraine, the loss of which would render Germany dependent on foreign countries for over 80% of her iron-ore consumption of 39 million tons. Through her victory in 1871, she gained possession of these huge deposits upon which great blast furnaces have since been erected. The output of iron ore in Germany before the war was 28 million metric tons, of which Germany proper only furnished 7 million metric tons; the remaining 21 million tons were taken from the mines of annexed Lorraine.

The acquisition of the ore-field of Briey, which continues the deposit across the frontier of 1871—an acquisition which was to be the consequence of our anticipated defeat—would have assured to Germany the whole extent of the ore-field, with an addition of a further 20 million tons or more per annum. With this realisation the French iron industry would have been ruined at the same time and would have become tributary to the victor.

What the loss of these ore-fields will mean for our enemies has been clearly pointed out by our President, Prof. Louis, in a paper entitled "La Guerre pour le Minéral de Fer" (this J., 1918, 323 n), in which he shows the importance of the Lorraine ore to Germany, and makes it clear that the object of Germany in making the war was to seize the whole basin of Briey and Longwy, and so to obtain practically the monopoly of iron ore in Europe.



The following passage occurs in a confidential memorandum addressed to the Chancellor, Herr von Bethmann Hollweg, on the 20th May, 1915, by six great German industrial and agricultural undertakings, concerning peace terms:—"The war is in fact only a struggle for the possession of the iron ores in the Lorraine and Longwy districts. Without the ore from Lorraine the war would be lost."

Unquestionably from the standpoint of metallurgy (and one knows what influence this has on German opinion) the object and perhaps the cause of the war was the seizure of the ore district of Briey and of the Belgian and French coal-fields.

This is clearly set out in the document mentioned above, and has since been repeated by the most important people in the German industrial world. They insist upon the annexation of these iron and coal mines, as well as on the dispossession of their tenants.

In restoring to France the iron mines of Lorraine, the Allies have the opportunity of reducing Germany to impotence. Her aggressive force is destroyed, and peace is guaranteed for the whole world.

Let us turn to the question of potash. When she annexed Alsace, Germany, who already had a monopoly of potash, did not know of these Alsace deposits. Fate willed it that they should be in a territory that had become German—deposits which alone would suffice for the world's consumption, and which furnish salts of a richness unknown in Germany. While she possesses Alsace, Germany retains this monopoly, and believes, with reason, that its loss would be for her an irreparable disaster. Germany will never give up Alsace until we are able to dictate our terms to her.

The Société de Chimie Industrielle of France has decided to publish an important work on the potash industry and the Mulhouse deposits by M. Camille Binder, of Mulhouse. It will be a new edition of a work published by the Mulhouse Société Industrielle before the war, but revised and enlarged. This work, from which I have taken some of my facts, will appear, I hope, before the end of this year.

It is not my intention to enlarge here upon the necessity of potash as a fertiliser. There are, however, some figures which it is important that we should realise. The following table gives, from official statistics, the relative production in France and in Germany of the principal crops, as well as the yield of each per hectare. I have not been able to obtain the comparative figures for the United Kingdom.

TABLE I.

Crop.	Country	Area under cultivation (hectares)	Total production (in metric tons)	Output per hectare (in metric tons)
Year 1913-1914				
Wheat ...	Germany	1,974,098	4,655,956	2'360
	France	6,542,230	8,843,127	1'352
Rye ...	Germany	6,414,143	12,222,390	1'905
	France	1,201,630	1,238,200	1'105
Oats ...	Germany	4,438,209	9,713,965	2'189
	France	3,979,270	5,182,600	1'302
Barley ...	Germany	1,654,000	3,673,265	2'220
	France	760,205	1,043,760	1'373
Potatoes	Germany	3,412,201	54,121,146	15'862
	France	1,548,070	13,585,965	8'776
Sugar-beet	Germany	535,309	16,946,000	31'780
	France	216,200	6,070,000	28'080

The soils which furnish these crops in Germany are for the most part the ancient sand deserts.

In order to render them the most fertile land in Europe (some Prussian land produces 5 metric tons of sugar per hectare), it has only been necessary to supply them with potash.

The soils which in France yield crops which seem to us so poor, and which nevertheless are much superior to those of former times, judging by statistics, are most favoured by nature; they are capable of producing a classic granary of plenty.

The second table is striking, and gives the explanation of the preceding one:—

TABLE II.

*Consumption of potash fertilisers (in metric tons of K<sub>2</sub>O) in Germany, United States, England, and France.*

Year	Germany	United States	England	France
1910	359,335	244,910	18,258	22,849
1911	422,340	237,442	20,902	26,468
1912	463,383	215,965	23,414	31,691
1913	536,102	231,689	23,410	33,115

In 1912 German lands received 1400 kilos. of potash per square kilometre, English lands 183.9 kilos., French lands 96 kilos., and American lands 128.9 kilos.

During the war, we have not been able to put potash on our fields; our crops have been diminishing. The Germans, on the contrary, have considerably increased their consumption, employing for their own agriculture the amount that they could not sell to the Allies. In the first year of the war their consumption fell to 483,627 tons, in 1915 they used 520,210 tons, in 1916 681,000 tons, and in 1917 more than 800,000 tons. They were thus able to increase their crops and discount the blockade.

I do not mean to assert that potash alone has produced these results, and that the crops are directly proportionate to its use alone. It is known that it is the association of nitrogen, phosphoric acid, and potash, in varying proportions according to the nature of the soils and the crops required, which controls the maximum yield. But if, in this partnership, potash had been omitted, the other factors, nitrogen and phosphoric acid, could not, as is well known, alone have ensured the results obtained.

The Germans foresee that after the war, in consequence of agricultural experience and the results obtained, their consumption of potash will be double what it was in 1913, that is to say, will exceed 1,000,000 tons per annum. The Potash Syndicate, thanks to the propaganda of an organisation which is always being improved, and thanks to the teaching of numerous agricultural schools, has diffused throughout the country enough of the elements of agricultural science to make the extensive use of potash, combined with other chemical manures, understood as a necessity.

Potash is so necessary for obtaining rich crops that one is tempted to maintain that the State would be justified in finding means to compel farmers to use it. One might also maintain that if the State went so far as to furnish potash gratuitously to farmers, it would be rewarded by the enlarged receipts from the taxes, which would put into the coffers of the State sums vastly exceeding the cost of the fertiliser distributed. In Germany, the people, trained to adopt the advice of scientific men and experts, have no need of this stimulant. Having followed the advice given them, it appears that they use every year more of this fertiliser, which gives them such marvellous results.

One may say that before the war Germany alone furnished the whole world with almost the total quantity of potash used. She thus possessed the

most powerful monopoly that any nation ever had, and she endeavoured with zealous care to ensure its continuation.

Dr. O. N. Witt, late Professor at the Charlottenburg Polytechnic, one of the signatories to the famous manifesto of scientists, published in the early days of the war, wrote:—

"Our enemies will not succeed in destroying our salt, potash, and soda industries—they cannot even disturb them; for Germany is the richest country in the world in its saline minerals and only uses indigenous means for working and transforming them. Our position as sole producer of large quantities of potash compounds gives us over and above this the power of causing grave injury to the agriculture of our enemies, by stopping the export of the salts which are indispensable to them for manual purposes.

"The re-taking of Alsace, that obsession which has become in France a national monomania, has not failed to engender, in certain minds, the hope of acquiring the deposits of potash which have lately been discovered in the Alsace sub-soil. This covetousness is the result of borings made in the neighbourhood of Belfort, where negative results have shown that the potash layers had been friendly enough to confine their distribution to the political frontier of Germany."

Whenever strangers tried to acquire an interest in German mines, they were prevented. This was notably the case fifteen years ago in connection with an American company which had bought mines at Solstedt. The German Government intervened to annul the sale, and for a short time diplomatic tension ensued between the two countries.

In choosing the title of my paper, I intended to speak only about the Alsace Potash, but it may be more interesting to give this survey a more general character and to glance also at the other sources from which the world's consumption can be supplied.

*North German deposits.* It was about 1850 that potash was discovered, at a depth of about 260 metres, in the rock-salt mines of Stassfurt. At that time the value of potash salts as a fertiliser was not known, and it was principally the working of the rock-salt which was aimed at. It was only later, when the fertilising value of potash was realised, that it was searched for. Numbers of companies were formed, not only in the neighbourhood of Stassfurt, but gradually over a wide district, which to-day includes the Duchies of Brunswick, Hanover, Mecklenburg, and Thuringia. Potash salts were worked for the first time in 1862. In 1907 thirty-nine companies for their working existed, united into one huge syndicate, the famous Kall Syndikat, which to-day holds practically a potash monopoly. This syndicate has organised a patient and effective campaign of propaganda in order to induce farmers to use this fertiliser. Its efforts were crowned with such success that in 1913 Germany was obtaining annually 11,607,000 tons, of which huge quantity she consumed half herself.

The composition and the richness of the salts from the German mines both vary materially. The richest is sylvinite, mixed chlorides of sodium and potassium, in which the deposits of Northern Germany are decidedly poor; it contains 26.3% of potassium chloride. Next comes kainite, a mixed salt of potassium chloride and magnesium sulphate containing 26.6% of potassium chloride. In addition to these are "hartsalz," a mixture of potassium chloride, sodium chloride, and magnesium sulphate, containing 21.2% of potassium chloride; carnallite, composed of the chlorides of potassium and magnesium containing 15.5% of potassium

chloride; polyhalite, which contains potassium, magnesium, and calcium sulphates.

With rare exceptions, all the salts from the North German basin contain a fairly large proportion of magnesia, and for the most part must be refined before they can be used even in agriculture. The companies which work them own, for this purpose, huge refining factories, which deliver the salts to the trade with a fixed proportion of potassium chloride.

In 1904 the Alsace deposits were discovered, with much richer salts than those of Stassfurt, and these, immediately coming under the control of the syndicate, completed the formidable economic weapon wielded by Germany.

*Galician deposits.* The deposits of Stassfurt and Alsace are not the only deposits of potash salts. Those of Galicia have been worked for some years. These beds are of small importance and do not seem to be able to provide even for Austria-Hungary alone, since in 1913 that country imported 21,000 tons of potash ( $K_2O$ ) from Germany.

*Spanish deposit.* The Spanish deposit, on the contrary, has perhaps great possibilities. At Suria, in Catalonia, potash was discovered shortly before the war, in a district where rock-salt mines were already worked. The potash was found at depths varying from 38 to 55 metres, and at certain points in Cardona there was even an outcrop.

A Franco-Belgian syndicate acquired the prospecting rights and continued the exploration. The discovery was one of capital importance for Spain and the countries of the Entente, who saw in it the possibility of shaking off the German monopoly. The Stassfurt syndicate, realising the danger that threatened them, and supported by the German authorities, used all means in their power to prevent the development of the Catalonia deposit. Their agents obtained concessions and took shares in Spanish companies; they had already obtained concessions in the adjoining Franco-Belgian deposits. The Spanish Government has just taken energetic action; it will compel the concessionaires to work the mine under a penalty of the loss of their rights, and it will grant no more concessions. It has decided to continue the sinking itself in order to define the limits of the basin.

The deposit extends over a considerable area, but is not continuous in the beds, as is the case in Alsace; moreover, it appears that the working is rendered difficult because the beds, instead of being horizontal as in Alsace, are steeply inclined.

The original Franco-Belgian concession of Suria and Cardona now seems to be the most important. It comprises both carnallite and sylvinite; the carnallite ore, which is the more plentiful, contains at least 12%  $K_2O$ . This concession includes 12,000 hectares and works 12 shafts, 300 to 850 metres in depth. The area in which the condition of the beds is favourable for working is about ten kilometres in width. A shaft which should yield 1000 tons per day is being sunk, and should be completed by the commencement of 1919. The Sociedad de Industrias y Comercio, which has also obtained a concession, has sunk three shafts near Cardona.

The development of the work so far carried out shows that Suria is the centre of an old lagoon which at the close of the Eocene and the beginning of the Oligocene period covered the North-west of the Province of Barcelona and the East of the Province of Lerida. We have here a very important reserve of potash in an advantageous situation from the point of view of working, which presents a new threat to the supremacy of the German syndicate.

The *United States deposits* are not potash mines but salt lakes situated in the Rocky Mountains in the States of Nebraska, Utah, and California; the Lake of Searles, in California, is worked by the



American Trona Corporation, which is largely in English hands. This company treats the liquors by concentration and crystallisation in its refinery at San Pedro. The reserves are considerable, and it seems that when the extensions now in progress are completed the Trona Corporation will have a daily output of salt equivalent to 100 tons  $K_2O$ .

The *Italian deposit* at Erythra is of a recent lacustrine formation. It is being worked by a company and will have an output for 1918 of 50,000 tons. The reserves are inconsiderable; on the other hand, the deposit is extremely important on account of the richness of the salt, which contains 80% of potassium chloride.

In *Tunis* to the south of Gabes, a salt lake of 15,000 hectares has been worked since 1915, primarily for bromine, though also for potash. The salt is obtained by solar evaporation and crystallisation; it is called *sebkainite* and contains 40%  $K_2O$ . In 1917 this deposit yielded 20,000 tons of salt and it is expected that this year it will yield 25,000 tons. The installations in course of completion should increase the production to 100,000 tons per annum. A refinery has been set up for the production of pure potassium chloride.

In *Chile*, in the Province of Tarapaca, in the Lakes Pintados and Bella Vista, deposits of potassium chloride have been found of which the richest contain 36% of chloride. The reserve, estimated at two million tons, has a content of from 3 to 12% of chloride.

There are also deposits in Peru, in Russia, in Morocco, and in the Province of Overijssel in Holland. None of these has been hitherto developed. A deposit is also said to exist in Sicily.

To complete my list there are the natural deposits of potassium nitrate in India and in Chile. In the latter country it is found associated with nitrate of sodium, but the proportion of potash is small. In Brazil a deposit of potassium nitrate has been discovered containing 89% of this salt.

Finally, salt marshes provide us with mother liquors from which potassium chloride can be extracted, notably at Salin-de-Giraud, in the delta of the Rhone. These works have an annual output of 600 tons containing 72% of chloride.

In addition to these deposits of salt and the other saline sources of supply which have just been reviewed, the war has compelled us to make a careful inventory of the other sources of potassium which we possess, and I doubt whether this work has been better done in any country than in Britain. It is not necessary for me to enlarge upon this; the work which Mr. Chance communicated recently to this Society is so complete that there is nothing to add to it. He has told us that before the end of this year the Oldbury Works will be capable of producing all the potash salts necessary for the country in time of war (see this J., 1918, 222 T). Mr. Chance has recognised that although sources of supply, usually wasted and carelessly squandered, which are provided by blast furnaces, cement furnaces, wool washings, etc., will yield a large proportion of the supplies necessary for the country, in practice it will always be difficult to recover the whole of the potash from these sources and recourse to natural salts in addition must always be necessary.

It must be remembered that the needs of agriculture increase every year, and if we take as a basis of comparison what has happened in Germany, it is not rash to conclude that before many years have passed the 23,000 tons of potash ( $K_2O$ ) that England consumed in 1913 for agriculture may reach 50,000 or perhaps 100,000 tons. The mine can never be replaced except to a comparatively small extent.

The *Alsace potash* beds are situated in the south of the province in the plain, bounded on the south by the Jura, on the west by the Vosges, and on the

east by the Rhine (see map, page 295 T). Recent borings have shown that secondary beds of the same formation are to be found beyond the Rhine. The deposits extend as far as the suburbs of Mulhouse. They consist of two beds, of which the first, the more important of the two, has an average thickness of four metres and occupies an area of about 200 square kilometres. It is found at a depth of about 650 to 1000 metres (see diagram, page 296 T). The upper bed is less important both in area and thickness; it is fairly regularly parallel to the first and is 15 to 25 metres above it. The beds are formed for the most part of pure *sylvinite*. They are situated below a deposit of rock-salt, which attains a thickness of 240 metres. The upper bed has an average content of 35% of potassium chloride and the lower one of 30%. The layers are nearly horizontal, which greatly facilitates the working. They are not subject to infiltration of water.

The total cubic contents of the two beds is about 700,980,000 cubic metres, that is to say, about 1,472,058,000 tons, the average content of the salts being 22% of potash ( $K_2O$ ). The whole deposit represents therefore more than 300 millions of tons. One may accordingly reckon that it would suffice for the world's consumption for many years.

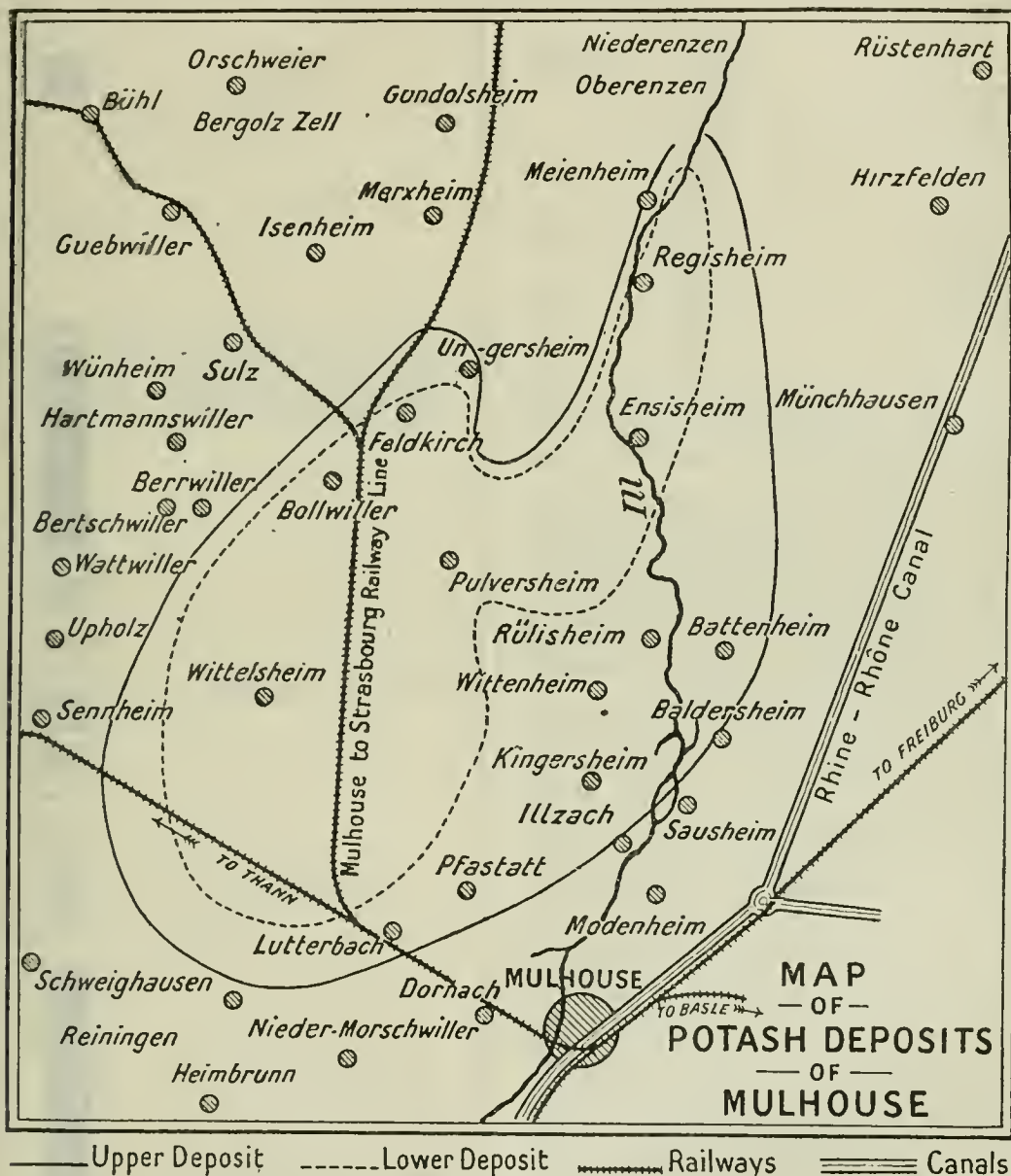
But the most interesting fact is that the purity of the salt greatly surpasses that of all other known deposits of importance, and is notably very superior to that of Stassfurt. Refining is unnecessary for the salt intended for agriculture. The cost of transport is accordingly considerably reduced.

The first boring was made in 1904, near Wittelsheim, by a group of prospectors brought together by the enterprise of Messrs. Joseph Vogt and J. B. Grisez. Their object was to look for coal. At 358 metres, they reached beds of rock-salt; the boring was continued to a depth of 1129 metres; potash-bearing strata were reached without its occurrence being detected. It was only later, in examining samples of a dark red colour, that the presence of potash was recognised. Other borings, decided upon immediately by the prospectors, showed that the deposit was of large extent. The company formed to work this discovery made 165 borings, and began working in their first shaft in 1910. This first company, the *Gewerkschaft Amelie*, has founded affiliated companies in order to profit by the regulations of the German potash cartel, which allows a given output to each centre of extraction working on a concession of 1800 hectares. At the end of the same year, the German company in Stassfurt, the *Deutsche Kaliwerke*, recognising the value of the Alsatian beds and fearing the independent spirit of these new competitors, bought enough shares to secure for themselves the control of the Amelie company. They paid 30,000 marks for shares which had cost their owners 6000. Another group of French and Alsatian capitalists founded the "*Ste. Therèse*" company. This company obtained a certain number of concessions covering 5000 hectares, and afterwards bought most of the shares in two other companies which owned concessions adjacent to their own. The different concessions of the *Ste. Therèse* company have just begun to be worked. Two of the shafts have reached the deposit, and two are now being sunk. Two other German groups have acquired concessions and are carrying out development work. One may therefore say that the potash basin of Upper Alsace is divided among four parent groups or companies.

The German Potash Syndicate has taken care to limit the working of the Alsace deposit, and only allows an output of 5% of the total German output. In order to safeguard the interests of the powerful shareholders of the mines of Northern Germany, the paradoxical situation has been created, that the deposit which furnishes the

richer product and the mines which can be most easily and most economically worked are sacrificed to the poorer. In addition, vexatious regulations were made (which seem, however, to have been annulled shortly before the war) enacting, for instance, that Stassfurt should be taken as the basis from which transport charges were to be calculated whatever the destination might be. Thus, for Switzerland or for France, which are 30 kilometres from Mulhouse, the cost of transport was

represent the State. The President is nominated by the Secretary of State for Home Affairs, and can appeal to the Reichstag. In order to be allowed to sell, a mining company must first join the Syndicate and must conform to its regulations with regard to the organisation of its workings, both underground and surface. The principle of the law of May 25, 1910, which officially established this cartel, is above all to control the sale in order to avoid over-production, and to regulate



as high as for the 600 kilometres, as the crow flies, between their frontiers and Stassfurt. These tariffs were in force even for the countries surrounding the workings.

The Potash Syndicate comprises all the companies which work potash beds in the whole extent of the Empire. Its operations are under Government control. It is managed by a committee of seven, of whom four represent the mines, and the other three, of whom the President is one,

this sale in such a way that Germany shall be favoured at the expense of the foreigner. The State has laid it down as a principle that potash must be considered as a national asset, and must, before everything else, favour the agriculture and industries of Germany. The committee fixes each year the quantities that may be sold by each mine in Germany and the amount that may be exported; it also fixes the selling prices. Factories treating the raw materials cannot be installed, even in



foreign countries, without the authorisation of the syndicate. The syndicate also institutes propaganda for making potash fertilisers better known, and its organisation from this point of view is especially well developed.

A general view of what the situation will be after the war with regard to the world's consumption of potash may be gathered from the following data.

According to the statistics for 1913, and for 1916 where these are available, the consumption was as follows:—

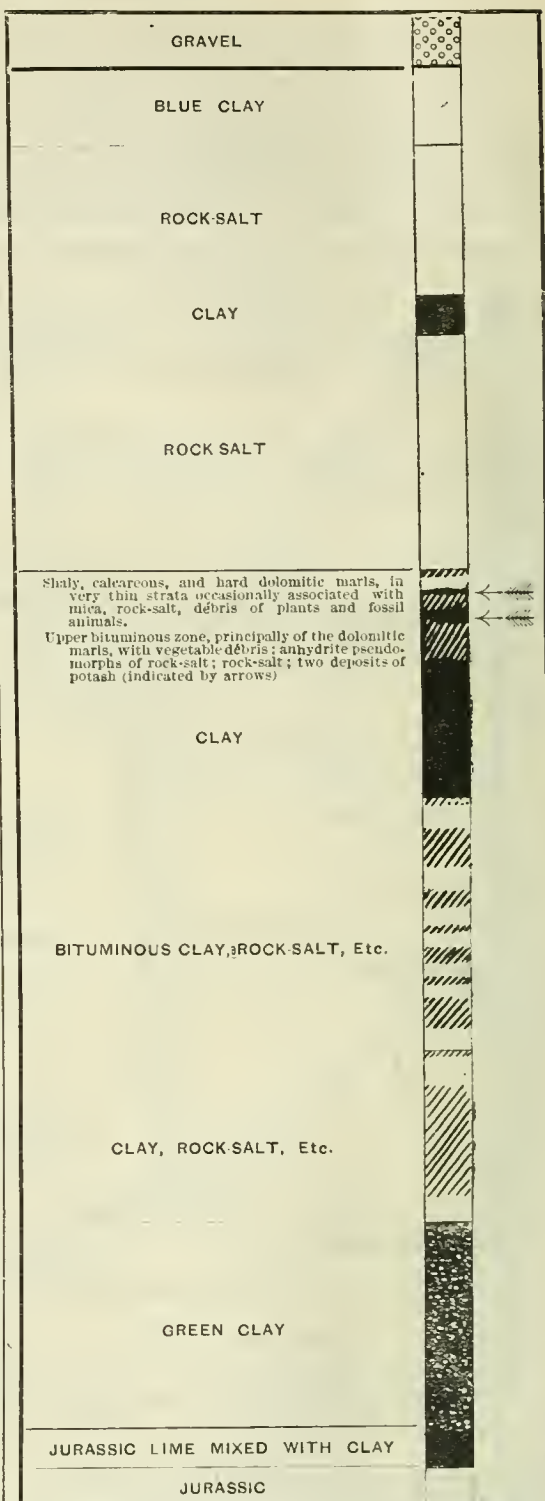
	1913	1916
Germany ... ..	536,102	681,000
Austria-Hungary ... ..	25,073	—
United States ... ..	231,683	—
France ... ..	33,115	—
England ... ..	23,410	—
Belgium ... ..	13,182	—
Italy ... ..	6,354	—
Portugal ... ..	1,241	—
Holland ... ..	43,478 (?)	56,850
Denmark ... ..	7,478	—
Russia ... ..	24,260	—
Sweden ... ..	19,513	23,000
Norway ... ..	3,593	—
Switzerland ... ..	3,308	6,830
Spain ... ..	8,292	—

The world's consumption (calculated as  $K_2O$ ) for agricultural requirements alone, for the year 1913 amounted to 1,004,000 tons. For the chemical industries it is estimated at 135,000 tons, making a total of 1,139,000 tons.

One may say, generally speaking, that the potash produced by sources other than salt mines, that is to say, from salt marshes, vinasses, kelp, suint, the mother liquor of nitrate of soda, and dust from blast furnaces and cement furnaces, will provide a quantity which almost equals the amount necessary for the chemical industries, so that without committing a grave error, one may admit that the whole of the needs of agriculture must be supplied by the salt mines.

It is interesting to estimate what the needs of the different countries will be after the war and how they can be supplied. It is probable that Germany will supply Austria (except for what is furnished by the mines of Galicia), Russia, Sweden, Norway, and Denmark, to which we may add Holland, although it is to be hoped that Alsace will be able to supply this country by way of the Rhine. Total, according to the 1913 figures, 616,019 tons. Spain will have enough from her own mines and will also supply Portugal. Total, 9533 tons. Italy possesses the mines at Erythrea, and may eventually have the deposit located in Sicily. She will be self-supporting: say 6354 tons. There remain France, England, the United States, Belgium, and Switzerland to be supplied, with a total of 304,704 tons. All these figures are those of 1913, and may be at least doubled for the years after the war. We have already seen that Germany, during the war, has nearly doubled her consumption for agricultural purposes. Certain adjacent countries, such as Switzerland, Holland, and Sweden, have also largely increased their consumption. Deprived of the markets of the countries of the Entente, and with the object of improving her economic position, Germany has made great efforts to use potash as a medium of exchange.

I have already explained that by the loss of the Alsace potash mines, Germany will be deprived of the economic weapon of bargaining, which she thought she could use in order to obtain the imports that are indispensable to her existence when peace is discussed. Without the monopoly



BORE HOLE THROUGH THE POTASH DEPOSITS  
AT WITTELSHEIM.

of potash all her system of defence will collapse. Her newspapers, which do not yet admit the possibility of this loss, have continually pointed out the importance of these riches for obtaining favourable economic terms.

The following is an extract from the *Vossische Zeitung* of 23rd November, 1917:—

"The Alsatian potash will multiply the harvests of France. The French are already thinking of the advantages that they will derive from the possession of the Alsatian mines. America comes like the third thief, to show England what an advantage it will be for the Allies if they can get potash independently of Germany.

"America would be able to get larger quantities of potash at lower prices, and we should thus be deprived of the best economic weapon that we possess against her.

"Potash is both wealth and weapon. We shall restrict the delivery to neutrals and to our enemies. If raw materials are refused us, we shall revenge ourselves on enemy agriculture, with this war-cry, At the enemy, with a kilo. of potash!"

In the same paper (26th June, 1917) under the title of "Our Ally Potash," Dr. Roth shows by statistics that the wheat harvests of the United States have been deficient since the shortage of potash. He concludes his article by saying:—

"By a fortunate coincidence, America is not only our principal source of raw materials, but also our greatest buyer of potash. Though the value of our imports from America is much above that of our exports to that country, it must not be forgotten that potash supplies a biological necessity. It is a primordial necessity for the increase of harvest, which nothing can replace and which we must sell at a high price. It is therefore impossible for America to prolong the war indefinitely, unless she consents to see her harvests continually diminishing. If we rejoice at having potash as an ally during the war, we are also justified in the belief that it will swing the balance strongly in our favour when the Peace Treaty is discussed."

In the same newspaper, the next paper, 27th June, 1917, in an article entitled "Alkali and Cotton," Kuhne and Lange bring the same arguments, based on statistics, to bear on the subject of cotton, showing that since the United States had been deprived of potash the crop has sensibly diminished, with the result that prices have been more than doubled. They add:—

"We should treat our potash with the utmost respect, because it is capable, owing to our enemies' lack of it, of producing great fluctuations in securities, or at least of contributing powerfully to this end, without counting its influence on other agricultural products, notably wheat.

"Our experience during the course of the war is that our export of potash to the United States of America (71,000,000 marks in 1913) has an importance far surpassing what had ever been admitted before. It puts into our hands a weapon which our Government will know how to employ usefully when peace negotiations are begun."

Here is another important point. It is known that the German cartel sold at higher prices for export than in Germany, that is to say, that the export trade allowed German agriculture to have its potash cheap. The greater part of this export being suppressed, the syndicate will be obliged to reimburse themselves by raising the prices for home consumption. One may suppose therefore that in the future German agriculture will pay a higher price for potash than foreign agriculture. This is an obvious consequence.

Another point not less important is that amongst all products exported by Germany, potash formed the highest in tonnage. That allowed her to be sure of a return freight in exchange for imports from foreign countries. It has been calculated that potash made up 30% of the tonnage carried by two large German companies. If potash is wiped out as a return freight, since it cannot be replaced

by any other article produced by Germany, the cost of freight for her imports will be considerably increased.

Let us also consider the effect on the yield of the taxes of which the potash mines pay an important quota, and also the effect on the exchange. German economists already estimate at 500 millions of francs the credits that the sale of potash salts in foreign countries will amount to annually for Germany, while she possesses the monopoly of potash.

The *Deutsche Zeitung* of 10th October shows how important the possession of Alsace-Lorraine is considered from the industrial and economic points of view:—

"The German iron industry draws three-quarters of the necessary ore from Alsace-Lorraine. Before the war, the German output of iron ore reached 28 million tons, of which 21 millions came from Lorraine. It must be added that German technical industries have succeeded during recent years in making products of superior quality with Lorraine iron to such good effect that new markets have been opened for her steel industry in the future. The supply of ore is the principal question for the German iron industry, that same industry which, side by side with agriculture, is the foundation of our economic edifice. Hence arises the painful question as to what is to become of the workers in the metal industries and of all the people whose existence is linked up with the iron trade, if the principal district which produces ore is taken away from us for ever. To cede this district would be the downfall of the economic life of our country, it would take away from an important part of the German nation their means of subsistence and either abandon them to misery or force them to emigrate.

"Alsace has another special significance for German economic life. The last years before the war witnessed the development of the Alsace potash industry, which would already have reached its full output had the war not intervened. The Alsace potash beds are amongst the richest that have ever been found. If these deposits passed into the hands of the enemy, it would be the end of the German monopoly of potash. Our enemies would speedily increase the number of companies engaged in this industry, which already amounts to 17, and they would supply the world's market with potash. We need not point out what would follow for our own potash industry and of what a financial weapon the enemy would deprive us.

"Neither do we need to insist upon the way in which these economic facts would react upon the world of workmen, upon the towns, and, in a general way, upon public life in Germany. It is from such considerations that during the last fortnight the total value of the shares in the Wintershall Company, which is interested in the workings of Alsace, has fallen from about 75 million marks to less than 50 million marks."

I have shown by the above considerations that in order to ensure peace for the future, and save the world from German economic dominance, it is indispensable that Alsace should again become French. I have shown that it is not only a question of sentiment and of lofty justice which concerns France alone, but that essential interests which concern all the Allies are linked to this act of justice, and it cannot be too much insisted upon. It is only by the restoration of Alsace-Lorraine that an economic and military victory over Germany can be gained.

Alsace-Lorraine, which was to have been the symbol of German unity built on the defeat of France, will become the symbol of her own defeat. But this is merely sentiment; it will not be enough to stimulate the German soul to revenge. The two



lost treasures, in the eyes of the Germans, will not be Alsace and Lorraine, but the iron mines of Lorraine and the potash mines of Alsace.

#### DISCUSSION.

Prof. HENRY LOUIS said he felt sure they would all agree that the paper was one of the utmost importance and they were greatly indebted to M. Kestner for having brought it before them. It was important not only on account of the valuable matter it contained but also on account of the psychological moment of its delivery. A week earlier it would have been too early; a week later it might be—he hoped it would be—too late. Let no one say that M. Kestner had touched upon subjects he should not have touched upon, and that the chemist had no right to enter into the domain of politics; this case above all others was the very one on which the chemist ought to speak with no uncertain voice, for this had been above all a chemists' war. It looked like the irony of fate that the First Republic should have sent Lavoisier to his death and that a fresh Republic should have been saved by the efforts of Lavoisier's scientific descendants, among whom M. Kestner could be regarded as one of the foremost. Let them bear in mind that the commencement of this war had been conditioned by advances in chemical industry. The first steps that rendered this war possible were taken forty years ago when chemists applied certain reactions between phosphoric acid and lime to found the basic process of steel-making. The last step that rendered it possible for Germany to enter upon the campaign was taken when other chemists applied the processes for uniting atmospheric nitrogen and oxygen to practical purposes. The first of these steps in industrial chemistry furnished Germany with the steel she needed for her weapons; the last furnished her with the explosives she required for the war. If the war, then, was started in this sense through chemistry, surely chemistry should have a word to say in the ending of it. When Bismarck was settling the peace terms of the Franco-German War of 1871, his first intention was not to have included Lorraine in the German demands. He consulted, however, the head of the Geological Survey of Prussia, who pointed out that iron ore outcrops existed in Lorraine and that it was accordingly advisable to annex it. Bismarck acted on this advice. He (the speaker) feared that we could not claim to have on our side to-day a statesman of the calibre of Bismarck, a man who had the brains at any rate to know his limitations; but surely we might hope that those who were guiding our fortunes had got common sense enough to be guided by his example and to consult technologists who were able to advise them on technical matters in relation to an important subject of this character. He thought M. Kestner had made the prime importance of the Alsace potash deposits very clear and had shown that when these reverted to France—as they would revert to France—the German potash monopoly would be broken for ever. M. Kestner had spoken of the other deposits in various parts of the world. He (Prof. Louis) had worked upon some of them and he believed he was the first to recognise the alunite deposits in the South of Spain, but he was an old miner and he did not trust any deposit, however promising it might be, until pits had been put down and it was possible to know what was actually there. They must not allow themselves to be led away by the fact that because there were promising deposits in Catalonia or elsewhere it was safe to neglect the Alsatian deposits. It might also be relevant to point out that the Spanish deposits lay at steep angles and were much more difficult to work than the flat-lying Alsatian deposits. Furthermore, M. Kestner had not drawn attention—it was outside the strict limits of his

paper—to the petroleum deposits of Alsace. They were not very large certainly, but in a region where there was not much fuel they were important and would add considerably to the mineral wealth of Alsace.

With regard to the iron ores of Lorraine their importance could hardly be over-rated. One point which M. Kestner had not mentioned was that the Lorraine ores were highly phosphoric and the basic slag produced in their smelting had been one of the mainstays of German agriculture. The increase in the output of German agriculture had not been solely due to the use of potash; it had also been due to the large quantities of basic slag obtainable from the Lorraine iron ores. Anybody who followed German technical literature would have seen constant references during the four or five years preceding the war to the iron ore deposits of Briey and Longwy on the French side of the frontier. There had been constant complaints and a bitter outcry from the Germans that France had opened up her iron ore deposits. It had been found that they contained 3 or 4% more iron than the German deposits and bitter complaints had followed that the German ore was being ousted by the French. He had no doubt whatever that the bribe that induced the big German ironworks of Krupp and other works to help in this war—because the war would not have been started if they had not consented—was a promise that they should have the French iron ore deposits as their reward. It was repeatedly stated in the German technical papers that they were going to take the French iron ore deposits so that they would then have a monopoly of iron ore in Europe and be armed for the next war. It was therefore essential for the world's peace that the Lorraine iron ore deposits should not remain in German hands and that France should re-enter into possession of what Germany had pillaged from her. Let them remember that they would leave Germany with ample potash for her own agriculture and leave her with ample supplies of iron ore for the manufacture of ploughshares and looms. But we must take away the iron ore she would use for making guns and submarines. So long as Germany was left in possession of her potash monopoly and of the iron ores she was a source of danger to the peace of the whole world.

Mr. KENNETH CHANCE said there were two points in the paper which especially appealed to him. The first was the complete manner in which M. Kestner had dealt with the potash deposits of the whole world. To have given this information in a paper which was arguing the necessity for Alsace to revert to France was the strongest argument he could possibly have put forward, because in the new world which it was hoped would be developed after this war it was going to be the case of the whole world against Germany until she behaved herself and became worthy of inclusion amongst other nations. The second point was that one could imagine the Germans, who were so famous for their looting propensities, clinging to Alsace because it was about the only thing left which they could not take back to Germany with them.

With regard to the industry in this country, any development at present would only be very small in comparison with the huge figures which M. Kestner had put before them. He did not think, however, that that made it any the less necessary for this development to be carried out to the fullest extent, because although at the present time and for many years to come it was perfectly clear that our friends in France and our other Allies and ourselves would have to work together in every possible way in order to produce what we required for ourselves and for one another, there would come a time when we could each—or those of us who were sufficiently favourably situated—produce



enough for ourselves and have a surplus over. When that time came he hoped we should have developed the potash industry in this country sufficiently to be able to enter into an economic contest with our friends in France to see who could produce potash more cheaply, a contest in which the welfare of humanity was based upon the success of both parties. Although, as he had said, this country's output was very small in comparison with the huge deposits of Alsace, we had one advantage here even though, as M. Kestner had said, the Alsace deposits were richer than those in any other part of the world. From the by-product potash in this country we got a raw material even richer than that of Alsace. It ran from 50 to 60% potassium chloride, which corresponded to about 35 to 40%  $K_2O$ . If M. Kestner came before them with the idea that a strong plea was needed to convince them that these deposits should revert to France he must have realised that he was speaking to the converted. There were very few people in England who had not long ago made up their minds that this was bound to come about.

It might be of interest for him to add that caustic potash of the very first quality was now being made by electrolysis from the muriate of potash recovered from blast furnace dust in this country. He had not yet seen a sample, but he understood that the purity was at least as great as if not greater than anything hitherto manufactured by Germany. That was the first development in the British potash industry of this character, and other developments were following on rapidly. The immediate problem, however, was the manufacture on a large scale of the fertiliser, muriate of potash. A full development in this direction would take some years; in the meantime they all hoped that their French friends would supply all the potash required for the needs of this country.

Mr. LAWRENCE WEAVER, of the Board of Agriculture, said that in reference to the figures which M. Kestner had given, the yield of wheat in Germany, translated from metric figures to English measures, was about one ton per acre. The English yield was about 4 quarters. This increase in output in Germany was no doubt, as M. Kestner had said, the result of the enormous amount of potash which the German agriculturist had been able to obtain at a price which, before the war, was about half the price which the English farmer had to pay. Turning to the potato crop, the French yield was roughly  $3\frac{1}{2}$  tons to the acre, which struck the English farmer as extraordinarily low. The German yield was about 7 tons to the acre. Last year the average for England and Wales was just under 7 tons per acre, so that without any potash at all we did as well as the Germans. Too much importance must not be attached to the fact that a country like Holland used, on the face of the figures given by M. Kestner, about double the amount of potash that we did. In the first place, some discretion must be used with regard to the English figures. The consumption of potash was given as 23,410 tons for 1913, but it was not known how much of this quantity was used as a fertiliser. Unfortunately, our statistics of fertiliser consumption before the war were extremely deficient. He hoped that things were a little better now; in any case he could promise that when the war was over much more complete statistical information would be available, even to the extent of the use in any particular county of England. It was possible that only about 15,000 tons was used in 1913 for fertilising purposes, and it was probable that for the 1918-19 season we should have used about 2000 tons recovered from fine dust. That was a very trifling quantity, but still it was something, particularly as it was used mostly for the flax crop, which was essential

for aviation purposes. It was quite obvious that we could and should, in the ordinary way, enormously increase our pre-war consumption of potash because the English farmer was now fully alive to its importance. A further consideration was the fact that the land had been so much starved of potash during the last four years. As to whether the increased consumption would be anything like so great per acre as to bring it up to Germany's depended not on the intelligence of the farmer but on the broad lines which agricultural policy was likely to follow in this country. If we were going to increase our arable area very much and maintain the output which was obtained last year our capacity for using potash would be greatly increased, because in order to increase our arable land we should have to take into use light land which in pre-war days was not ploughed. Those who were inclined to blame us for our smaller use of potash compared with Germany should bear in mind that our land was markedly heavier. Germany had a very large area of very light land with which nothing could be done without the use of potash in considerable quantities. Again, the use of potash depended on the crops that we grew. The crops of most importance so far as potash was concerned were potatoes and flax. If we were to build up industries based on our potato crop and use the potatoes for industrial purposes such as the manufacture of spirit and starch we should use a very great deal of potash. If we were going to maintain a large flax area in order to provide aeroplane fabrics we should want potash. Therefore it really depended on what our agricultural policy was going to be, and that in turn depended on what our fiscal policy was going to be. The nation would have to decide what was to be done in that connection. The extent to which the chemical fertiliser trade would flourish would be determined by the character of these developments.

Captain GOODWIN drew attention to potash bearing rocks such as feldspars, glauconite, and leucite as sources of potash, and suggested that in the future one or two processes for the manufacture of potash from potash bearing rocks would play a full share in the solution of the potash problem.

The CHAIRMAN, in conveying to M. Kestner the sincere thanks of the meeting for his invaluable address, said that in view of its outstanding importance a special effort would be made to distribute it not only to the members of the Society but to as wide a circle as possible, including members of Parliament and of Government Departments, so that the great economic importance of the subject might be brought home to them without delay. Prof. Ostwald, he stated, was reported to have said that it rested with Germany to decide whether in the future the world was to be nourished or starved. M. Kestner had answered this decision; he had added one more nail to the coffin of Germany's arrogant ambitions, and they were sincerely grateful to him for having done so.

They all rejoiced that M. Kestner, the distinguished President of the Société de Chimie Industrielle, had been able to come over to address them. They thanked him both personally and as the representative of his Society for his acceptance of their invitation, and in doing so were mindful of the great honour he had paid not only to the Society but also to all that chemistry stood for in this country and to the nation itself.

The Société de Chimie Industrielle had been founded with aims akin to those of their own Society, and they looked to M. Kestner's visit as a first step towards securing a comradeship in effort for the mutual development of chemical industries as a fitting sequel to the glorious comradeship of French and British in arms.



## Sydney Section.

Meeting held on July 17th, 1918.

MR. B. J. SMART IN THE CHAIR.

### NITRATION OF POSIDONIA FIBRE.

BY B. J. SMART, B.Sc., AND P. PECOVER.

Up to the present time the cultivation of cotton fibre in Australia has not been successful owing to the high cost of labour, and, unless some special means are adopted for the encouragement of the industry, it appears desirable to consider other possible sources of cellulose suitable for the manufacture of gun-cotton. The attention of the authors was directed to the fact that large quantities of marine fibre (*Posidonia australis*) are readily obtainable at Wood's Point on Spencer's Gulf, South Australia, which it might be possible to purify at a moderate cost so as to render it suitable for nitration. With this object in view the work now described was carried out, and, although only preliminary in character, the results obtained would certainly suggest that further investigation on a large scale would be profitable.

A full account of the distribution and methods of recovery of the fibre is given in Bulletin No. 4 of the South Australian Department of Chemistry (see this J., 1917, 542). It may be stated here that the fibre is best obtained by dredging to a depth of 7 feet from certain flats on the shores of Spencer's Gulf. It is then washed, cleaned, and dusted by means of special machinery, and is marketed in bales. At the present time its principal commercial application has been as a heat insulator, since it has been shown to have a low heat conductivity (Pecover and Smart, Commonwealth Engineer, Dec., 1917, 127). It consists of short, harsh fibres somewhat resembling jute, and usually has a quantity of broken shells associated with it. A sample examined by the Imperial Institute gave 55.9% of cellulose (Reports of the Imperial Institute, No. 58, 1909, p. 133). The fibre as placed on the market contains a large quantity of mineral matter, the percentage of ash in the sample obtained for this work being 16.1. This was, however, considerably reduced by mechanical shaking, the resulting fibre showing an ash content of 11.5%. The ash obtained from the clean fibre was analysed with the following result:—Residue insoluble in 20% HCl, 1.65%; silica (soluble), 2.05; iron and alumina, 12.75; lime, 51.60; magnesia, 9.05; potash, 4.78; chlorine, 6.16; phosphates, 1.81; sulphuric acid, 7.80%; sodium, present.

**Purification of the fibre.**—The fibre was subjected to a number of processes and determinations of the ash content were made until a value less than 1% was obtained. The methods chosen were purely arbitrary, and it is possible that other methods might be found equally satisfactory. When, however, it was shown that a product practically free from ash could be produced, this product was used for the subsequent nitration.

The following table shows the results obtained:—

Method of treatment	Ash in product, %
1. Treated with nitrating acid and washed ...	6.5
2. Boiled twice with excess of 2% caustic soda ...	3.8
3. Fibre shaken for several hours, four times with water ...	2.8
4. Boiled with (1) 4% sulphuric acid, (2) 1% caustic soda, (3) 1% caustic soda, (4) water ...	0.55

5. Boiled with (1) 4% nitric acid, (2) 1% caustic soda, (3) 3% caustic soda, (4) water ... 3.0
6. Soaked in 10% hydrochloric acid all night and washed several hours in running water ... 2.12
7. Product from (6) soaked again in cold water ... 2.06
8. Product from (7) boiled with 5% hydrochloric acid ... 1.1
9. Boiled with 3% sulphuric acid for three hours; washed ... 0.68

**Nitration of the fibre.**—The product from process 9 was considered to be a suitable material for nitration. It was thoroughly dried in a water oven until the weight remained constant, and nitrated for 12 hours in a mixture of 3 parts by weight of sulphuric acid (sp. gr. 1.84) and 1 part of nitric acid (sp. gr. 1.5). The nitric acid was previously prepared to give the required specific gravity by distilling with concentrated sulphuric acid. In order to study any peculiarities attending the nitration of the fibre, other than those which generally accompany the nitration of cotton, a sample of ordinary cotton was nitrated under exactly the same conditions. It was found that greater care had to be exercised in adding the fibre to the acid than was necessary with the nitration of the cotton. The velocity of reaction was greater owing to the fact that a portion of the fibre was in a very finely divided state. The nitrated products were purified by repeated boiling with water. The process of purification took place more rapidly than with nitrated cotton under the same conditions. The finished product gave on analysis 12.3% of nitrogen, and ash content nil. Yield 72%.

**Stability.**—Heat tests were carried out both on the nitrated fibre and on cotton at 170° F. (76.6° C.), and the following results were obtained:—Nitrated fibre, 10½ minutes; nitrated cotton, 15 minutes; nitrated fibre (after two years' storage), 12 minutes. It was not possible to obtain a higher heat test of the fibre than 10½ minutes, but it appeared probable that this was due to the degree of comminution as shown in Robertson and Smart's work on the Abel heat test (this J., 1910, 130).

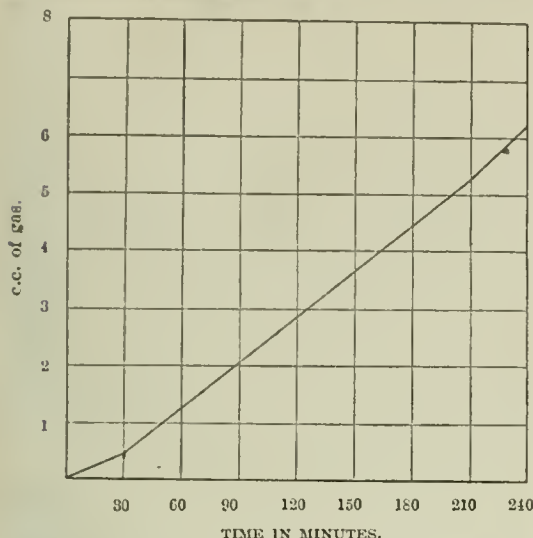
Further tests of the stability of the nitrated fibre were made by subjecting it to the Will test at 135° C. For this purpose 2.5 grms. of the purified fibre was heated to 135° C. in a current of carbon dioxide, and the nitrogen evolved measured, with the following results:—

Quarter hours	Temp. of bath, °C.	Vol. of gas, c.c.	Diff., c.c.	Temp. of air, °C.
1	135.0	0.20	0.20	30.0
2	135.2	0.45	0.25	29.0
3	135.0	0.85	0.40	30.0
4	135.1	1.25	0.40	32.0
5	135.0	1.65	0.40	32.0
6	135.0	2.05	0.40	32.0
7	135.1	2.45	0.40	32.0
8	135.0	2.85	0.40	32.0
9	135.0	3.25	0.40	32.0
10	135.2	3.65	0.40	32.0
11	135.1	4.05	0.40	32.0
12	135.0	4.45	0.40	32.0
13	135.0	4.85	0.40	32.0
14	135.2	5.25	0.50	32.0
15	135.0	5.75	0.50	32.0
16	135.0	6.20	0.45	32.0

Air correction per quarter hour, 0.02; total air volume 0.32 c.c.; 2.5 grms. gave 6.4 mgrms. N per 4 hours.

The result showed that the gun-cotton produced was comparable in stability with Waltham Abbey gun-cotton, which gives 6.0–6.5 mgrms. in 4 hours.

The values obtained in the above test, namely, "Vol. of gas c.c.," are plotted against the time as shown in the accompanying figure (decomposition curve). The first two values are low, owing to the fact that about 30 minutes is required before equilibrium is established.



**Purification of the fibre on a large scale.**—In view of the fact that the fibre can be nitrated successfully, it was considered desirable to carry out more extensive experiments on the purification of the crude fibre, in order to ascertain whether the results obtained in the laboratory could be obtained with bulk quantities on a commercial scale. These were carried out by Posidonia Fibres, Ltd., under our direction, and we are indebted to this firm for permission to publish the results.

Process 9 was resorted to in the following work:—An acid boiling vat having a boiling capacity of 150 gallons was constructed of kauri, which was found to resist 3% sulphuric acid. The acid was heated to boiling point by means of steam passing through a lead coil secured to the inside of the vat. The crude fibre, before being subjected to the acid treatment, was loosened in cold water in order to free the material from the loose sand and to save the short fibres which would otherwise be lost if the fibres were merely shaken. After treatment of the first batch with boiling acid solution the fibre was washed by agitation in cold running water, but at the end of 6 hours it was still found to be strongly acid. This method of washing was considered too long and tedious, with the result that boiling water was resorted to and found to be very effective, the acid being completely removed in 2 hours. The purified product was finally dried in the open air on metal trays. The results obtained with various batches of fibre are given below:—

Batch No.	Weight of fibre taken	Time in acid vat	Time in final washing vat	Ash content
	lb.	hours	hours	%
1	50	3	6	0.28
2	50	3	2	1.80
3	50	3	2	0.70
4	50	3	2	1.2
5	50	3	2	1.27
6	50	16	16	1.09
7	50	3	3½	0.73
8	50	3	3	5.4
9	35	3	2	1.28

In batch No. 1 cold water was used for the final washing and found to be unsatisfactory. In batches Nos. 2 to 6 boiling water was used in final washing and found to be quite satisfactory. For batch No. 7 fresh acid solution was put in the acid vat. For batches Nos. 8 and 9 the same acid was used as for No. 7.

The high ash figure obtained with batch No. 8 is probably due to an error on the part of the foreman; it cannot otherwise be accounted for.

**Conclusion.**—From the work carried out it has been shown that a staple gun cotton can be produced from *Posidonia* fibre, and that the fibre can be purified on a commercial scale so as to render it suitable for nitration. In order to investigate the possibility of manufacturing gun cotton commercially, plant would be required which we have no means of getting at the present time. Enough has, however, been done to provide *prima facie* evidence of the practicability of using this fibre as a raw material for the manufacture of nitro-cellulose should circumstances demand it, and it certainly would appear to us worth while to carry through one or two nitrations on a large scale to elucidate this more completely. This must, however, be left to those having suitable plant at their disposal.

## Annual Meeting, Bristol.

Wednesday, 17th July, 1918.

### COPARTNERSHIP.

BY ERNEST WALLS.

(ABSTRACT.)

The story of British industry is the story of a long series of evolutionary changes. With the advent of the steam engine the self-contained craftsman passed away, and modern developments have brought with them their problems and complexities.

Chemical industry came into being long after the disappearance of the craftsman, and it would therefore be a difficult task to rehabilitate the worker in this industry as a self-contained unit. Our present wages system is the empirical result of the application of various wages theories. The "subsistence theory of wages" was unfortunately applied to "industrial" wages in England at the time of the industrial revolution. Later came the "wages fund theory." Under the previous theory if the workers increased their earnings increased population redressed the balance; under the new theory capital performed the same function, for wages could only come from a fixed fund, and if they were increased beyond the limit thus available, capital was depleted and the fund reduced. Then came the fixed time wage, although the theory of supply and demand associated with it disappeared when the Trade Unions developed and collective bargaining began to make its influence felt. Bonus systems, piece rates, and other methods of gain-sharing gradually appeared, and eventually it was realised that the product of industry was a joint product of capital and labour.

Copartnership, however, starts from an entirely different ethical and scientific standpoint. Under this theory production is joint as between capital and labour, but their sole *raison d'être* is service to the third copartner—the community. True copartnership, as distinct from profit-sharing, seeks suitable means of division of the total product of industry, and makes copartners not only profit-sharers but shareholders in the business. An ideal copartnership would be framed on the following lines. The first charges on every industrial under-



taking are a minimum or standard interest on capital and a minimum or standard wage to labour. Capital being further protected and conserved by necessary charges, such as depreciation of plant, against the earnings of the business, it is reasonable that labour should be similarly protected and conserved by equivalent charges, such as a labour reserve for old age and so on. After charges for capital, labour, and their respective reserves have been met, there remains the residuum which, if excessive, is evidence that the consumer has paid too much for the services, and *vice versa*. The consumers' part of the residuum would be returned to the State in such a way as to ensure its ultimate return to the consumers, while the remainder would be divided as between capital and labour on the basis of the profit-sharing capital and the total wages paid.

The initiation of copartnership schemes in England coincided with the Limited Liability Act, although one scheme is recorded in 1829. Copartnership schemes have existed in France during the last 50 years, and a very notable one, dating back to 1880, is that of Godin of Guise. In England 38 gas companies practice copartnership on a model arrangement drawn up by the late Sir George Livesey, who initiated the South Metropolitan Gas Company's scheme in 1889. The division of the profits with the third copartner is, in the case of gas companies, unique.

As far as the South Metropolitan Gas Company's scheme is concerned, 4% dividend on capital and 3s. 1d. per 1000 cubic feet of gas is taken as a basis. For every penny reduction in the price of gas the shareholders receive an additional 2s. 8d. % and the employees receive  $\frac{2}{3}$ % (15s.) upon the amount of their annual wages. Thus in 1912 the price of gas was 2s. 2d. per 1000 cubic feet, the shareholders received £5 9s. 4d. % dividend and the employees 81% bonus. In this scheme increased efficiency benefits the three copartners, namely, the consumer, the shareholder, and the worker.

It will readily be realised, however, that before the ideal division is obtained, capital will need to be scientifically standardised because of such features as "watered" or written-down capital, and an absolute, as against a relative, meaning assigned to it. Nevertheless, the essential principle is not affected by these difficulties, which are inseparable from the complex character of our modern industrial system.

The method of division adopted by Messrs. Lever Brothers, Ltd., is worthy of notice. Partnership certificates, of purely nominal character and having no exchangeable value, are held by employees and correspond to the share certificates held by the ordinary shareholders. The same dividend is declared both on the ordinary capital (after paying 5% interest) and on the partnership certificates. These partnership certificates are of value only to the owner, but they pass to the widow of the copartner, and upon the death of the survivor they cease to exist. When the copartner is no longer employed by the company his partnership certificates cease to exist, but he is entitled to any accumulated dividends standing to his credit. In the distribution of certificates an attempt is made to distribute the award in accordance with individual merit. In the first place the certificates are based upon a percentage of the previous year's earnings. Secondly, the individual employees are assigned to several classes dependent upon the individual merit of each worker, and in accordance with this grading the percentage on wages may be above or below the average allotment. Appeals from copartners can be made to democratic committees. There is a maximum holding of partnership certificates. As it is impossible to assign to each labour unit its due

share in achieving any realised surplus, the copartners, as in the majority of other schemes, share collectively, but in the case of partnership certificates individual merit is enabled to be appraised and receive its due reward.

As to the form of distribution, almost all the pioneers of copartnership realised that one essential of true copartnership was that of the encouragement of thrift. It is seen in the evolution of the modern industrial system from the old craftsman system that labour and capital in the same individual became necessarily separated. The means whereby the worker may be re-created as the owner of the means of production has been constantly sought by economists and reformers, especially by Mill and Mazzini, and copartnership aims at making every copartner a joint owner of the means of production. Before the war it was calculated that the average capital required per worker in British industry was £200, and this amount invested in the business in which he is a copartner has for the worker under present day conditions a productivity far more than the equivalent of the old craftsman's workshop with £200 worth of tools; there are to-day something like 200,000 copartners in this position. Thus a linking of capital and labour in the sharing of capital as well as in the earnings is the aim of every copartnership.

To encourage thrift the gas company method is to place half the bonus to which the copartner is entitled yearly to a deposit or savings account at 3% interest, and the other half is placed to a trust account for investment in the company's stock. Although all the bonus is the absolute property of the copartner, he must not sell or pledge his stock nor withdraw from his deposit account except under special circumstances. In the author's opinion this seems to restrict individual freedom and thereby to lead to suspicion.

In the case of J. T. and J. Taylor, Ltd., of Yorkshire, surplus profit over 5% is divided at the same percentage rate between capital and labour, and copartnership earnings must be allowed to accumulate in the form of shares by each employee until a sum equal to one year's wages or salary is reached, after which copartnership earnings may be withdrawn.

By the Lever Brothers' Copartnership Trust, founded in 1909, absolute freedom is given to the copartner as to the disposal of his copartnership earnings, but there is a strong incentive in the scheme to induce the copartner to retain them as shares. The dividend on the partnership certificates is distributed in the form of 5s. shares in the business, which form a special block of 5% cumulative preferred ordinary shares, and these a copartner can sell back to the company at par. With the ordinary public these shares carry 5% cumulative dividend, but an active copartner receives an extra bonus, making the total dividend on the shares equal to the ordinary dividend for the year. It is therefore conceivable that under a copartnership scheme based upon such a plan by the effluxion of time, capital held by the copartners will take the whole of the surplus, as has happened in the French scheme of Godin. Shares remain, in Lever Brothers' scheme, the absolute property of the copartner, but on death revert to 5% interest-bearing shares. Those therefore who become shareholders do so by conviction rather than by the less gentle method of conscription adopted by gas companies.

Provision for sickness and old age should be a reserve and a charge prior to the ascertainment of profits, although in many existing copartnership schemes the withdrawal limitation practically makes them into a special form of sickness and old age fund. The copartner should be free to make use of the opportunities copartnership affords him,



but if there are shackles on the opportunity, it is no true copartnership.

In the Godin scheme, dating back to 1880, and in the business of which the members have now become the sole owners, the surplus profit after payment of all fixed charges and 5% on capital, is divided, 75% to capital and labour, and 25% to management. The division of the 75% between capital and labour is based in the case of labour on the total wages paid, and in the case of capital on the fixed interest received by capital. An ingenious method also exists of grading the workers according to ability. Dividends are paid to the workers in the form of savings certificates which receive the same interest as the shares, and on retiring each member receives cash for his certificates, thus carrying with him the cash representing his lifetime's savings. Among other features of the scheme is a community housing scheme. Godin's summing up of copartnership is expressed in his words: "To each according to his capacity, to each capacity according to the service rendered."

The Works Committees advocated in the Whitley Reports are virtually a creation of copartnership, as the management of the trust funds has invariably led to the establishment of a democratic council. In the cases of the South Metropolitan Gas Company and the South Suburban Gas Company this principle has been further extended by the election of three directors of the company by the employee-copartners themselves. Copartnership in profits, however, precedes copartnership in control, and progress can only be gradual.

Properly speaking there are three agents of production, namely, labour, management, and capital, but true copartnership includes both controlling management and controlled labour, and this incidentally solves the difficult problem of remuneration to management. In Godin's scheme management consists of a council selected from the employees with a supreme controller, and such council takes 25% of the surplus.

The interdependence of the units of production is better understood in a copartnership business, and the important part played by management and the possibilities that await enlightened labour respectively are mutually realised. "Copartnership is comradeship, brotherhood in business," said Sir George Livesey. Advancement depends of course on individual effort and determination, and the regular distribution of copartnership earnings in conjunction with a grading plan ensures a continual review of all workers.

The author puts forward copartnership as a positive scheme based upon the existing industrial structure. While in no sense a panacea, it is a scientific attempt at an equitable distribution of the proceeds of industry, and as such, copartnership schemes are successful and fruitful. Failures have almost entirely been failures of false copartnerships. The possibilities are immense, and are no less remarkable from the point of view of productivity, which must inevitably and voluntarily increase. With the passing of years the copartners' savings are accumulated, and a right understanding of copartnership is therefore only a question of time, although some suspicion of a hidden motive must naturally be encountered. Finally, what copartnership needs most of all is an accession of strength, and a source of this might well be found in the chemical industry.

#### DISCUSSION

Dr. CHARLES CARPENTER said that the chemical industry would reap none of the benefits from copartnership after the war unless labour took up an attitude different from that which actuated it to-

day. When Sir George Livesey twenty-nine years ago had been able to put into effect the ideas that had been in his mind for many years, he had described the condition of affairs which then existed as one in which the worker endeavoured to do as little as he possibly could and get as much as he possibly could for it. That appeared to express to a large extent the attitude to-day. But whilst they wanted the highest possible results from the point of view of chemical reactions and also from the point of view of the workmen whom they employed, they did nothing to instruct or educate the average worker. The result was that after he left school it was only the socialist agitator who took any trouble to instruct him as to the real basis of industry. Copartnership educated the worker into a knowledge of the principles upon which industry should be conducted, and having educated the worker on those principles they found that he was not very different from other people. Another aspect of copartnership, which was a most important one, was that it gave the worker an opportunity of providing for old age, and of being independent of those clubs and organisations to which most of the workpeople had been driven. In that respect there was something to be said in favour of what had been described as the Gas Company's system, according to which a man was given his bonus in the form of ordinary stock of the Company. If he died it became his wife's property and it was handed on to his children. It was his property in the real sense of the word and was as inalienable as property should be. In building up systems of copartnership one of the initial difficulties was that as soon as a worker who had never been accustomed to anything but his wages got something in the way of a bonus, he almost invariably spent it very quickly. A great many of the schemes had come to grief because instead of the money being properly used it was wasted, and instead of the workman being better off for it he was worse off. Therefore there was an advantage and not a disadvantage in making it inconvenient for the worker to get rid of his stock as long as he continued in the particular employment of which he was a profit-sharer or copartner. The original idea had been that one half of the bonus should be invested in permanent stock and the other half invested so that the investor could, if necessary, draw upon that fund for temporary assistance.

As regarded the effect of the system on labour, there had been opportunities of making a direct comparison between labour output with and without copartnership, paying the same rate of wages and using identical machinery, and it had been found that there was a great advantage to the credit of copartnership workers. The gas companies were not in a happy position at the present time. Copartnership, or profit-sharing, existed still with them, but there was no profit to divide and the workmen were getting no bonus and only the ordinary wage applicable to the industry. The copartners had approached their committee and after discussing the question of the 12½% award, they had decided that the conditions of the company did not warrant their pressing for the award. That was a very remarkable example of how working men trained in copartnership would look at the question from both sides. Another point was the much debated one on the value of having working men on the board of management. The men themselves would be the first to say that the great bulk of business carried on round the board table was quite beyond them, but there were many matters appertaining to the conditions of employment and work generally where the advice and counsel of the men were extremely valuable. There was also the advantage that the working men knew that they



were directly represented on the managing body. After an experience of many years he would be sorry to depart from the principles practised by the South Metropolitan Gas Co. The future of industry, and the chemical industry no less than any other, depended on how labour could be educated to take its share in the management, and if they took the same pains in getting the maximum yield from humanity as from brains, he was sure they would get as valuable a result from it. The South Metropolitan Gas Company had a combing-out committee of the workmen which had dealt with 2000 cases, quite apart from the authorities, as to whether men were or were not indispensable, and in that way hundreds of men had been released for the army who would otherwise have been going on as usual. At the time of the Boer War, when the allowances to the dependants of soldiers who had been killed or who had died in the war were very small, the copartners took upon themselves the problem of making allowances to the widows and dependent children, so that the latter could be brought up until they reached the age of 16. It was only about the time that the present war broke out that the last of these responsibilities ceased. They had done precisely the same thing in the present case, and many hundreds of cases were being dealt with through committees individually.

Mr. W. D. A. Bost expressed the opinion that profit-sharing ought to be applied to the whole country, although difficulties would be encountered with the small employer having limited capital to whom a return of 5% would not be adequate. It would be necessary to fix a minimum wage before any profit-sharing scheme was devised. With regard to the excess profits tax, he thought that the most rational means for obtaining the objects in view of producing at the cheapest price and competing on the best terms would be to continue that excess profits tax and divide the money so obtained among the workmen either according to trades or uniformly throughout the country. That would be an inducement to the workmen to give maximum output. But the first thing was to arrive at an adequate minimum wage for the lowest paid workmen.

The PRESIDENT, in closing the discussion, said that industry in this country could not survive unless we succeeded in persuading the workers that their interests lay not in producing as little as possible, but as much as possible. Until that economic fact was brought home to the workmen of the country our task would be a difficult one. One drawback to copartnership was that it did not directly reward productiveness. His own view was that some modification was necessary by which the workman was paid in proportion to his production. It was not easy to do this but he did not regard the problem as insoluble. Again he somewhat disagreed with Mr. Walls' view that management should be merged in labour. He thought the copartnership idea would have to be extended to include three partners, *viz.* capital, labour, and administration—by which he meant administration in the very widest sense possible—each of which deserved an adequate reward. There was a great deal in Dr. Carpenter's view that it was distinctly injurious to the working man, certainly until he was better educated economically than he was to-day, to trust him with large sums in the way of bonuses, because the money was often squandered. The manager of one of the largest coal and iron concerns in the country had said that the three years' drink bill of the workmen of that company was equal to the capital of the entire company. With facts like these, one was impressed with the danger of entrusting the workman as he was to-day with lump sums of money.

Mr. W. D. A. Bost writes as follows:—All the examples given by Mr. Walls are actual or virtual monopolies who can raise prices at will to cover any bonuses given, so that those who have actually put money into them will not be losers, and it is the public therefore who pay these bonuses. There is one exception, *i.e.*, Godin, and in this the original shareholders were forcibly paid out. If one or a few firms in any trade give special terms and inducements to their men, they will secure the best men and those who will work so as to secure the benefits aimed at, but if every firm in that trade were to do the same they would all revert to the *status quo ante* unless the terms were such as to obviate this. It is our duty to ascertain whether such terms can be made as would attain this end, and whether they would be immediately acceptable to the men and those who have sunk their money or propose putting money into any business. Any scheme must be such as to induce the investment of capital in home industries. The effect of it must be such as to cheapen production compared with the rest of the world, so that we may sell the extra production and also increase the purchasing power of the wages, thus avoiding the necessity for giving higher wages. Any scheme based on a return on the capital invested would fail, as either small capitalists putting their money into a business to be worked by themselves could get out of it by allowing themselves big salaries, or they would be prevented from starting for themselves. It would also tax industry while leaving the professions and commerce untaxed. Profits arise from capital, management, and cost of production, and no workman (in its accepted sense) could be entitled to share in any but the latter category except under special circumstances. Also if bonuses dependent on profits were paid on the profits of individual firms, the men would be underpaid in one works and overpaid in another, though each might have given equal service. These are a few of the difficulties in the way of a comprehensive, equitable scheme, but I venture to suggest the following crude idea: I divide wages into two portions, *i.e.*, a basis wage variable according to the index number or in any other fair way, and a capital wage being, as it were, interest on time spent in acquiring a trade. I then take the ground that any profit owners may make greater than before the war is due to sacrifices made by all in winning the war and securing our freedom of action. A further profit may be made by cheapening production. If by either or both of these conditions greater profits are made, a part of them should come back to those who have made them possible. If, on the other hand, no extra profit is made they have no right to them. If a greater output is required per man per machine, the machines must be kept capable of turning out the greater output, and far greater allowances for this purpose must not only be made before taxation steps in but be enforceable before profits are divided. There would be probably other safeguards necessary, but if, after the basis wage is paid to unskilled labour and basis plus capital (the addition of these being the same amount as before the war), there should remain a profit greater than before the war over the total number of firms in any particular trade, this should be divided in certain proportions between the owner and the workman, the workman getting a part (spread over the ensuing year) as additional wages but the greater part retained for disbursement later on as old age pensions or provision for incapacity to work through accident or disease. This extra profit would be collected as a tax by Government and the amounts disbursed by firms in extra wages the following year reimbursed to these firms by Government out of the funds at their disposal.

## Communications.

### AGEING OF VULCANISED PLANTATION RUBBER.

#### PART 1.

BY HENRY P. STEVENS.

In a previous communication<sup>1</sup> there were recorded the results of a series of experiments on crêpe and sheet rubbers in which the vulcanised specimens were re-tested physically at intervals of a few months. It was shown that, in those cases in which the coefficient of vulcanisation exceeded the figure 3·2, deterioration set in a short time after vulcanisation, and that the higher the coefficient the more rapid the deterioration.

It was thought desirable to extend these experiments, and a fresh series was begun with a view to carrying out ageing tests over a longer period and to include a wider range of types, among them rubber from matured coagulum (slab). These experiments are now complete as regards ordinary pale rolled sheet and unrolled sheet (slab). Mixings

ordinary fluctuations of room temperature. When comparing the results it must not be forgotten that determinations of breaking strain are subject to appreciable experimental error when the rubber is overcured and brittle or "perished."

In a recent communication<sup>2</sup> it was shown that at summer temperatures the effect of ageing on the physical properties is much greater than at winter temperatures. Consequently the effect of ageing on the physical properties, as brought out in these tests, will be more evident in summer than in winter, and the recorded figures will not be directly related to the period of ageing.

The coefficient of vulcanisation was also determined shortly after vulcanisation and again at the conclusion of the ageing period. The results were as follows:—

Hours vulcanised	(1) Rolled sheet		(2) Unrolled sheet (slab)	
	Beginning of ageing period	End of ageing period	Beginning of ageing period	End of ageing period
2	2'33	2'16	3'16	3'44
2½	2'62	2'72	4'17	4'30
3	3'05	3'33	4'93	5'11
3½	3'98	3'90	5'71	5'81
4	4'36	4'62	6'41	6'88
4½	4'73	4'96	7'01	—

FIG. 1 A. Ordinary Sheet.  
Breaking Strain

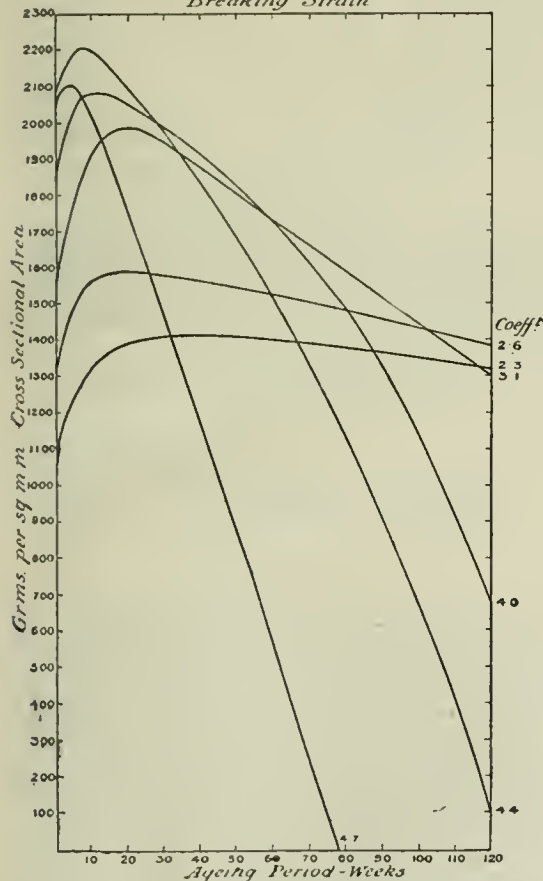
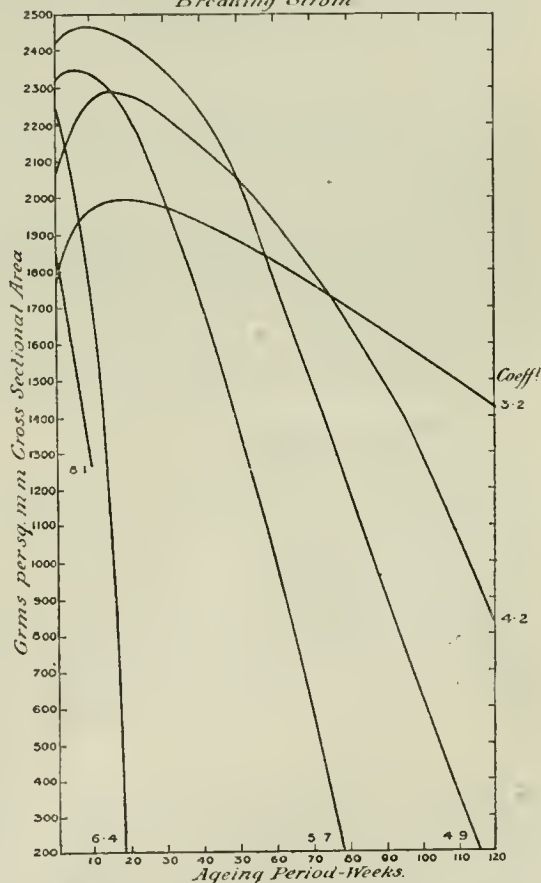


FIG. 2 A. Unrolled Sheet  
Breaking Strain



of 90 parts of rubber and 10 parts of sulphur were cured at 35 lb. steam pressure for 2, 2½, 3, 3½, 4, and 4½ hours, and the vulcanised specimens were tested one week after vulcanising and subsequently at intervals extending over 120 weeks. The specimens were preserved in the dark, or in semi-darkness, but otherwise no particular care was taken with them, and they were exposed to the

These figures indicate that with coefficients of over 3 there is a tendency for the coefficient to increase with the ageing of the specimen, the effect being greater the higher the coefficient. It is probable that this increase is accompanied by decomposition of the rubber. It is well known that sulphuric acid is formed when rubber perishes. It appears, however, that where the coefficient is

<sup>1</sup> This J., 1916, 872.

<sup>2</sup> This J., 1918, 281 T.



low appreciable deterioration does not set in during the period under test, and little or no increase takes place in the coefficient.

The curves plotted for the breaking strain (figs. 1A and 2A) first rise and then fall again when the coefficient does not exceed about 5 units. With higher coefficients the curves fall from the start. Taking (1A), the graphs for ordinary pale sheet, an increase in the breaking strain takes place with ageing at all cures. The higher the coefficient the shorter the period of increase in breaking strain. The figures are approximately as follows:—

(1) Ordinary rolled sheet		(2) Unrolled sheet (slab)	
Coefficient	Period during which increase of breaking strain takes place	Coefficient	Period during which increase of breaking strain takes place
2.3	35 weeks	3.2	21 weeks
2.6	22 "	4.2	15 "
3.1	18 "	4.9	6 "
4.0	10 "	5.7	2 "
4.4	7 "	6.4	nil
4.7	5 "	8.1	"

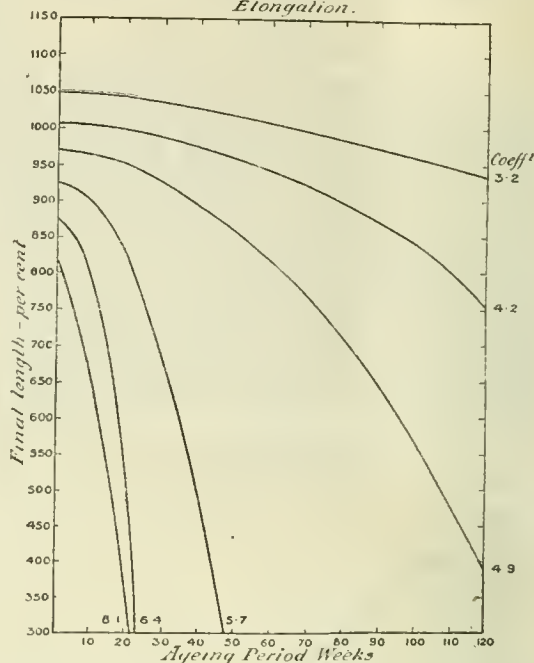
In general the curves are flatter the lower the coefficient. These results confirm the conclusions drawn from earlier experiments, namely, that a rubber vulcanised to give a coefficient exceeding 3.5 is certainly overcured, and cannot be expected to retain its physical properties for a reasonable length of time. To secure approximate permanency the coefficient should not exceed 3. The cure for the coefficient 3.1 with the ordinary sheet (1) corresponds with a curve resembling closely that for the coefficient 3.2 with the "slab" rubber (2), showing that, as regards these two types, at any rate, the coefficient is a fair indication of the lie of the curves.

Finally, it is clearly seen that the maximum breaking strain cannot be obtained without overcuring the rubber to such a stage that it commences to deteriorate within a few days of vulcanisation, and loses tensile strength at an average rate of about 1% per week.

The curves indicating the change in final length of the specimens (figs. 1B and 2B) are of a simpler

nature than the breaking strain curves because there is little or no tendency to an initial increase in elongation with period of ageing, and at the commencement the elongation is inversely as the

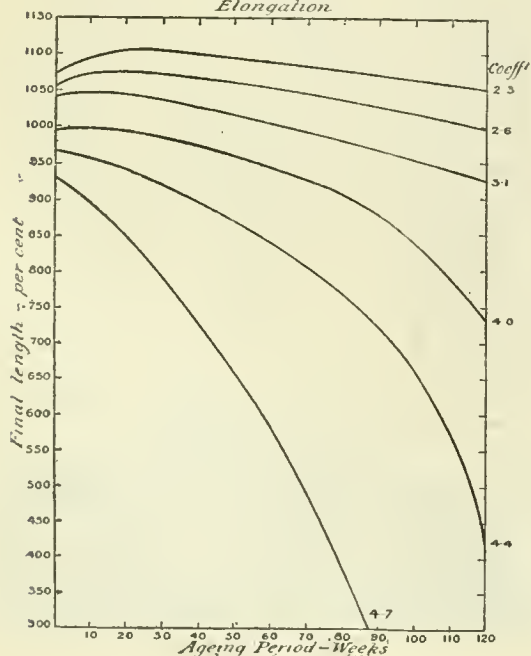
FIG. 2 B Unrolled Sheet.  
Elongation.



coefficient. The curves are flatter and more nearly approach to the horizontal, the lower the coefficient. With coefficients under 3 or thereabouts, the curves approximate to the horizontal. The slope becomes more pronounced the higher the coefficient, and with high coefficients is very steep.

I am indebted to the Rubber Growers' Association for permission to publish this research.

FIG. 1 B Ordinary Sheet  
Elongation.



#### THE INFLUENCE OF TEMPERATURE ON THE DECOMPOSITION OF SODIUM CARBOLATES AND CRESYLATES BY CARBON DIOXIDE.

BY G. J. DENBIGH, B.S.C.

The fractions of coal tar which contain the bulk of the tar acids are crude naphtha, light oil, and carbolic oil, and the crude acids are extracted from these by washing with caustic soda solution. The separation of the crude acids from the sodium salts is effected most economically by decomposing with carbon dioxide, thus regenerating sodium carbonate which can be again causticised and used for the further treatment of fresh oils. The crude acids thus obtained are neutralised and partly dehydrated by treatment with a small quantity of sulphuric acid.

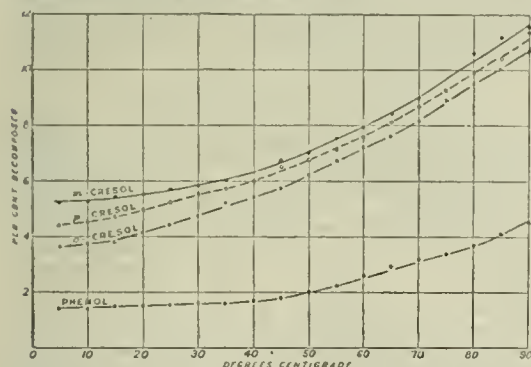
In the course of superintending the working of a crude carbolic plant, the author noted the apparently abnormal decomposition by the carbon dioxide. This was made in a small lime kiln, and after purification usually contained 17–20% of  $\text{CO}_2$ . Careful testing of the gas proved that a small variation in the percentage had little or no effect on the rate of decomposition, so attention was directed to the effect of temperature.

In the first instance gas was bubbled through an ordinary cresylate solution obtained from the plant, and it was at once evident that decomposition was

much more rapid at high temperature than at lower ones. More careful experiments were then arranged, using pure chemicals for the necessary solutions.

For this purpose phenol with a solidifying point of 40.5° C., *o*-cresol, sol. pt. 30° C., *p*-cresol, sol. pt. 35.4° C., and *m*-cresol were obtained, the purity of the latter being ascertained by Raschig's nitration test to be 99.4%. In each case the calculated amount was dissolved in 800 c.c. of a 10% solution of sodium hydroxide and the solution diluted until titration with *N*/1 sulphuric acid showed the solutions to be exactly 2*N*, using phenolphthalein as indicator. Carbon dioxide generated in a Kipp's apparatus was passed through a solution of sodium bicarbonate and collected in a gas holder. A measured quantity (2½ litres) was then slowly bubbled through 100 c.c. of the sodium phenoxide solution. This represented a considerable excess of gas. The phenoxide solution was afterwards shaken with 50 c.c. of benzene, in two portions; after standing for an hour the benzene separated into a well-defined layer. The undecomposed phenoxide was removed and 20 c.c. and 10 c.c. of a 10% solution of sodium hydroxide successively shaken with the benzene solution to re-form sodium phenoxide. After again allowing to settle, this was run off, heated to 80° C., cooled, and decomposed with 30 c.c. of a 25% solution of sulphuric acid, and the liberated phenol measured in a small graduated cylinder. These experiments were repeated with the same quantities at different temperatures, the results obtained being exhibited on the accompanying curves. The volumes of carbon dioxide used were

Graphs showing the influence of temperature on the rate of decomposition of sodium carbolate and cresylates by carbon dioxide.



corrected for temperature and pressure, and the temperature of the phenoxide or cresylate solution regulated by a thermostat. The chief difficulty found in the work was in separating the benzene solution, at least one hour being required for proper separation.

The results established are:—(1) That all the cresols are more easily liberated than phenol. (2) That the cresols only differ slightly, *m*-cresol being the most easily liberated, *o*-cresol the least easily. These results agree fairly well with the other known combining powers of the cresols. (3) That in the case of both phenol and cresols the decomposition is more complete when warm, and that the rate increases more rapidly with all the cresols than with phenol.

From these data it might be considered possible to separate cresol from phenol by fractional decomposition. This is possible to a certain extent; but, like fractional washing, which gives a carbolate containing some cresylate or a cresylate containing some phenoxide, it is by no means as complete as could be desired. Thus a carbolate containing

20% of tar acids of which 30% was phenol was decomposed in two stages; the crude tar acid obtained from the first part was distilled and found to contain 15% of phenol, that from the second part 45%.

Other experiments bearing on the same point gave the following results:—

In tar acids of carbolate	% Phenol In first part decomposed	In second part decomposed
43	22	63
30	15	44
35	22	46
12	4	19
10	4	14.6

These results are naturally incomplete, but it is hoped by further experiments to ascertain more accurately the relative proportions of phenol and cresol liberated in different stages of the reaction, and the possibility of separating them by working at a temperature of 90°–100° C.

In conclusion, I desire to thank Messrs. E. A. Brotherton and Co., Ltd., in whose laboratories the work was carried out, for permission to publish these results.

## THE INTERCONNECTION OF ECONOMIC BOTANY AND CHEMICAL INDUSTRY.

BY EDWARD WHEELER, A.C.G.I., A.I.C.

(ABSTRACT.)

By economic botany is understood the study of those plants, cultivated or wild, which are or may become industrially important. The work of an economic botanist is firstly to investigate the characteristics of plants of ascertained or prospective value with the factors of yield and distribution; secondly, to determine how far the conditions of soil, manuring, and climate influence the yield and quality of the raw material obtained; and thirdly, to endeavour to improve the plant by seed selection or breeding experiments so as to obtain a strain best suited to different localities and ranges of climate, or most immune to diseases.

The aim of economic botany has been recently very tersely summarised by Rendle, who stated that "improvement from an economic point of view implies co-operation of the botanist and plant breeder. The student of experimental genetics, by directing his work to plants of economic value, is able, by means of agriculture and horticulture, to produce forms of greater economic value" (1).

A brief consideration of the problems met with in the manufacture of the principal products prepared by the different chemical industries from the raw materials furnished by the economic botanist will indicate how far economic botany has assisted chemical industry, and in what directions further work is desirable.

### 1. Vegetable dyestuffs.

One of the chief obstacles to the successful cultivation of the indigo plant, namely, its liability to the disease known as "wilt," brought about by the constantly wet condition of the soil, has been overcome by discovering the most suitable planting areas and seasons (2), and by breeding a strain of the plant immune to the disease (3). Again, the yield of indican has been found to be dependent on the conditions favouring the maximum absorption of nitrogen from the air by the root nodules of the plant (4).

The recent work of Howard and Davis in India in studying the fermentation changes taking place in the vats, whereby the indican is converted into indigo, has demonstrated the importance of control-



ling these changes for the production of a higher yield of dye and a more uniform product (5).

There are many other less important vegetable dyestuffs which by improved methods of cultivation, of breeding and selection of the plants, or of extraction of the colouring matter, might be successfully exploited, as in so many cases the crudest methods of production were still in use when coal tar dyes replaced the natural products.

### 2. Cellulose products.

Cellulose for industrial purposes is obtained from (a) the pecto-celluloses such as cotton, ramie, flax, and hemp, (b) the ligno-celluloses such as jute and the woods, (c) cereal straws and grasses.\* The problems met with in the cultivation of the plants and isolation of cellulose may be briefly alluded to (6).

*Cotton*.—For the successful cultivation of the cotton plant (*Gossypium*) attention has to be paid to the climate and soil, as slight variations in these factors always give rise to variations in the cotton crops from season to season as regards yield of waste on spinning and general quality of the fibre. Recent work in Egypt has aimed at obtaining a pure strain of cotton plant which will yield cotton of greater regularity in length and tensile strength (7).

*Flax*.—In the case of flax (*Linum usitatissimum*) study has been devoted to the cultivation necessary to obtain good yields of both fibre and seed (8), the latter furnishing linseed oil. Research into the isolation of the fibre from the stems by retting—a fermentation process—has led to the use of pure bacterial cultures in order to obtain fibre of good quality and uniformity (9).

*The grasses*.—The grasses include the straws, bamboo, and esparto. Interest in the use of straw cellulose (10) has recently revived owing to the increased production of cereals in this country, while the utilisation of bamboo cellulose has been carefully studied by Pearson and Raitt in India (11).

*The woods*.—The study of the best conditions of growth in relation to climate and soil, rate of growth, pathology of trees, and allied investigations carried out by the Forest Departments will prove of increasing value to the cellulose industries (12).

*Paper*.—Constant investigations are being carried out by various workers and by the Imperial Institute as to the suitability of various vegetable fibres for papermaking. In time special crops may be cultivated solely for the production of papermaking materials (13).

*Cellulose nitrates*.—The cellulose nitrates are generally manufactured from waste cotton obtained from the textile factories. For nitration purposes uniformity of cotton is most important. It has been claimed that cotton produced in a cold, wet season does not nitrate so readily as cotton grown under more favourable conditions (14). Consequently improvements tending to give cotton of greater uniformity will also affect favourably the uniformity of the waste cotton.

Both cotton and wood cellulose have been used as raw materials for cellulose nitrate for artificial silk manufacture, though the "silk" made from the former was found to be superior to that made from the latter (15).

*Cellulose xanthate*.—Cellulose xanthate (known in solution as viscose) has been most successfully utilised for the manufacture of artificial silk, wood cellulose being the raw material employed (16). Variations obtained in tenacity and dyeing properties of artificial silk are sometimes ascribed to the original wood cellulose used. A study of the

best time of cutting the wood, the best type of tree, and its conditions of growth in relation to the wood cellulose produced from it would be of value in relation to papermaking and artificial silk manufacture.

### 3. Potash and iodine.

Our knowledge as to the most profitable kinds of waste wood and plants to exploit as sources of potash is very meagre, and there is much need for a systematic survey of the potash content of the ashes from such waste material. Thus it is only recently that the use of bracken and hedge clippings (17) and of banana skins and stalks (18) has been advocated for potash production.

Extended workings of seaweed for potash and iodine have been commenced recently in British Columbia and the United States, but the results so far have not altogether realised expectations (19). As Hendrick has pointed out, a survey of the seaweed supplies and their rate of renewal, the potash and iodine distribution in different varieties, and the best methods of isolating the products, are all questions to be answered if the industry is to hold its own economically (20).

### 4. Vegetable oils, fats, and waxes.

Amongst products prepared from natural oils, fats, and waxes, for soaps are required mainly a cheap and plentiful supply of suitable oils, for food products as margarine an almost colourless, odourless, and tasteless oil, for paints and varnishes good drying oils, and for lubricants non-drying or less strongly drying oils. The number of oils satisfying such requirements are many, and the work of the economic botanist is chiefly concerned with the question of increasing the existing sources of supply and studying the physiological processes underlying the nutrition of the plant, and the effect of fungoid and insect diseases in relation to their oil-bearing capacity (21).

### 5. Varnishes and lacquers.

The qualities required of an oil varnish are good colour, consistence, drying properties, lustre, durability, and flexibility. These are furnished *par excellence* by the fossil copals; but there seems no reason why, in the future, present day trees should not furnish as good material as the fossil products provided that the economic botanist or forest expert is able to control the methods of cultivation and tapping of the trees, so as to accentuate such qualities in modern resin (22).

There is a considerable need for physiological and chemical research in order to understand the conditions necessary for the formation and exudation of all resins and the changes undergone by them whereby they become of value as raw materials for varnishes and lacquers.

### 6. Indiarubber.

*Hevea brasiliensis* (23) has been carefully studied as regards the influence of climate, soils, manuring, straight *versus* forked trunks, etc., on the yield of latex and the best conditions and times for tapping (24). Generally it has been found that plantation rubber lacks uniformity and shows variations in its appearance, especially noticeable in its behaviour on vulcanisation.

As Dunstan pointed out in 1914 (25) the origin, nature, and functions in the tree of the latex, which carries the rubber, are not known precisely.

### 7. Vegetable tannins.

The scientific exploitation of the tannin-yielding plants has recently increased; thus in India the investigation of the best species of mangrove for the production of mangrove tannin has been undertaken (26), while in Natal *Acacia mollissima* has been

\* The classification adopted is that given by Schwalbe in "Die Chemie der Cellulose," pp. 392, 459.

acclimatised and successfully cultivated, good yields of wattle tannin being obtained.

A considerable amount of work has also been done in Sicily (27), India (28), and America on the cultivation of sumac, questions as to the kind of planting, methods and seasons of harvesting, and age of plant being correlated with the yield of tannin, work which might be usefully extended to other sources of tannin.

A research into the physiological processes involved in the formation of tannins in the barks of woods would probably yield useful knowledge as to the best periods and methods of harvesting.

#### 8. (a) Sugars.

The sugar cane (*Saccharum officinarum*) has been cultivated for many years in tropical countries, and much work has been done in determining the best conditions as regards the choice of suitable fertilisers (29), the selection of the best cane for different soils and climates as in Java (30) and India (31), yet a recent writer has observed that so little is known as to the essential botanical characters of the various types of sugar cane that experiments on Mendelian lines should be started, as at present no fixity of type can be assured, for even if improved canes are produced they disappear in eight years (30).

The cultivation of the sugar beet (*Beta vulgaris*) has been most successfully carried out in European countries, where as a result of continuous research work, the sugar content has been raised from 5–6% to 14–18% (32), while the ash content has been reduced from 3.8% to 2.7% (33). Experimental agriculture carried out in this country has shown that the beet can be cultivated here as successfully as abroad (33 and 34).

#### 8. (b) Starches.

The chief source of starch for alcohol production is barley, which has been carefully studied with a view to the production of the most suitable kinds for the brewing industry (35). Other sources of starch for alcohol production are potatoes and maize (36), the starch content of which has been materially increased by pedigree selection and breeding experiments for the purpose. Reichert has suggested that by methods of breeding it may be possible to produce plants containing starch with the desired properties for textile works more pronounced (37).

#### 9. Fermentation products.

The study of mycology is of great importance to chemical industry in two respects: firstly, in regard to the study of those fungoid diseases which so often affect the yield and quality of many raw materials; secondly, in regard to the study of those decompositions which can be more readily and cheaply brought about than by other agencies.

Besides alcohol, other products are obtained industrially by means of the bacteria or moulds; thus lactic acid is obtained from sugar by means of *Bacillus Delbrückii* and *B. longissimus*; acetic acid from alcohol by means of *Bacterium aceti*; while the moulds *Aspergillaceae* ferment glucose solutions producing citric and oxalic acids.

Doubtless in the future many more products will be made industrially by such agencies; thus, for example, the study of the fermentation of cellulose waste is almost an unexplored field, while the extended knowledge of well-known fermentation processes may be of great benefit to chemical industry, e.g., in the cases of indigo and leather.

#### 10. Foodstuffs.

The conditions affecting the regularity of the growth of wheat are dependent on its supply of food, water, warmth, air, light, the absence of poisons and pests, the possession of straw strong

enough to support the plant, all of which have an influence in one direction or another on the strength of the flour (38). The work of Biffen and Howard on Mendelian lines in producing strains of wheat resistant to rust and giving the best results as regards yielding power and quality may be mentioned (39).

The methods involved in the cultivation and production of tea, coffee, and cocoa all necessitate careful control to enable the best quality to be put on the market.

#### 11. Pharmaceutical products.

**Alkaloids.**—The amounts of the three principal alkaloids present in *Papaver somniferum*, morphine, narcotine, and codeine, vary according to the variety of plant, methods of cultivation, collection, and preparation of the raw materials adopted, questions which are being studied in India at the present time (40). In the case of the mydriatic alkaloids, considerable research work has been carried out in determining the influence of such factors as the effect of fertilisers (41), weather conditions, time of collection (42), and geographical area (43), on the content of alkaloid in the plant. Again by suitable selection the content of quinine in cinchona bark has been raised from 3% to 10% (44).

There is undoubtedly much work to be done in similar directions with other alkaloids, while the study of the mechanism by which the alkaloids are synthesised in the plants may be of great importance to the plant breeder (45).

**Essential oils.**—Work has been carried out recently in India (46) and by Baker and Smith in Australia (47) in the direction of surveying trees available as sources of oleo-resin, in studying the most suitable methods of tapping them, and the quality and yields of turpentine obtainable from such resins.

A very careful study of the distribution and quality of camphor obtained from trees of different age has been recently carried out by the U.S. Department of Agriculture (48)—work of great importance if the natural product is to compete successfully with its synthetic rival.

The botanical survey that has been carried out in the eucalypts by Maiden (49) and the chemical work by Baker and Smith (50) might with advantage be extended to many other species.

Generally in the essential oil industry there is much need for correlating the conditions of cultivation and breeding with the quantity and quality of the oil obtained. The metabolic processes underlying the production of the various constituents of the essential oils which in many cases render them so valuable are not understood (51). Moreover, the right period for separation of the oil will only be found from a study of the physiological changes taking place in the plant (52).

#### Conclusion.

Much work has been done by the Rothamsted and other experimental agricultural stations, by the different experimental stations connected with the rubber and sugar industries, by the different forest departments such as those in India (31) and the United States (53), and by the Imperial Institute. Nevertheless, the manufacturer has to deal with raw materials received from a distance and is consequently often out of touch with the cultivation and preliminary preparation of the raw material he receives and is unable to indicate directly to the economic botanist what his special requirements are. Hence the formation of a Raw Material Research Association would seem desirable for studying the problems connected with the cultivation, breeding, and diseases of plants and the furnishing of the supply of raw materials in relation to the requirements of the different chemical



manufacturers concerned, a special department or section being formed for each industry or raw material. In this way the interconnection of economic botany and chemical industry would be strengthened to the mutual advantage of science and commerce.

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## Edinburgh and East of Scotland Section.

Meeting held at Edinburgh on October 15th, 1918.

MR. D. R. DOTT IN THE CHAIR.

### A SPECIAL BLEACHING POWDER FOR USE IN HOT COUNTRIES.

BY THEODORE KETTIE, D.Sc., J. LORRAIN SMITH, F.R.S.,  
AND JAMES RITCHIE, M.D.

The use of hypochlorite solutions in surgery has now become world-wide, but great difficulty is encountered in tropical countries in preparing solutions of a definite composition owing to the instability of hypochlorite compounds. Ordinary commercial bleaching powder which, apart from electrolysed sodium chloride solutions, is the source from which all hypochlorites are derived, is unfortunately a very uncertain and unstable compound and deteriorates rapidly at temperatures considerably lower than those encountered in even moderately hot countries.

Summer temperature in Egypt varies from 98° F. (37° C.) on the coast to 117° F. (47° C.) in the Sudan: the deterioration of bleaching powder in Alexandria is so great that a sample assaying as low as 11% "available chlorine" is still regarded as of good quality. In a laboratory experiment we found that a sample which assayed originally 34% "available chlorine" when exposed to a temperature of 37° C. (98.6° F.) was reduced to 1% strength in 8 weeks, while at 45° C. (113° F.) and 50° C. (122° F.) the available chlorine had completely disappeared in three weeks and a fortnight respectively.

It is obviously impossible, without continual testing by chemical methods, to make up solutions for surgical or general disinfection purposes from a product which is steadily deteriorating, and in a field hospital testing is quite out of the question. We have been experimenting for a considerable time past with a view to produce a hypochlorite compound which could be relied on to give a fairly constant assay value under the conditions met with in the tropics and have succeeded in producing a bleaching powder which loses at a temperature of 45° C. from 3 to 10% of its efficiency in 3 months instead of, as with ordinary bleaching powder, losing the whole of it in 3 weeks.

Calcium oxychloride reacts in presence of water giving calcium hypochlorite and calcium chloride: the moisture in the bleaching powder therefore contains calcium hypochlorite and calcium chloride in solution, and it is the decomposition of this solution of hypochlorite which causes the instability of the bleaching powder.

The reactions involved are extremely complicated, but at ordinary temperature, *e.g.*, 15°–20° C., the main reaction may be represented by the equation

$$2\text{Ca}(\text{OCl})_2 = 2\text{CaCl}_2 + \text{O}_2.$$

At higher temperatures, *e.g.*, 45° C., the reaction is more complex: the main feature is the oxidation of hypochlorite to chlorate, but free chlorine and water are also formed. The formation of water is important as tending to hasten the decomposition of the remaining bleaching powder. The formation of chlorate is of considerable importance, as its presence is undesirable when the bleaching powder is to be used for sterilising drinking water.

In our first experiments we endeavoured to prevent the decomposition of the bleaching powder by removing the water by desiccation. It is very difficult to remove the last traces of moisture by this method and a considerable amount of chlorine is lost in the process. A good bleaching powder

assaying 31.95% available chlorine, when dried *in vacuo* over sulphuric acid at room temperature for three days, lost 6% of its "available chlorine," and this dried powder in sealed tubes when incubated at 37° C. for 2 months lost 92% of its "available chlorine." Another sample of the same powder was left at room temperature *in vacuo* over sulphuric acid for 8 months: at the end of that time it assayed 35.14% "available chlorine" and still contained 0.8% of moisture. This almost dry powder when heated to 45° C. for 10 weeks lost 50% of its "available chlorine," a decided improvement on ordinary bleaching powder which as before stated lost 100% of its "available chlorine" in 3 weeks. This desiccated powder was very hygroscopic, rapidly re-absorbing enough moisture to effect decomposition.

We then tried mixing an absorbent with the bleaching powder, and by using freshly ignited quicklime for this purpose, have succeeded in producing a dry powder which will stand a severe test. A mixture of freshly ignited quicklime with bleaching powder in equal proportions kept at a temperature of 50° C. for 6 weeks lost 9% and another sample of the same mixture at 37° C. lost only 4% of its "available chlorine" in 3½ months.

The bleaching powder on the market at the time these experiments were carried out, was not of such good quality as that obtainable before the war: the best samples we could procure assayed 33 to 35% "available chlorine," whereas 36 to 38% was the pre-war standard. The moisture varied from 9.5 to 16%.

Theoretically a bleaching powder containing 10% of moisture requires 31 grms. quicklime per 100 grms. to absorb the water: or a mixture of 76.4% bleaching powder and 23.6% lime should be quite dry, all the water having gone to form calcium hydroxide. The quicklime used in our experiments was prepared by igniting marble and contained 92% CaO, therefore the proportion for the above should be 74.4% bleaching powder to 25.6% quicklime, in actual practice better results are got if excess of lime is used: in this case the proportion should be 70% bleaching powder to 30% CaO.

Seven samples of bleaching powder were used in the experiments detailed in the tables: they are numbered A to G and are referred to in the tables under these letters—

		" Available chlorine "	Moisture
		%	%
Sample A	...	34.08	9.7
" B	...	33.37	12.6
" C	...	28.75	15.6
" D	...	35.5	16.0
" E	...	31.06	—
" F	...	35.14	0.8*
" G	...	28.0	— †

\* Sample dried *in vacuo* over sulphuric acid.

† Bulk sample kept in cask for disinfection purposes.

Table I. gives details of some experiments with unmixed bleaching powders: they show very rapid deterioration and serve as controls to the experiments in Table II. The percentages of calcium chlorate in the samples are shown in the table as they are referred to later in connection with the sterilisation of drinking water.

Table II. is in three sections, giving experiments at 45° C. and 37° C., and also showing the difference in the results when the samples are in sealed glass tubes, or in corked bottles. The difference in the results in experiments 1 and 18 is very striking: both samples were made up at the same time and received the same treatment throughout, except that No. 1 was sealed up in glass tubes, and No. 18 was in a wide-mouthed bottle with a tightly fitting cork. There was a considerable pressure of gas



in the sealed tube after 15 weeks, the gas was colourless and contained hypochlorous acid: it is probable that the pressure of gas establishes equilibrium in the system and prevents further decomposition. This and other chemical problems encountered in the course of these experiments are being further investigated and will be dealt with in a subsequent communication.

Another point requiring further investigation is brought out in Exp. 7, sample D: this sample assayed 16% moisture and 35.5% "available chlorine" and when mixed with lime showed a distinct loss of strength, the 30% mixture assaying 22.36 "available chlorine" instead of 24.85 theoretical: in sample A with 9.7% moisture to 34% "available chlorine" the loss is not so great—the actual value is 23.07 instead of 23.85% theoretical. This reaction is of considerable importance and shows that it is advisable to choose a sample of bleaching powder containing as low a percentage of moisture as possible for preparing the quicklime bleaching powder mixture.

Referring to the figures in the column showing percentage loss of "available chlorine," two experiments, Nos. 4 and 7, show a much higher loss than the others: in both cases the amount of lime in the mixture was below the theoretical required for absorption of moisture in the samples. In the other experiments in Section I, where excess of lime was used, the decomposition was very much reduced. The figures in this column should be compared with those in the last column of Table I. As a general conclusion from the tables it may be stated that if a bleaching powder containing as low a percentage of moisture as possible is mixed with one-fourth more than the theoretical amount of freshly ignited quicklime necessary to absorb the moisture, a product will be obtained which, when suitably packed in air-tight containers, will withstand any temperature likely to be encountered in the tropics. The question of packing in bulk must be tried on a commercial scale, and arrangements have been made to have this done.

For the preparation of "Eusol" the new quicklime bleaching powder can be used without any difficulty: if allowance is made for the fact that the average value of the new powder (containing 30 to 40% CaO) is from 20 to 23% "available chlorine."

As remarked earlier in this paper, the presence of calcium chlorate in a bleaching powder which is to be used for sterilising drinking water is very undesirable owing to its unpleasant taste. In commercial bleaching powder the percentage of

chlorate is very small, and when the bleaching powder deteriorates at ordinary temperature very little chlorate is formed. Pattinson<sup>1</sup> showed that no chlorate was formed in 12 months at 15° C.: 1% was formed at 21° C.: and 3.7% at 26.5° C. Continuing the experiments at higher temperatures we obtained the following results:—9% of calcium chlorate formed in 8 weeks at 37° C.: 10.92% in 4 weeks at 45° C.: and 9.0% in 3 weeks at 50° C. The intermediate figures are given in detail in Table I.

A method employed for sterilising drinking water in the field is to add a definite quantity of bleaching powder to successively increasing measured quantities of water in suitable vessels till a point is reached at which the solution just reacts for free chlorine when tested with potassium iodide starch paper: from the data thus obtained the amount of bleaching powder to be added to the water tank is calculated. It is obvious from Table I. that at a high temperature deterioration is rapid and the amount of calcium chlorate formed is large. In applying this method in a hot country more bleaching powder is needed on each successive occasion and therefore more calcium chlorate is added till a point is reached when the water becomes absolutely undrinkable. Of the complaints which are made with regard to chlorinated drinking water the most bitter come from the Mediterranean area: this is probably due to the amount of chlorate which has been formed when the bleaching powder deteriorated at a high temperature. With quicklime bleaching powder the conditions are much improved, as may be seen from the figures in the last column on Table II. Waters were made up from different samples of ordinary and quicklime bleaching powder which had been exposed to high temperatures, and it was found that those from the bleaching powders containing 6 to 7% calcium chlorate were much more disagreeable than those from the quicklime powders, none of which contained more than 1.3% calcium chlorate.

*Conclusion.*—Bleaching powder in hot countries loses efficiency owing to the moisture present in the powder. If quicklime be added it combines with the moisture and to a large extent prevents decomposition of the hypochlorite.

The details set forth in the paper show that there is much room for improvement in the present method of supplying this important chemical to tropical countries. The treatment described has the advantage of a conservation of efficiency with a consequent saving in shipment.

<sup>1</sup> This J., 1888, 188.

TABLE I.

Exp. no.	Sample	T.mp.	Percentages of "available chlorine" and calcium chlorate after exposure for						Loss of "available chlorine"	
			0 week	1 week	2 weeks	3 weeks	4 weeks	8 week	Per 100 grms.	%
1	A	37° C.	34.08 0.75	28.57 1.5	—	—	15.26 5.4	1.21 9.0	32.87	96
2	"	45° C.	34.08 0.75	—	9.58 7.5	—	0.16 10.92	—	33.92	99
3	"	50° C.	34.08 0.75	8.1 6.5	—	Nil 9.0	—	—	34.08	100
4	B	45° C.	33.37 —	—	4.68 —	—	—	—	28.69	86
5	C	45° C.	28.75 —	—	0.5 6.75	—	—	—	28.25	98
6	D	45° C.	35.5 —	—	0.88 12.0	—	—	—	34.62	97
7	F	45° C.	30.14 —	30.53 —	29.46 —	—	5 weeks 26.92 3.0	10 weeks 17.75 6.15	17.39	50
8	G	15° C. to 20° C.	23 —	—	after 3 years			11.14 1.17		

NOTE.—The upper figures in columns 4 to 9 are percentages of "available chlorine" and the lower figures calcium chlorate.

TABLE II.

Experiment and sample	CaO Theory	CaO Present	Percentage of available chlorine after exposure for								Loss of available chlorine		Calcium chlorate	
			0 week	2 weeks	5 weeks	8 weeks	9 weeks	13 weeks	15 weeks	Per 100 grms. powder	%	Original	Final	
Samples heated in sealed glass tubes at 45° C.														
1 A	25	30	23.07	22.18	—	22.18	—	—	22.18	grms.	0.89	3.8	0.52	1.2
2 B	30.5	40	20.02	19.17	18.28	—	18.5	—	—	1.52	7.5	0.79	1.2	
3 „	„	50	16.15	15.79	15.08	14.9	—	14.9	—	1.25	8.1	0.6	0.9	
4 C	35	30	20.12	17.75	14.2	—	—	—	—	5.92	29.4	—	—	
5 „	„	40	16.68	16.33	15.62	—	15.17	—	—	1.51	9.0	—	—	
6 „	„	50	14.37	13.12	—	—	—	12.78	—	1.59	11	—	0.6	
7 D	16	30	22.36	21.30	20.23	—	17	—	—	5.36	23.9	—	—	
8 „	„	40	20.23	18.1	17.92	—	17.92	—	—	2.31	11.4	—	—	
9 „	„	50	15.97	15.26	—	—	—	14.75	—	1.22	8	—	1.05	
Samples heated in sealed tubes at 37° C.														
10 B	30.5	40	20.02	18.46	18.46	18.1	—	18.1	—	1.92	9.5	—	—	
11 „	„	50	16.15	—	16.15	—	—	—	15.97	0.18	1.1	—	—	
12 C	35	30	20.12	18.1	17.75	17.75	—	17.39	—	2.73	13	—	—	
13 „	„	40	16.68	16.33	15.26	15.62	15.17	—	—	1.51	9	—	—	
14 „	„	50	14.37	12.35	—	—	12.78	—	—	1.59	11	—	—	
15 D	36	30	22.36	21.30	21.30	19.52	—	—	19.87	2.49	11	—	1.35	
16 „	„	40	20.23	18.46	18.46	18.63	—	17.92	—	2.21	10.9	—	—	
17 „	„	50	15.97	15.26	—	—	—	15.62	—	0.35	2	—	—	
Samples heated in corked bottles at 45° C.														
18 A	25	30	23.07	22.01	20.59	18.81	—	16.68	—	6.39	27	0.52	1.2	
19 „	„	40	19.88	19.17	18.46	17.04	—	—	13.49	6.39	32	0.45	1.0	
20 B	30.5	30	23.29	19.52	—	17.39	—	15.62	—	7.67	32	—	—	
21 „	„	50	16.15	15.79	15.08	14.9	—	14.9	—	1.25	7.7	—	—	
22 E	—	30	21.74	20.95	—	—	—	—	19.17	2.57	11	—	—	

## Nottingham Section.

Meeting held at University College on October 30th, 1918.

MR. F. H. CARR IN THE CHAIR.

### NOTE ON TRYPSIN, AND A NEW METHOD OF PURIFYING ENZYMES.

BY JOSEPH T. WOOD, F.I.C.

In a paper entitled "A new method of isolating trypsin," L. Holzberg<sup>1</sup> refers to some work carried out by Robertson,<sup>2</sup> who found that when one drop of a saturated solution of safranin is added to 5 or 10 c.c. of neutral, or very faintly alkaline, 0.5% solution of Grüber's trypsin, a light, flocculent, coloured precipitate slowly appears and gradually settles. It was assumed that the substance precipitated by safranin is actually trypsin, since the supernatant liquid, although containing substances precipitated by alcohol, is practically free from enzymes. Holzberg has repeated Robertson's experiment. To solutions of Grüber's trypsin, Fairchild's trypsin, and aqueous extracts of sheep's pancreas, he added three-eighths of the volume of a 0.8% solution of safranin. The precipitates were collected and washed, and their proteolytic action tested by digesting with a solution containing 2 grms. of casein (Gross test). The results are given in the following summary:—The substance which is precipitated by the addition of safranin to aqueous solutions of Grüber's or Fairchild's trypsin, or to aqueous extracts of pancreas, has a strong proteolytic action, contains safranin, is very sparingly soluble in water, and insoluble in organic

solvents. After the substance has been removed from aqueous solutions of commercial trypsin or pancreas extract, a considerable quantity of a substance remains in solution, which is precipitable by alcohol, and in the case of pancreas extract, a further precipitate is obtained by means of an alcohol-ether mixture (3 to 1). These substances are practically devoid of proteolytic activity.

I repeated this experiment by adding a 0.1% and also a saturated solution of safranin to various infusions containing trypsin. An aqueous extract of the pancreas gave a considerable precipitate, which behaved in exactly the same way as the precipitate obtained by Holzberg. Instead of using the Gross test, I used the gelatin test (this J., 1912, 1105). I found it impossible to separate the precipitate from the filter, as it dried into a horny film. The paper was therefore cut up into small pieces, and in order to obtain some quantitative data it was cut into one-inch squares, which were weighed, and these were then ruled into 100 divisions and the small squares placed in the gelatin tubes. It was found that six squares liquefied, but five did not. The approximate weight of precipitate on one square inch, assuming it to be uniform, was 5.5 mgrms. The precipitate on 0.01 sq. in. would therefore be 0.055 mgrm., so that the quantity of dry precipitate which is active lies between 0.33 and 0.27 mgrm.

Since the whole of the gelatin was liquefied, it was evident that the substance causing the liquefaction was soluble, and it therefore seems that Holzberg, by washing his precipitate, has removed considerable quantities of enzyme. On repeating the experiments with one of my own trypsin preparations, which was comparatively free from albuminous matter, I was surprised to find that no precipitate was formed, i.e., the ordinary preparations of trypsin, and aqueous extracts of pancreas, gave precipitates, but the purified preparation (the strength of which was equal to that of the crude

<sup>1</sup> J. Biol. Chem., 1913, 14, 335.

<sup>2</sup> J. Biol. Chem., 1907, 2, 317.



preparation) gave no precipitate. It appears, therefore, that the matter precipitated by safranin is an albuminous compound, which carries the enzyme down with it, and that the precipitation by this method does not produce an enzyme-safranin compound. The method appears to be analogous to that used by Cohnheim<sup>3</sup> for the precipitation of diastase from saliva by means of calcium phosphate, in which a certain quantity of phosphoric acid was added to saliva and the liquid neutralised with lime water, which caused a copious precipitate of calcium phosphate. This precipitate carried down with it a large proportion of the proteins of the saliva, together with the diastase or ptyalin. The precipitate was then collected by filtration, and extracted with a volume of water equal to that of the saliva originally used. The diastase is slightly more soluble than the proteins, and accordingly passes into solution first. Cohnheim repeated this process several times, and finally precipitated the last extract by the addition of alcohol. This precipitate was collected, washed with alcohol, and dried over sulphuric acid, appearing then as an amorphous white powder, freely soluble in water. The author considered it to be free from proteins as its solution did not yield the usual reactions characteristic of these bodies.<sup>4</sup>

I have not yet had the opportunity to compare the nitrogen content of the purified trypsin with that of the trypsin before purification by the method I am about to describe. This would have an important bearing on the constitution of the enzyme.

On referring to Robertson's book on the proteins,<sup>5</sup> I find that, speaking of his own work two years earlier, he says, in the course of a discussion on the compounds of proteins with dyes, "I have found that in faintly alkaline solution trypsin or some constituent of Grüber's trypsin (nach Spalteholz) forms an insoluble compound with safranin"; from this it appears that Robertson himself was doubtful whether the precipitate was a compound of trypsin and safranin.

The method of purification of the enzyme which I adopted is a very simple one, and so far as I know has not been previously described; it depends on the fact that cellulose absorbs the impure compound of proteins and enzymes, and that after drying, the colloidal protein portion adheres firmly to the paper, whereas the enzyme is very easily soluble. The method is as follows:—

Ordinary circles of Swedish filter paper (J. H. Munktel), 12½ cm. diam., area 122 sq. cm., ash 0.00095 grm., averaging in weight 0.87 grm. air-dry, were soaked in the impure enzyme solution, and dried quickly in a current of hot air. The average increase in weight was 0.054 grm., i.e., they contain about 6.0% of added matter, or 0.41 mgrm. per sq. cm. When such paper is placed in water, the enzyme dissolves quickly to a perfectly clear solution in a few minutes, but the colloidal matter with which it is associated adheres firmly to the paper; the solution is filtered after 15–20 minutes, by which time the whole of the enzyme matter is dissolved. If left for a longer time, proteins begin to dissolve, and the enzyme strength decreases.

When a solution of safranin is added to the enzyme solution prepared in this way, no precipitate is produced, and the solution passing through the filter is as active as the original solution, due account being taken of the dilution. The following experimental results were obtained:—1.8366 grm.

of the paper impregnated with enzymes was stirred in 183 c.c. of distilled water for 15 minutes, and then filtered; 0.6 c.c. of the filtrate was found to liquefy the standard gelatin tube under the same conditions as in the test previously described (this J., 1912, 1105), corresponding to 6 mgrms. of the paper. The 6 mgrms. of paper contained 0.34 mgrm. of original extract.

A test of the original extract showed 0.35 mgrm. of matter capable of liquefying the gelatin, but the soluble matter in the 6 mgrms. of paper was found by evaporation of the filtrates to be 0.23 mgrm., so that the enzyme has been purified to this extent; i.e. 35% of non-enzyme matter has been removed. For comparison it may be useful to give results for Grüber's pancreatin, which I have always used as a standard of comparison: it is labelled "Pankreatin Dr. G. Grüber & Co., Leipzig"; 0.1 grm. dissolved in about 30 c.c. of alkaline water digests 8–10 grms. of moist blood fibrin; 0.52 mgrm. of the original solution liquefied, and 0.56 mgrm. filtered through paper liquefied, i.e. my purified preparation is 2½ times as strong as Grüber's trypsin. It may be noted that about 20% of the Grüber trypsin was insoluble in water.

I do not suppose for a moment that the preparation I have made is a pure enzyme, and I have not been able to ascertain how far the purification can be carried by the above method, but I have reason to believe that the purer the enzyme the less colloidal is its character, and that it will pass through a membrane. Professor Kipping has kindly examined a solution of the purified enzymes with the polariscope in order to ascertain if it is optically active. The solution contained 0.1184 grm. of solids per 100 c.c., which was a strong solution from the point of view of enzyme activity, being about three times as strong as those I have usually employed. No rotation of the plane of polarisation could be detected in a 200 mm. tube. The following are the minimum quantities of material required to liquefy the gelatin in the test described:—Grüber's pancreatin (unfiltered), 0.56 mgrm.; do. (filtered), 0.52; safranin precipitate from pancreas extract, 0.30; pancreas extract on paper, 0.34; purified enzyme, 0.23 mgrm.

From the above facts I draw the conclusion that the enzyme itself is extremely soluble, whereas the protein matter with which it is associated is difficultly soluble, and this difference in solubility may be used to purify the enzyme.

#### DISCUSSION.

The CHAIRMAN said that most people who had attempted to purify enzymes had come to the conclusion that the more they were "purified" the less pure they became. That, however, did not seem to be the experience in this particular case.

Professor F. S. KIPPING said that he thought the discovery of Mr. Wood might be one of the most far-reaching importance. The difficulty of purifying enzymes was one with which workers were well acquainted, but it seemed probable that the new method would enable chemists to find out a great deal more about enzymes than they knew at present. It was remarkable that although the enzyme was so soluble it was completely precipitated and yet when the product was merely washed with water the enzyme was almost completely extracted.

Dr. R. M. CAVEN asked if the enzyme was precipitated along with protein matter, and if so was it a case of adsorption?

Mr. J. M. WILKIE, referring to the point raised by Prof. Kipping, asked if it was not likely that the phenomenon referred to was not wholly colloidal. It seemed not impossible that on drying, the protein was coagulated and so its solution rate suffi-

<sup>3</sup> Virchow's Archiv, 1863, 28, 241. J. Reynolds Green, *The Soluble Ferments* (Camb. Univ. Press, 1899), p. 45.

<sup>4</sup> Since writing this paper the author has attempted to apply Cohnheim's method to trypsin, but has been unsuccessful.

<sup>5</sup> *The Proteins* (Univ. of California publications in Physiology), Oct., 1909.

clently diminished to enable a partial separation to be effected.

Mr. Wood, in reply, explained that the safranin precipitate was practically insoluble; when the precipitate was wet he considered that the enzyme adhered firmly to it, but when it was dry the enzyme dissolved rapidly. Referring to Dr. Caven's and Mr. Wickle's remarks, he said that the phenomenon was undoubtedly one of colloidal adsorption.

Meeting held at University College on  
November 20th, 1918.

DR. R. M. CAVEN IN THE CHAIR.

## THE EFFECT OF HEATING OPIUM ON ITS MORPHINE CONTENT.

BY HAROLD E. ANNETT, B.SC.(LOND.), F.I.C., AND  
HARDAVAL SINGH, B.SC.

(Agricultural College, Cawnpore, India.)

We have observed that if opium is heated in a water oven for some days the morphine content as determined by the B.P. process appears to be much lower than if the fresh sample is analysed in the moist condition and the moisture then allowed for.

Evan<sup>1</sup> refers to the loss of morphine which takes place on heating moist opium. Carles<sup>2</sup> has shown that opium extracts decompose on concentration at 100° C. Debourdeaux<sup>3</sup> states that the amount of morphine in opium decreases with age, and that this decrease appears to be due to a chemical change. If this is so then one could expect a more rapid change to take place on heating. Lesure<sup>4</sup> finds that morphine hydrochloride solutions undergo a change when heated, oxymorphine being formed but no apomorphine, and that the change is favoured by the presence of a minute trace of alkali. Feinberg<sup>5</sup> agrees that no apomorphine is formed during the heating or storage of solutions of morphine or of its hydrochloride.

### Experimental.

The morphine in a sample of well mixed moist opium was estimated by the B.P. 1914 and 1898 methods respectively, both on the fresh sample and after heating for six days in the water oven and then making up to the original weight with distilled water. The samples lost weight continually when heated. The results were as follows: 8 gm. sample, unheated 7.18%, after 6 days' heating 6.31% of morphine by B.P. 1914 method. 14 gm. sample, 7.15 and 6.36% respectively by B.P. 1898 method.

Three other samples of opium were then taken and a portion of each was spread on a glass plate and dried in the water oven. After one day the opium was scraped off the glass plate, ground to a powder, and sifted, and the drying continued for two days more. Another portion of each of the three samples was dried *in vacuo* over concentrated sulphuric acid at the air temperature. The samples were powdered as soon as they were dry enough and the drying continued. In all this took six days. The samples dried *in vacuo* were extremely hygroscopic. The morphine was then estimated in 8 grms. of each of the dried samples by the B.P. 1914 process. The following results were obtained:

Sample No.	Morphine content in dried opium	
	Sample dried in water oven	Sample dried <i>in vacuo</i>
1	6.86	9.68
2	7.52	9.57
3	8.20	10.36

Another sample of moist opium was analysed by the method of the B.P. 1911 in triplicate, both before and after heating in a water oven for 48 hours. After heating the sample and prior to the analysis the loss in weight on heating was made up by the addition of distilled water. The following results were obtained:

Sample No.	Percentage of anhydrous morphine found	
	Moist sample, unheated	Sample after heating for 48 hours
1	7.25	7.45
2	7.14	7.43
3	7.02	7.49

Here it would seem that heating for 48 hours has slightly increased the morphine content. Certainly it has not decreased it.

A series of experiments was next carried out to determine the effect on the morphine content of heating the opium for various lengths of time. The method of analysis used was that of the B.P. 1914. A mass of moist opium was thoroughly mixed and 26 portions of 8 grms. each were rapidly weighed off, each portion being spread in a thin layer on a glass plate. Two portions were analysed at once and the remaining 24 portions heated in the water oven. After one day two more samples were removed, the weight of each made up to 8 grms. by addition of distilled water, and analysed. Daily two more samples were similarly removed for analysis. The results were as follows:

No. of hours heated	Anhydrous morphine in moist opium		Loss in weight at end of heating	
	(a)	(b)	(a)	(b)
—	6.78	6.87	—	—
24	6.43	6.57	1.773	1.748
48	6.97	6.91	1.829	1.803
72	6.66	6.97	1.856	1.851
96	6.57	6.62	1.888	1.881
120	6.12	6.08	1.870	1.876
144	5.60	6.14	1.911	1.894
168	5.66	5.62	1.951	1.981
192	4.92	4.79	1.951	1.960
216	4.79	4.50	1.954	1.969
240	4.19	4.24	1.954	1.983
264	4.26	4.34	1.972	1.985
288	4.63	5.00	1.955	1.934

The heating has therefore had a very marked effect on the opium. This effect, however, did not become apparent until after four days' heating, from which time up to 264 hours there seems to have been a steady increase in the loss of morphine; during the subsequent 24 hours the morphine content appears to have increased. The figures shewing the loss of weight on heating after the various intervals of time indicate that after the seventh day practically no further loss took place. This fact is of interest in that it indicates that the loss of morphine does not coincide with a loss of volatile constituents from the opium.

A series of experiments was designed to compare the results obtained when the opium sample is

<sup>1</sup> Analytical Notes, 1914, 8, 46.

<sup>2</sup> J. Pharm. Chim., 1913, 8, 250.

<sup>3</sup> J. Pharm. Chim., 1912, 6, 491.

<sup>4</sup> J. Pharm. Chim., 1909, 30, 337.

<sup>5</sup> Z. physiol.-Chem., 1913, 84, 363; this J., 1913, 32.



dried (a) *in vacuo* and (b) in a water oven with those obtained when (c) the sample is analysed in the fresh moist condition. For this four different lots of opium were taken, the method of B.P. 1914 being used throughout. After two days the samples in the vacuum desiccator (a) were powdered and returned to the desiccator until a constant weight was registered (7 days); the loss of weight was then made up with distilled water and the morphine estimated. The samples heated in the water oven (b) were powdered after three days and replaced in the water oven for 9 days, after which time the loss in weight was made up with distilled water and morphine estimated. The results were as follows:

Treatment	Anhydrous morphine in samples			
	No. 1	No. 2	No. 3	No. 4
	%	%	%	%
Dried <i>in vacuo</i> ...	8.12	5.66	4.11	3.17
Dried in water oven ...	5.43	3.16	1.63	1.16
Undried ...	7.50	5.36	3.62	2.57

The drying of the samples in the water oven for 12 days has therefore resulted in a much lower recovery of morphine. On the other hand, drying *in vacuo* appears to have slightly increased the amount of morphine.

We have as yet had no opportunity of inquiring into the nature of the change which the morphine undergoes. If, however, samples of opium are dried *in vacuo* or by heating in a water oven, we have observed certain marked differences in the resulting products. Firstly the heated opium is much darkened and the lime extract of heated opium, prepared as in the B.P. process, is much darker than the extract obtained from unheated or from vacuum-dried opium. Moreover the morphine precipitated from heated opiums is always contaminated with much black material. The physical character of the heated opium is also very different from opium which has been dried *in vacuo*. The latter on exposure to moist air absorbs moisture rapidly and the surface of some of the samples might almost be said to be deliquescent. In the case of samples dried in the water oven the absorption of moisture is very slow in comparison.

#### Conclusions.

1. Opium heated in the water oven at 97°–98° C. may suffer a large decrease in morphine content. We have found no trace of this effect during the first five days' heating, and therefore it can hardly affect results obtained by the B.P. method.
2. Opium when heated in the water oven suffers a marked change in its physical properties.

#### DISCUSSION.

Mr. H. DROOP RICHMOND said he thought there was no doubt that the loss of morphine on heating was due to oxidation: morphine could be converted completely to pseudo-morphine by peroxide, but he did not think that was a complete explanation of the non-hygroscopic property of the opium when dried at 100° C. as against the hygroscopic character when dried in vacuum. Five days' heating of opium was comparatively ineffective with regard to the morphine content. That was rather a long time for drying powdered opium, and in very much less than that time it lost its hygroscopic nature. That loss and the fall in morphine content were not due to the same cause. Some time ago they had observed in Messrs. Boot's laboratory similar phenomena to those described by the authors, and, as would be seen from Miss Iles' remarks, their results broadly confirmed those of the authors.

Miss L. E. ILES said that during the past few months she had made numerous estimations of the morphine content of Persian opium and had found that moderately prolonged heating (120 hours) in the water oven had not appreciably diminished the morphine content, thus confirming the authors' experience with Indian opium. After that period of heating the morphine content was found to remain practically constant, and in one case even showed a slight increase. The temperature of the water oven used was about 90° C., compared with the authors' 97°–98°. The following results were obtained, the alkaloid in each case being titrated:—

	After heating in water oven for	and in air oven (100° C.– 115° C.) for	Morphine %
SAMPLE A.	204 hrs.	12 hrs.	8.69
9.16% morphine,	241 "	20 "	6.52
8.36% moisture	276 "	39.5 "	5.51
	324 "	65.5 "	4.92
SAMPLE B.	120 "	12 "	10.2
10.62% morphine,	196 "	24 "	9.97
8.9% moisture	221 "	35.5 "	8.38
	256 "	58 "	6.70
	304 "	84 "	5.69

\* 3 hours at 120° C.

For three hours of the second period of heating of A and the third period of heating of B, the temperature of the air oven was maintained at 120° C. Both samples were found to have charred slightly, showing in the case of A, 6.52% morphine and in the case of B 8.38%. From then onwards the temperature of the air oven was kept below 115° C. After the last period of heating of B, a gravimetric estimation was made according to the B.P. 1888 method, showing 6.2%. A portion of this alkaloid was titrated indicating 5.69% morphine. It should be pointed out that the alkaloid became darker in colour, and required much larger quantities of morphinated water to produce colourless washings, and was less readily soluble in acid water.

The CHAIRMAN said that it would be interesting to know if the loss of morphine began when the opium was drying, or whether the drying was complete in 4 or 5 days and the loss commenced then.

Mr. RICHMOND suggested that during the drying there was a good deal of water vapour present and oxidation was therefore slow.

Mr. J. M. WILKIE said that Indian opium had always suffered in comparison with the Smyrna variety because of its low morphine content. The Persian variety was intermediate between Indian and Smyrna. The paper appeared to have an important bearing upon the original morphine content of opium, and raised the question whether Indian opium of a higher morphine content could be produced by a modified method of curing. In the B.P. (volumetric) method of estimating morphine, the bulk of the resinous constituents was rejected owing to the formation of insoluble calcium compounds, and only bases strong enough to affect the selected indicator were taken into account: it was possible that if the alkaloids were weighed instead of titrated one might get some valuable information. Most of the moisture in opium was probably driven off in about 6 hours and probably all of it in 10 or 12 hours. It was well known that morphine was very readily affected by small amounts of alkali, and it was possible that the much greater susceptibility of Indian opium to heat was due to some such cause. Heated opium presented certain points of distinction in the morphine determination. Instead of a homo-

geneous suspension in the lime stage, dark coloured resinous masses separated which might occlude alkaloids. The resins might have a distinct acid effect on the morphine molecule.

Mr. E. H. BUTLER said that in his experience the loss in morphine value on heating Smyrna opium was not so great as with the Indian opium used by the authors. If moist heat reduced the morphine value to this extent, there must be an enormous percentage of morphine lost in making extracts by water heating.

## Communication.

### VALUATION OF FERTILISERS.

BY J. MAN MURRAY.

The unit hitherto employed in the valuation of fertilisers was the ratio of price to percentage, and was used to assess the prices of other grades of the same material by multiplying it by the percentages of fertilising ingredients in them. This method of calculation, it will be seen, is merely simple proportion and may be expressed as:

$x' = \frac{x}{p} \times p'$ , where  $x$  is the market price of the grade that contains  $p$  per cent. and  $x'$  is the estimated price of that which contains  $p'$  per cent;  $x/p$  is the so-called unit.

The system was simple and well understood, but when the necessity for controlled prices arose the narrow limits within which it is applicable appear to have been recognised for the first time. At all events it broke down utterly and had to be abandoned. It was at once perceived that manufacturers could not be expected to sell 20% superphosphate at half the price of the 40% grade, and that farmers should not be expected to pay twice as much for the latter as for the former.

In this system it was assumed (1) that the value (to the farmer) of a fertiliser is proportional to the percentage of fertilising ingredients in it, and (2) that the price varies as the value. Neither of these assumptions has any foundation in fact.

If a quantity of basic slag containing 40% of phosphate be mixed with an equal weight of sand, or other non-reactive substance, the mixture will contain 20% of phosphate and, when applied to the land, will produce precisely the same effect as half its weight of the original slag. It does not follow, however, that if the original slag were valued at £4 per ton the mixture should be valued at £2 per ton. Assuming that the expenses of application amounted to 25s. per ton (see note 1) in both cases, the value (to the farmer) of the mixture would be only 27s. 6d. per ton because the expenses of application are twice as much for the same amount of phosphate (see note 2). If the difference in percentage were greater than in the example above, the difference in value would be larger. The "value" per ton of the 1% grade, i.e. the unit, would not be 2s.; it would be a minus quantity. In short, the cash "value" of fertilisers is not proportional to the percentage of fertilising ingredients; it depends upon the expenses of application (freight, cartage, etc.) and varies therefore with the circumstances in particular cases.

Obviously no reliable inference regarding the efficiency of fertilisers can be drawn from the prices of the same. It is equally impossible to infer market prices from the efficiency as estimated by chemical analysis or otherwise. Unquestionably the price that will be paid (in free markets) for any fertiliser increases with the effective value, which in turn depends upon the percentage of fertilising ingredients, but not necessarily, or usually, in direct proportion. Demand is only one of the conditions

which govern market prices. The fatal defect of the system of unit values was that it took no account of the supply price, which is governed by the cost of production.

Any scheme of valuation of which the object is to assess the prices of different grades of the same material must be based on the fact that the price, in free markets, comprises two elements, viz. (1) the cost of production, which may or may not be constant, and (2) another quantity which varies with the demand and, therefore, with the quality (percentage of fertilising ingredients). In general this is expressed by  $x = k + np$  where  $x$  is the price,  $k$  the cost of production,  $p$  the percentage of fertilising ingredient and  $n$  is a coefficient which depends upon  $x$  and  $k$ . Since  $np = x - k$  is the difference between price and cost it represents profit.

The schedule prices under the "Fertiliser Prices Order, 1918," reflect this view to some extent and may be used to illustrate it.

In the case of superphosphate, for instance, the schedule price is 92s. 6d. per ton for 15% grade and rises by 2s. 6d. per ton for each additional 1% up to 30%; beyond that point the price rises at the rate of 4s. per ton up to 44%, the highest grade affected by the Order. Within the limits specified these variations in price are concisely expressed by:—

$$(1) x = 92.5 + 2.5(p - 15) = 55 + 2.5p$$

$$(2) x' = 130.0 + 4.0(p - 30) = 10 + 4.0p$$

where  $x$  is the price, in shillings, and  $p$  is the percentage.

When  $p = 30$ ,  $4p + 10 = 2.5p + 55$  and the price of that particular grade may be calculated by either formula; it is 130s. per ton.

The schedule prices include cost of carriage by rail, estimated at an average of 12s. 6d. per ton. This amount has been added to the price and for present purposes should be treated as part of the cost of production. If it is deducted we must put  $x - 12s. 6d. = k - 12s. 6d. + np$ . The above formulae for the prices of superphosphate, if altered in this way, become:—

$$(1) x = 2.5p + 42.5 \text{ and } (2) x' = 4p - 2.5.$$

Similarly, the schedule prices (including carriage) of basic slag may be expressed by:—

$$(1) x = p + 48 \text{ and } (2) x' = 1.5p + 37.$$

For sulphate of ammonia the formula is:  $x = 13p - 13.5$ .

It will be seen that in each case the price is made up of two quantities corresponding to  $k$  and  $np$  respectively. It must not be assumed, however, that the values found for  $k$  in these equations necessarily represent the actual cost of production. The method by which they were deduced and the fact that two widely different values—one of them a minus quantity—have been found for  $k$  in superphosphate, preclude any such inference. This does not imply that the prices themselves are not fair and reasonable. It means, merely, that the "Order" is not a scheme of valuation and that no attempt has been made to resolve the prices accurately into their elements. The values assigned to  $n$  in the schedule prices are purely arbitrary and the difference ( $x - np$ ) is not therefore the true value of  $k$ . In formulating a scheme of valuation the procedure must be reversed; the actual cost of production must first be ascertained and the value of  $n$  deduced by  $n = (x - k)/p$ .

The cost of production can only be ascertained from data supplied by the manufacturer. In the case of superphosphates it includes (1) rent or royalty payable to the owner of the raw phosphates, (2) cost of excavating and shipping the same to this country, (3) cost of treatment (grinding, acid, labour, plant, establishment charges, etc.), and (4) cost of packing and distribution if payable by the producer. Items 1 and 2 do not, as a rule,



vary much within a limited period, *e.g.* a single season. The difference in cost of treatment for grades below 30% is probably insignificant and *k* should therefore be constant. The cost of treatment for the higher grades rises progressively and *k* must be increased accordingly, whereas *n* should remain the same throughout.

In the formula deduced from the schedule prices of superphosphate, *k* is constant for all grades from 15 to 30%. Assuming for the moment that this value for *k* (55s. per ton including carriage) is the actual cost of production and that 25*p* accurately represents the effect of demand on the prices, the value of *k* for the several grades mentioned below would be as follows (see note 3):—

grade	20%	25%	30%	35%	40%	45%
<i>k</i>	55	55	55	62·5	70	77·5

It will be seen, therefore, that for 30% grade  $x=25p+55$  and for 40% grade  $x=25p+70$ .

The cost of production of basic slag is not so easily determined. It may be debited with part of the cost of the ore, lime, fuel, etc.; or it may be regarded strictly as a by-product chargeable only with the cost of grinding and packing, the balance of the price being treated as profit. For the purposes of a scheme of valuation the latter would appear to be the more correct as well as the simpler view. Adoption of it would not, of course, preclude the producer from using part of this profit to reduce the price of or to increase the apparent profit on the other product. The effect of adopting the suggestion would be merely to lower the value of *k* and thus, automatically, to raise that of *n*. If *k* were reduced to zero *n* would become the obsolete unit value.

As in the case of superphosphates the actual cost of production of the lower grades (up to about 22%) of basic slag is probably nearly constant; the cost of production of the superior grades is higher but it does not increase progressively as it appears to do, or may be made to appear, from the schedule prices. Thus, if  $x=p+48$  represents the proper subdivision of the price of the lower grades, the value of *k* for the several grades mentioned below would be as follows (see note 4):—

grade	12%	18%	22%	28%	32%	38%	42%
<i>k</i>	48	48	48	51	53	56	58

It will be seen therefore that for the 22% grade  $x=p+48$  and for the 42% grade  $x=p+58$ .

The price of sulphate of ammonia previously mentioned is subject, under the Order, to an addition of 10s. per ton every three months from the end of August to the end of May. In effect this is due to the greater demand for the product as the season for its use approaches. The increase should therefore go to augment *n*, not *k*. For trade purposes it is probably not necessary or advisable to embody this addition in a formula, but in order to make its incidence clear we may put  $(x+10)=(13p+10)-13·5$ .

Both buyer and seller will probably profess indifference as to whether a given alteration in price be called *k* or *n*, and truly, in any particular case, or in a schedule of particular cases, it matters not at all. But in order to formulate a scheme of valuation, the object of which is fairly to assess the prices of different grades of fertilisers, it is essential to know the causes of variation of the prices and the manner in which they operate. It is, in fact, precisely because it was not based on this principle that the system of unit values failed of its purpose. The application of the principle to the more complex cases of guanos and mixed fertilisers presents no obvious difficulty, but the work can only be successfully accomplished by or in co-operation with those who are thoroughly conversant with the trades concerned.

As every farmer is aware, the criterion of cheapness is not the price of a fertiliser but the ratio of price to percentage, *i.e.* the old-fashioned "unit."

In the notation used in this article the unit is  $x/p$  [ $=k/p+n$ ] and it is clear that if *k* and *n* are constant the unit will diminish as *p* increases. The highest grades will therefore be the cheapest though they are the most costly. The same is true even when *k* increases progressively unless it increases faster than *p*. The unit values ( $x/p$ ) of several grades of superphosphate, basic slag, and sulphate of ammonia, calculated from the schedule prices, including carriage, are given below:—

Grade	" Unit values "		
	Superphosphate	Basic slag	Sulphate of ammonia
	<i>s.</i> <i>d.</i>	<i>s.</i> <i>d.</i>	<i>s.</i> <i>d.</i>
12%	—	5 0	11 10
15%	6 2	4 2	12 1
20%	5 3	3 5	12 4
25%	4 8	3 0	12 5
30%	4 4	2 9	—
35%	4 3	2 7	—
40%	4 3	2 5	—
45%	4 2	—	—

These results are even more striking when plotted in graphic form.

*Note 1.*—Under the head of expenses of application are included all expenses other than the price of the goods, *viz.* freight charges, cartage, and spreading, here estimated at 12s. 6*d.*, 6s. 6*d.*, and 6s. per ton respectively, or 25s. per ton in all. Freight and cartage charges vary widely in different cases; 12s. 6*d.* is the average assumed for the former in the "Fertiliser Prices Order, 1918"; 6s. 6*d.* for cartage is the estimate of a farmer located about three miles from a railway station, and 6s. for spreading is also a farmer's estimate. It was necessary to take some definite figures for purposes of illustration, but the argument does not depend upon the accuracy of those selected or upon their applicability to particular cases.

*Note 2.*—If the price of the slag, *f.o.r.*, is 80s. per ton and the expenses of application 25s., the total cost is 105s. per ton, or 52s. 6*d.* for half a ton, which contains the same amount of phosphate as one ton of the mixture. But as the expenses of application are the same (25s.), in order to balance the account, the farmer could only afford to pay [52s. 6*d.*—25s.=] 27s. 6*d.* per ton for the goods. In other words the mixture would be worth, to him, only 27s. 6*d.* per ton because he could obtain the same fertilising effect at a total cost of 52s. 6*d.* by purchasing half a ton of the original slag.

*Note 3.*—If  $x=25p+55$  accurately represents the proper subdivision of the price for the lower grades of superphosphate, the formula  $x=4p+10$ , which is applicable to the higher grades, should be altered as follows:—

$$\begin{aligned} 4p+10 &= (25p+15p) + (55-45) \\ &= (25p+55) + (15p-45) \\ &= 25p+55+15(p-30) \end{aligned}$$

$$\text{when } p=40, x=25p+55+15(40-30)=25p+70.$$

*Note 4.*—If  $x=p+48$  accurately represents the proper subdivision of the price for the lower grades of basic slag, the formula  $x=15p+37$ , which is applicable to the higher grades, should be altered as follows:—

$$\begin{aligned} 15p+37 &= (p+05p) + (48-11) \\ &= p+48+05p-11 \\ &= p+48+\frac{1}{2}(p-22) \end{aligned}$$

$$\text{when } p=42, x=p+48+\frac{1}{2}(42-22)=p+58.$$

University College,  
Reading.

Received October 23rd, 1918.

## London Section.

Meeting held at Burlington House on December 2, 1918.

DR. CHARLES A. KEANE IN THE CHAIR.

### COMMERCIAL "CONCENTRATED AMMONIA-LIQUOR" AND ITS IMPURITIES.

BY H. O. COLMAN, D.SC., PH.D., AND E. W. YEOMAN, B.SC.,  
A.R.S.M., A.R.C.SO.

Commercial "concentrated ammonia" is manufactured from the crude gas-works or coke-oven ammoniacal liquor in two qualities. The first of these qualities is obtained by distilling the liquor with addition of lime in the usual way, and condensing the distillate, and contains most of the hydrogen sulphide and carbonic acid and of the other volatile impurities of the original liquor, and is therefore largely a solution of ammonium sulphides and carbonates. The ammonia strength of this quality cannot be made more than about 16-17%, owing to the large amount of carbonate present, which at higher strengths crystallises out, causing stoppages in the manufacturing plant, and deposits in the vessels in which the liquor is stored.

The second quality is manufactured on a much larger scale, and consists for the most part of free caustic ammonia, the carbonic acid being more or less completely removed, as well as most of the other volatile impurities, but it still contains material percentages of hydrogen sulphide, the amount of which is usually required not to exceed 0.5%. The ammonia strength of this quality is about 25%.

This type of concentrated ammonia has for a long time been manufactured in considerable quantity as an "intermediate" product, being employed in the ammonia-soda manufacture, and in the manufacture of ammonium salts other than the sulphate, for which purposes a limited amount of hydrogen sulphide is not a serious objection, and is more than compensated for by the lower price of the product. The output of this type of concentrated ammonia has been very greatly increased owing to the requirements for ammonium salts for war purposes, but it has been found that in the manufacture of certain ammonium salts from it, especially the nitrate, difficulties may occur from the minor impurities present, and in particular from cyanogen derivatives. The present paper gives some account of the extent and nature of these minor impurities, and the variations found in samples from different plants.

The starting point for the manufacture is in most cases the crude ammoniacal liquor obtained by the carbonisation of coal in gas-works or coke-ovens, which usually contains an amount of ammonia varying between 0.9 and 2.2%; about four-fifths of this ammonia is present as sulphides and carbonates and is all evolved by simply boiling the liquor, as is also any small amount of ammonium cyanide which may be present. The remaining fifth of the ammonia is present in the form of salts with non-volatile acids, and chiefly in the form of thiocyanate, thiosulphate, sulphate, and chloride, and the ammonia thus combined is only evolved on distillation when sufficient fixed alkali (usually lime) is added to combine with the whole of the fixed acid radicals present. In addition the crude liquor always contains appreciable amounts of phenols and pyridine bases.

In general outline, and without going into manufacturing detail, the method of manufacture of the

25% ammonia is as follows:—The crude liquor, freed from all tarry matter, is first passed through a preheater wherein it is raised approximately to its boiling point, at which temperature the ammonium sulphides and carbonates undergo dissociation, and the greater part of the hydrogen sulphide and carbon dioxide is driven off, but these are accompanied by only a small proportion of ammonia; under the above conditions, this remains for the most part dissolved in the solution. The ammonia carried away with the effluent gases is recovered by washing the water or by other suitable means.

The hot liquor from the preheater, thus largely freed from hydrogen sulphide and carbonic acid, then passes to the ammonia still of the usual vertical type, in which the liquor travels downward from tray to tray, meeting as it falls an ascending current of steam which bubbles through it in each tray, lime being added (in the lower portion of the still in most cases) in sufficient quantity to liberate the whole of the "fixed" ammonia. The ammonia, steam, and volatile impurities pass from the top of the still to a reflux condenser, where a large proportion of the steam condenses with some ammonia, the condensate returning to the still; the bulk of the ammonia passes on to a further condenser where it is completely cooled and dissolved in water. Naturally considerable variations exist in the character of the plant used and in the method of working, but these matters are beyond the scope of this paper.

An examination has been made by the authors of a large number of samples of concentrated ammonia from different plants in all parts of the country, with the object, more especially, of ascertaining the amount and nature of the cyanogen compounds present, and some examination has also been made of the other impurities. Apart from the cyanogen compounds, the impurities found in appreciable quantity consist of the following:—

(1) *Hydrogen sulphide*. It is usually specified that the amount of this impurity shall not exceed 0.5%, and in the majority of samples examined, the quantity varies from this figure down to *nil*. A minority, however, contain much higher amounts, rising in one instance to 3.1%, evidently owing to insufficient elimination of the hydrogen sulphide in the preheating process.

(2) *Carbonic acid*. In many cases this is entirely absent, and is usually only present in small quantities, but in some instances up to 11% of  $\text{CO}_2$  was found. Such high amounts are always accompanied by excessive amounts of hydrogen sulphide, and are due to inadequate treatment of the crude liquor in the preheater. For most purposes the presence of carbonic acid in moderate amount is not in itself objectionable, but when it is present in considerable quantity the amount of other impurities is always high.

(3) *Phenols*. The samples always contain phenols, derived from those present in the crude liquor, which are partly volatilised with the steam in distillation. No systematic determination of the amount has been made, but in a few samples tested the quantity varied from 0.11 to 0.37 grm. per 100 c.c., averaging 0.23 grm.

(4) *Pyridine*. Pyridine bases are always present, the amount found in a few samples varying from 0.21 to 0.32 grm. per 100 c.c., averaging 0.26 grm.

(5) *Ammonium thiosulphate*. The freshly prepared liquor is probably free from this impurity, which is formed secondarily by oxidation of the ammonium sulphides. The analytical methods tend to give results which are slightly too high, but the figures obtained from about fifty samples of different origin indicate that the amount varies from 0.08 to 0.35 grm.  $(\text{NH}_4)_2\text{S}_2\text{O}_3$  per 100 c.c., no



higher figure than the last having been obtained. These samples were, however, all tested within a few days of their manufacture, and the amount probably increases steadily when the liquor is stored with access of air, owing to the further oxidation of ammonium sulphides. At the same time, we are not aware that any complaint has been made of any prejudicial effect due to the presence of considerable quantities of ammonium thiosulphate in the liquor.

#### Cyanogen Compounds.

The especial point on which information was desired, was the nature and amount of the cyanogen compounds present in samples of concentrated ammonia from different plants, these having been found of considerable disadvantage in the manufacture of ammonium nitrate, for which purpose it was desired that the total amount of cyanogen, calculated as ammonium thiocyanate, should not exceed 0.01 grm. per 100 c.c.

In the liquor as first produced, the only cyanogen compound present appears to be ammonium cyanide, formed by the action of the hydrocyanic acid almost always given off with the ammonia from the crude liquor during distillation. In presence of ammonium sulphide, however, this ammonium cyanide rapidly undergoes further changes; as soon as the oxygen of the air gets access to the liquor, oxidation of the ammonium sulphides takes place, with formation especially of ammonium thiosulphate and polysulphides, the latter at once reacting with ammonium cyanide forming the thiocyanate. Further, the ammonium cyanide solution produced acts on any iron (especially steel or wrought iron) with which it may come in contact, whether in the manufacturing plant or in the containing vessels, forming ammonium ferrocyanide. Hence the liquor samples may contain ammonium cyanide, ferrocyanide, and thiocyanate, though the tendency is for the cyanide to disappear rapidly owing to conversion into the other two forms, and in many cases ferrocyanide is absent, the only recognisable cyanogen compound being the thiocyanate.

There is, however, a further source of thiocyanate other than the hydrocyanic acid evolved with the ammonia in the distillation, for it was found that, on standing, the total cyanogen content in all forms of the liquor undergoes a material increase. This is shown in the following series of tests made on the same sample of liquor after the lapse of different periods, the fresh sample being almost free from cyanogen derivatives:—

		Total HCN calculated as NH <sub>4</sub> CNS	
Freshly prepared product	..	0.0054	grm. per 100 c.c.
After standing 3 days	..	0.0061	" "
" 7 "	..	0.0167	" "
" 11 "	..	0.0175	" "
" 17 "	..	0.0228	" "
" 24 "	..	0.0304	" "
" 31 "	..	0.0304	" "

The cause of this increase was found to be the small quantity of carbon bisulphide usually present in the distillation gases from the still, and formed from the ammonium thiocarbonate always present in the crude liquor, this being absorbed in the concentrated liquor with re-formation of ammonium thiocarbonate. The latter, on standing, undergoes slow change, forming, among other products, ammonium thiocyanate. This change is shown by the following series of tests, in which a solution was made up of 100 parts of pure 25% ammonia, 0.5% of hydrogen sulphide, and 0.1% of carbon bisulphide. If the whole of this carbon bisulphide were converted into ammonium thiocyanate, the amount of the latter in solution would amount to 0.1%. Shortly after the preparation of the solution, it was found that about one-fifth of the carbon bisulphide had already been

converted into thiocyanate, and on standing the amount increased, until after about 20 days the thiocyanate remained constant, one-third of the carbon bisulphide having then been converted into thiocyanate.

		NH <sub>4</sub> CNS	
Fresh solution	..	0.0198	grm. per 100 c.c.
After standing 3 days	..	0.0228	" "
" 6 "	..	0.0258	" "
" 10 "	..	0.0304	" "
" 13 "	..	0.0314	" "
" 20 "	..	0.0334	" "
" 27 "	..	0.0334	" "
" 40 "	..	0.0334	" "

A selection of the results obtained in the determination of the cyanogen compounds in a large number of samples from different plants is given in the accompanying tables.

TABLE I.

#### Concentrated Ammoniacal Liquor from Gas-Works.

Sample no.	H <sub>2</sub> S	Hydrocyanic acid				Total HCN calc. as NH <sub>4</sub> CNS
		present as NH <sub>4</sub> CN	present as (NH <sub>4</sub> ) <sub>2</sub> FeCy <sub>6</sub>	present as NH <sub>4</sub> CNS	Total HCN	
1	0.06	nil	nil	nil	nil	nil
2	0.36	nil	nil	nil	nil	nil
3	0.02	nil	nil	trace	trace	trace
4	nil	nil	nil	0.0014	0.0014	0.0038
5	0.03	nil	trace	0.0021	0.0021	0.0061
6	0.89	0.0032	nil	0.0027	0.0059	0.0166
7	0.39	nil	nil	0.0121	0.0121	0.0342
8	0.94	nil	trace	0.0140	0.0140	0.0395
9	0.62	nil	0.0032	0.0170	0.0202	0.0568
10	1.22	nil	0.0054	0.0267	0.0321	0.0903
Average	0.45	0.0003	0.0008	0.0076	0.0087	0.0245

TABLE II.

#### Concentrated Ammoniacal Liquor from Coke-Ovens.

Sample no.	H <sub>2</sub> S	Hydrocyanic acid				Total HCN calc. as NH <sub>4</sub> CNS
		present as NH <sub>4</sub> CN	present as (NH <sub>4</sub> ) <sub>2</sub> FeCy <sub>6</sub>	present as NH <sub>4</sub> CNS	Total HCN	
11	0.42	nil	nil	0.0005	0.0005	0.0015
12	0.09	nil	nil	0.0032	0.0032	0.0090
13	0.17	0.0016	nil	0.0046	0.0062	0.0175
14	0.22	0.0032	0.0027	0.0084	0.0143	0.0402
15	0.36	nil	0.0146	0.0219	0.0365	0.1028
16	0.55	nil	0.0265	0.0159	0.0424	0.1193
17	1.45	0.0016	0.0415	0.0162	0.0594	0.1672
18	0.71	nil	0.0286	0.0316	0.0632	0.1780
19	2.69	nil	0.0184	0.0578	0.0762	0.2150
20	0.76	0.0054	0.0415	0.0353	0.0822	0.2314
Average	0.71	0.0012	0.0173	0.0198	0.0383	0.1080

All results given in grms. per 100 c.c.

nil = not qualitatively recognisable in 100 c.c.

trace = qualitatively recognisable but not in sufficient quantity to estimate in 100 c.c.

Table I. gives the results for 10 samples from gas-works, and Table II. for 10 samples from coke-ovens. All the other samples tested give results falling generally within the limits of the 20 given in the tables.

It will be seen that in the samples as received the quantity of free ammonium cyanide is either nil, or only a very small amount, owing to its already having undergone complete or almost complete conversion into ferrocyanide and thiocyanate. Ferrocyanide is rarely present in any but small quantity in samples made from gas-works liquor, but is often present in much larger quantity in samples made

from coke-oven liquor, and the total hydrocyanic acid is on the average much higher in samples from coke-oven liquor than in those from gas-works liquor. In very few of the samples was the total hydrocyanic acid so low as the desired amount of not more than the equivalent of 0.01 grm. of  $\text{NH}_4\text{CNS}$  per 100 c.c.

The much higher amount of cyanogen derivatives in the concentrated liquor from coke-ovens probably arises largely from the fact, frequently pointed out in the Alkali Inspector's Reports, that crude coke-oven liquor itself contains much larger amounts of free ammonium cyanide than that from gas-works. This is mainly because, on gas-works, the crude liquor is usually stored for a considerable time before being distilled, and during the interval, by the action of the oxygen of the air, the free cyanide originally present is mostly converted into thiocyanate, whereas in coke-oven plants, the liquor storage capacity is usually small, and the liquor has to be distilled almost as soon as it is produced. But even where the crude liquor distilled contains no free cyanide, some hydrocyanic acid is nearly always formed by the distillation under normal conditions of working, and even if no hydrocyanic acid is produced, there may nevertheless be thiocyanate in the concentrated liquor after storage if any carbon bisulphide vapour is also evolved from the still.

Quite apart from the objections to cyanides in the finished liquor, their presence is very disadvantageous from a manufacturing point of view, as a solution of ammonium cyanide, even when very dilute, has a serious corrosive effect on iron, and especially on wrought iron and steel. Thus for example in one instance where steel tubes were employed in the reflux condenser, the condensate flowing back from the condenser to the still contained 0.00143 grm. of iron per 100 c.c. in the form of ferrocyanide, and though this amount looks small, the amount of iron removed thereby from the tubes was about 4 lb. per day, and these rapidly corroded through and had to be renewed.

In considering the relatively large amount of cyanogen compounds present in some cases, it must be remembered that in many instances the plants in use had to be hurriedly constructed, often with the utilisation of existing sulphate plants as far as practicable, and the samples of which analyses are given were in a number of cases of the product obtained before those in charge of the plant had had much opportunity of gaining experience in working, and with further experience the quantities will doubtless be much reduced. Some of the methods which have been adopted or might be tried to this end may be mentioned, although the information as yet available is not sufficient to say how far they will be generally efficient.

Unquestionably one of the most important points is the proper treatment of the crude liquor in the preheater, to ensure a good removal of the carbonic acid and hydrogen sulphide, as any hydrocyanic acid present in the crude liquor is also largely eliminated there with good working, and this is also probably true of the carbon bisulphide present as thiocarbonate. The changes taking place in the preheater present many points of interest from a chemical, and especially from a physico-chemical standpoint, and have recently been discussed by Parrish (Gas J., 1918, 144, 413).

A further matter which also largely affects the amount of hydrocyanic acid evolved in the still, is the addition of lime in sufficient quantity to ensure that at all times the whole of the fixed ammonium salts are decomposed, for if the lime is at any time insufficient for this purpose, considerable increase takes place in the cyanogen content of the liquor produced.

Quite apart from this point, deficiency of lime is very objectionable, as it results also in waste of

ammonia, which then passes away as fixed ammonium salts in the waste liquor.

A further plan which has been tried is the passing of a stream of air along with the steam through the ammonia still, the oxygen of which effects the retention of the hydrocyanic acid by converting it into thiocyanate, which passes away with the waste liquor. The following tests on a week's run on the same plant, first without any air, and secondly with the introduction of air, show the effect:—

Conc. liquor	Week's run without air gm. per 100 c.c.	Week's run with air gm. per 100 c.c.
$\text{H}_2\text{S}$	0.67	0.16
HCN as $\text{NH}_4\text{CN}$	0.0054	nil
" $(\text{NH}_4)_2\text{FeC}_2\text{O}_4$	0.0011	trace
" $\text{NH}_4\text{CNS}$	0.0230	0.0108
Total HCN	0.0295	0.0108
Total HCN, calculated as $\text{NH}_4\text{CNS}$	0.0830	0.0304

The total quantity of cyanogen was therefore reduced to about one-third, and the amount of hydrogen sulphide simultaneously reduced to about one-quarter.

The introduction of air in this manner increases the volume of uncondensed gases passing away from the final condenser, these carrying an increased quantity of ammonia, and necessitating a rather larger scrubber to recover this ammonia. In this particular plant the existing scrubber was found of quite sufficient capacity for the purpose.

Finally the hot gases passing from the still may be treated between that point and the reflux condenser with some agent which will remove the hydrocyanic acid. A mixture of ferrous sulphate solution and caustic soda has been quite successfully employed for the purpose, removing the hydrocyanic acid as sodium ferrocyanide, the concentrated liquor produced being free from cyanides, but the plan, under present conditions, has the disadvantage of a high cost for chemicals. In some plants a lime-washer is already introduced at this point to absorb hydrogen sulphide and carbon dioxide which have not been removed in the preheater, and the addition to the lime of sulphur as such or in the form of spent oxide tends also to retain the hydrocyanic acid as thiocyanate.

#### Analytical Methods.

The methods of analysis employed were in general those given by Linder (Alkali-Inspector's Reports) for the analysis of crude ammoniacal liquor, modified where necessary to allow for the much larger quantity of ammonia present in the concentrated liquor.

In these analyses the greater part of the time required is taken up in filtration, the precipitates such as Prussian blue and cuprous thiocyanate being under ordinary conditions difficult to filter. The following procedure very greatly reduces the time required for filtration and washing, and is applicable with advantage in large numbers of cases, where difficult precipitates have to be filtered and washed, but have not subsequently to be dried and weighed, but are either to be rejected or treated with reagents to dissolve or decompose them.

The filtration is effected through filter paper pulp which is readily prepared by shaking torn filter paper in a bottle with water until it is pulped. A layer of the wet pulp is then poured on to a perforated porcelain plate in a funnel connected to an evacuated filter-pump flask, and after the water has been extracted the mass is gently pressed down with the finger. Occasionally, difficult precipitates such as Prussian blue may run through the filter at first, and in such cases they are completely retained by using only a moderate vacuum at the beginning and passing the first filtrate through the same pulp bed a second time.

*Hydrogen sulphide.*—This may be determined by Linder's method of precipitation with ammoniacal



zinc chloride solution, and treatment of the zinc sulphide formed with hydrochloric acid and decinormal iodine. For the concentrated liquor the following method was found more rapid:—10 c.c. of the sample is added to 50 c.c. of *N*/10 iodine solution mixed with more than enough hydrochloric acid to neutralise the whole of the ammonia, and the unused iodine titrated with *N*/10 thiosulphate and starch, the difference giving the iodine used. A further 10 c.c. is then treated with cadmium chloride to remove all hydrogen sulphide, and, after filtering through pulp, added to 10 c.c. of *N*/10 iodine and hydrochloric acid, titrating back with *N*/10 thiosulphate. The amount of iodine used, which represents that taken up by substances other than hydrogen sulphide, is deducted from the amount of iodine found in the first test, the difference giving the number of c.c. of *N*/10 iodine corresponding to the  $\text{H}_2\text{S}$  in 10 c.c. of the liquor (1 c.c. of *N*/10 iodine = 0.0017 gm.  $\text{H}_2\text{S}$ ).

**Carbonic acid.**—10 to 25 c.c. (according to the amount of  $\text{CO}_2$  present) is run from a pipette into a hot solution of calcium chloride, avoiding as far as possible exposure to the air, from which the solution absorbs carbon dioxide. The beaker is covered and heated until the precipitated calcium carbonate granulates. This is then filtered and washed, the precipitated calcium carbonate dissolved in 25 c.c. of *N*/1  $\text{HCl}$ , the unused hydrochloric acid being determined by titration with *N*/1 caustic soda, using methyl orange as indicator (1 c.c. of *N*/1  $\text{HCl}$  = 0.0022 gm. of  $\text{CO}_2$ ).

**Phenols** were determined by the iodine method given by Skirrow (this Journal, 1908, 58) for the estimation of phenols in crude ammoniacal liquor.

**Pyridine bases** were ascertained by the method of Pennock and Morton (J. Amer. Chem. Soc., 1902, 24, 377).

**Ammonium thiosulphate.**—An approximate estimation of this salt is obtained from the number of c.c. of iodine required by 10 c.c. of the liquor after treatment with cadmium chloride, as found in the estimation of hydrogen sulphide above. Each c.c. of iodine = 0.0148 gm. of  $(\text{NH}_4)_2\text{S}_2\text{O}_3$ . The results tend to be slightly high, owing to the other impurities such as the thiocyanate and phenols having some action on iodine.

**Ammonium cyanide.**—The exact determination of the true amount of free ammonium cyanide actually present in a bulk of either crude or concentrated ammoniacal liquor is a matter of considerable difficulty. This arises, not from inexactitude of the analytical method, but on account of the rapid manner in which the cyanide undergoes conversion into thiocyanate by the action of the air during the taking of the samples, storing them, and handling them in carrying out the test. All figures of the amount of free cyanide in samples which have been in contact with air, or have stood for any length of time before analysis, merely give the amount of cyanide still remaining unchanged, which may be quite different from that of the bulk sample. For many purposes the total cyanogen content of the concentrated liquor is the chief information desired, and this change of cyanide into thiocyanate is unimportant, as any hydrocyanic acid not estimated as cyanide is found as thiocyanate; in these cases, therefore, the simplest plan is to add a few drops of ammonium polysulphide to the liquor taken for analysis to convert all cyanide into thiocyanate, and to omit the cyanide determination.

When required, the following method of estimation of the cyanide has been found most suitable, but with all precautions it is very difficult to avoid some conversion of cyanide into thiocyanate.

100 c.c. of the concentrated liquor is taken, if possible directly from the bulk to be analysed, if not, from a sample taken and preserved from

contact with air as much as possible, and slowly run from the pipette into a flask containing about 150–200 c.c. of boiling water to which about 10 c.c. of *N*/1 caustic soda has been added. The water in the flask should have boiled for some minutes before the addition of the sample so as to ensure the removal of all dissolved oxygen. When the bulk of the ammonia has been driven off, a solution of 5 grms. of lead nitrate is added, and the mixture distilled for 20–25 minutes; the distillate is collected in water containing 25 c.c. of *N*/1 caustic soda, the end of the condenser dipping beneath the surface of the liquid in the receiver. All hydrocyanic acid present as ammonium cyanide is found in the distillate, which is titrated with *N*/10 silver nitrate in the usual manner, adding a little potassium iodide as indicator (1 c.c. *N*/10  $\text{AgNO}_3$  = 0.0054 gm.  $\text{HCN}$ )\*.

**Ammonium ferrocyanide and thiocyanate.**—A few drops of ammonium polysulphide solution are added to 100 c.c. of the sample, till the solution shows permanently the yellow polysulphide colour, whereby any cyanide present is converted into thiocyanate. After standing about 15 minutes lead carbonate is added to remove hydrogen sulphide, and the filtrate boiled with addition of about 10 c.c. of *N*/1 caustic soda till most of the ammonia is driven off. The addition of a fixed alkali before boiling is necessary, as ammonium ferrocyanide loses part of its hydrocyanic acid when its solution is boiled, and the previous removal of hydrogen sulphide is also necessary, as in presence of ammonium sulphide hot alkali causes a partial conversion of ferrocyanide into thiocyanate, thereby rendering the results for the former too low and for the latter correspondingly high.

The solution is made slightly acid with sulphuric acid, and a solution of ferric alum added drop by drop till it shows the distinctive colour of ferric thiocyanate. The solution is then filtered through pulp, which retains the Prussian blue formed from the ferrocyanide, and is washed with water containing some dissolved electrolyte such as sodium or potassium sulphate. (Washing with distilled water often causes some colloidal Prussian blue to pass through the filter.) The precipitate contains all hydrocyanic acid present as ferrocyanide, and the filtrate all that present as thiocyanate.

(With small amounts of ferrocyanide, it frequently happens that the colour of the ferric thiocyanate makes it difficult to see whether any Prussian blue has been precipitated, but this shows up distinctly on the filter-paper pulp after washing. Filtration should be carried out in all cases, as even quantities of Prussian blue which cannot be estimated, if left in, interfere with the end reaction in the subsequent titration of the filtrate for thiocyanate).

About 10 c.c. of normal caustic soda is poured on to the filter pulp containing the Prussian blue, which decomposes it, forming sodium ferrocyanide. This is washed out with water, the filtrate strongly acidified with sulphuric acid, and distilled according to the method of H. E. Williams (this Journal, 1912, 468), with the addition of a little cuprous chloride, when all the hydrocyanic acid distils over, and is collected in water containing about 25 c.c. of *N*/1 caustic soda, as in the determination of the cyanide, the distillate being similarly titrated with *N*/10 silver nitrate. Williams' method has been found by a large number of tests, made with pure ferrocyanides, to give very accurate results, provided that the cuprous chloride used does not contain material amounts of cupric salts. A con-

\* E. Linder has drawn our attention to the fact that in boiling with caustic soda, some cyanide may be converted into thiocyanate by thiosulphate if this is present.

venient method is to use a 10% solution of cuprous chloride in hydrochloric acid, some copper strips being kept in the bottle to maintain the whole in the cuprous state.

*Ammonium thiocyanate.*—Two alternative methods may be employed for estimation of the thiocyanate in the filtrate from the Prussian blue, the first being the quicker, but rather less accurate for an unpractised eye.

The first method consists in adding to the filtrate a further quantity of ferric alum, heating to 95°, and after five minutes, cooling well, then adding dilute nitric acid (free from nitrous and hydrochloric acids), and titrating by Volhard's method with *N*/10 silver nitrate until the red colour of the ferric thiocyanate disappears (1 c.c. of *N*/10  $\text{AgNO}_3$  = 0.0027 gm. HCN or 0.0076 gm.  $\text{NH}_4\text{CNS}$ ). The solution in this case does not become colourless, but almost always retains a pink colour, due to the action of the ferric salts on the phenols present, and this makes the end-point of the titration rather less easy to decide, but with a little practice there is no difficulty in ascertaining the point where the colour due to ferric thiocyanate disappears.

To obviate this difficulty entirely, the second method, which effects the removal of the phenols and other impurities present, is preferable until experience has been gained in the titration by the first method. This method must always be employed if chlorides are present. In it the filtrate from the Prussian blue, which should be only slightly acid, is heated to boiling with the addition of more than sufficient sodium sulphite to reduce the whole of the ferric salt present to the ferrous state; if a large excess of ferric alum has been used for precipitation of the ferrocyanide a correspondingly large amount of sulphite is required. Copper sulphate solution is then added in excess, and the precipitated cuprous thiocyanate filtered through pulp, washed, and the pulp and precipitate stirred with a hot solution of caustic soda or sodium carbonate (free from chlorides) and filtered. The addition of a few drops of ferric alum solution to the alkaline solution (forming ferric hydroxide) often facilitates the filtering and washing of the mixture through pulp. The well-cooled filtrate is then acidified with dilute nitric acid (free from nitrous and hydrochloric acids), ferric alum added as indicator, and the titration with *N*/10 silver nitrate made as before till the colour of ferric thiocyanate disappears, the solution now becoming completely colourless.

If the amount of cyanide has been previously estimated the amount found must be deducted from that found as thiocyanate, as this figure includes all hydrocyanic acid present as cyanide in the original sample, owing to the preliminary treatment specified with ammonium polysulphide.

#### Check Tests.

A number of check tests have been made by adding known amounts of ammonium cyanide, ferrocyanide, and thiocyanate to pure 25% ammonia, small quantities of phenols being also added to the check solutions. In absence of hydrogen sulphide, very accurate results are obtained in the determination of the hydrocyanic acid present in all three forms, and in the total amount present when tested in the manner specified above, but when hydrogen sulphide is present, although the total hydrocyanic acid present is found correctly, the amount present as cyanide is always found too low, and that as thiocyanate correspondingly high. The following two examples, one with, and one without hydrogen sulphide, exemplify the results obtained:—

	(1) H <sub>2</sub> S absent		(2) H <sub>2</sub> S present	
	Taken	Found	Taken	Found
$\text{NH}_3$ (approx.)	25.0	—	25.0	—
HCN as $\text{NH}_4\text{CN}$	0.0200	0.0200	0.0200	0.0189
HCN as $(\text{NH}_4)_2\text{FeC}_6$	0.0440	0.0437	0.0440	0.0137
HCN as $\text{NH}_4\text{CNS}$	0.0306	0.0308	0.0306	0.0316
H <sub>2</sub> S (approx.)	nil	—	0.5	—
Phenols (approx.)	0.2	—	0.2	—
Total HCN	0.0946	0.0945	0.0946	0.0942

#### DISCUSSION.

Mr. P. PARRISH said that he had had occasion to use the air method for the fixation of cyanides and could confirm all the authors had said in regard to its efficacy. There was, however, one possibility in connection with the adoption of this method which might prove a difficulty. He did not know to what extent the authors recommended the application of air nor to what extent the volume of the waste gases leaving the dissociator increased, but it seemed to him that if the dissociator was in direct connection with the spent oxide burners, the hydrogen sulphide and carbon dioxide which were passing forward and which were burned in the spent oxide burners might bring with them an appreciable percentage of diluent gas in the shape of nitrogen and oxygen. That might constitute a distinct drawback from the point of view of intensive working of the sulphuric acid plant. That was a much greater difficulty than any which was likely to arise by reason of the association of air with the steam and ammonia, for which the authors suggested the enlargement of the reflux condensers. He had been hoping to hear from the authors some comments with regard to the application of this very comprehensive investigation to the possible utility of concentrated ammonia liquor in lieu of liquor ammonia in the process of oxidation of ammonia, by a platinum catalyst, to nitric acid. The majority of people who were operating ammonia oxidation plants at the moment were very loth to work with concentrated ammonia liquor. On the other hand, American experience was that neither the hydrocyanic acid, the hydrogen sulphide, the pyridine, nor the carbon dioxide had any inhibiting effect on the platinum catalyst. Personally he hoped to put into operation very shortly a plant working on commercial concentrated ammonia liquor, not specially purified, and that should give an opportunity of confirming a number of the data which had been brought forward in the paper. The intention was simply to operate a vaporiser through which the requisite quantity of air would be blown by a fan, and in that way it was quite conceivable there would be a very rapid oxidation of the ammonium sulphide with the consequent formation of ammonium polysulphide, and the fixing of any volatile hydrocyanic acid that might otherwise result. He would not be inclined to recommend, nor indeed would he countenance, the use of spent oxide for the fixation of the cyanides. When he had applied the air method three years ago he had suspected that it had caused a perceptible dilution of the gases going forward to the sulphuric acid plant and that interfered with the intensive working of the plant. He had therefore turned to the use of sulphur, which lent itself to ready application to the dissociator, but had found it was necessary to use something of the order of 1% of sulphur on the concentrated ammonia liquor in order to fix the cyanides to such an extent as to keep them below the limit of 0.01% as ammonium thiocyanate. That represented 22.4 lb. of sulphur with a sulphur content of about 99% purity. If spent oxide was used the sulphur content was only about 45% and that meant using something of the order of 50 lb. of spent oxide in order to secure the fixation of the cyanides to such an extent as to conform to the specification of the



Ministry of Munitions. He was certain that if the spent oxide had been used for a week difficulty would have been experienced by the blocking of the serrated edges of the decomposing still and much more frequent cleaning than usual would have been necessary.

He had had an opportunity of installing a vessel between the ammonia still and the dephlegmator, operating practically on the principle of the Williams thiocyanate process. The arrangement he had made was to insulate the vessel in order that it should retain its heat and thus prevent any undue condensation, and that was remarkably effective. He had been able to reduce the thiocyanate in the liquor to something of the order of 0.008%, so he quite appreciated that the addition of spent oxide, at the point at which he now understood the authors recommended its application, would be effective.

Prof. J. C. PHILIP, in connection with the analytical methods described, drew attention to the fact that ferric thiocyanate solutions were not stable, and emphasised the necessity of analysing these solutions within a comparatively short time of the addition of the ferric salt to the thiocyanate. The rate at which reduction of the ferric salt occurred was not by any means negligible. That was his experience when thiocyanate was in excess, and he imagined that the same precaution was necessary when ferric salt was in excess. In his experiments with ferric thiocyanate he had found a reduction of 10% in an hour at 25° C., but, of course, the conditions were not quite the same as in the authors' analytical procedure.

Dr. F. M. PERKIN said that the carbonic acid present in the normal concentrated ammonia liquor which was supplied commercially was driven off with the ammonia and was one of the bugbears of those who had to deal with platinum catalysts. It was not so much that trouble was caused to the catalyst itself but by the pipes and the compressors becoming blocked up with ammonium carbonate and carbamate, and it was necessary to put in purifiers in order to get rid of this. Probably also, if cyanides or cyanates passed forward they would have a deleterious effect on the catalysts. There was no doubt that ammonia liquor from gas was much more difficult to work with in a catalysing plant than pure ammonia liquor or ammonia produced from cyanamide. Very considerable difficulties had been met with in the plants which had to deal with the manufacture, on a large scale, of nitric acid, owing to the difficulty of the removal of the pyridine bases. Several methods were in use, but they all entailed cost and very considerable plant.

Dr. R. LESSING asked if, instead of the iron oxide mentioned by Mr. Parrish, which undoubtedly had to be added in the dissociator in considerable quantities, and which would also precipitate ferric hydroxide which would tend to block up the serrated edges of the stills, an iron sludge of ferrous hydroxide and carbonate would be useful. This could be added in quantities not much exceeding the actual quantity of hydrocyanic acid and would, he thought, effect the conversion of some of the hydrocyanic acid into ferrocyanide. It was a somewhat different proposal from that mentioned by the authors, in which the vapours from the still were actually scrubbed with a suspension of ferrous sludge.

Mr. H. HOLLINGS asked whether the fact that liquor from coke ovens contained four times the quantity of cyanogen compounds found in liquor from gas works, had been in any way connected

with the difference in the carbonising conditions in the two cases.

Dr. COLMAN said that he was glad to receive Mr. Parrish's confirmation of the beneficial result of passing air with the steam through the still. In respect to the addition of spent oxide, he gathered that Mr. Parrish's remarks referred to the addition of this material to the preheater or the still, whereas his own reference was to the addition of it to the lime washer, where this apparatus was interposed between the still and the reflux condenser. While spent oxide could be added to the lime washer where one was provided to remove hydrocyanic acid, he did not recommend it as a general practice, but only as an emergency measure. His own belief was that by proper attention to the preheater, and ensuring that a sufficiency of lime was added, together with the use of some air in the still, the hydrocyanic acid could be almost completely removed. As he had mentioned, many of these plants were put up as emergency plants, and many things had to be done which would not be recommended as a regular procedure, and this was one of them. The proper plan was, however, to try to arrange matters so that the hydrocyanic acid was retained in the still.

With regard to the point raised by Dr. Philip, whilst it was quite true, as Dr. Philip had shown, that under certain conditions thiocyanate underwent fairly rapid change when ferric salts were present, he had not found evidence of any such change under the conditions of the test in any interval likely to occur in practice between the filtration of the thiocyanate solution and its titration. With regard to the use of concentrated liquor for the catalytic nitric acid process, he had no knowledge as to the effects of the various impurities. This investigation had arisen through matters being referred to him in connection with the question of the presence of cyanides affecting the manufacture of ammonium nitrate, and the tests for other impurities were incidental, and rather for general information than with respect to any particular process. In respect to the use of iron sludge for removing the hydrocyanic acid from the still gases, whilst not wishing to speak too definitely, his experience was that iron sludge did not keep back the whole of the hydrocyanic acid unless a fixed alkali was also added, as the product was ammonium ferrocyanide, which in hot solution readily lost hydrocyanic acid, unlike the ferrocyanides of the fixed alkalis. In reply to Mr. Hollings he had not made as large a number of estimations of the hydrocyanic acid in coke-oven gas as in that from gas retorts, but so far as his figures went, the amount produced in the latter was distinctly greater. That in spite of this the amount of free cyanide in gas-works liquor was less than in coke-oven liquor was most likely due, as he had stated, to the gas-works liquor being stored for a much longer period than coke-oven liquor, during which time the cyanide was chiefly converted into thiocyanate.

#### ANALYSIS OF COMMERCIAL "PURE" BENZOLS.

BY F. BUTLER JONES, B.A. (CANTAB.), A.I.C.

*Theoretical considerations.* The depression of the freezing point of a liquid, caused by the presence of a dissolved substance, is, in general, proportional to the weight of solute dissolved in a constant weight of the solvent. Experiments carried out with benzene as solvent, and carbon bisulphide, thiophen, toluene, and paraffin as solutes, have shown that the depression of the freezing point of benzene, occasioned by the presence of up to 4%

of these substances, is nearly proportional to the weight per cent. of solute, i.e., to the weight per 100 parts by weight of solution. It is convenient, however, when dealing with a mixture of liquids, to express the composition in terms of volume percentages, i.e., in volumes per 100 volumes of solution. For liquids such as the above-named it can be shown algebraically that small volume percentages are nearly proportional to the corresponding weight percentages, so that within certain limits the lowering of the freezing point may be taken as being proportional to the volume percentage of solute.

From a mixture containing benzene, thiophen, toluene, and "paraffin" only, the carbon bisulphide and the thiophen can be successively and completely removed without any alteration of the quantity of the other constituents. It is evident that the removal of these substances produces an increase of the concentration of the remaining solutes, but, as will be explained later, this effect is negligible if the concentrations are small. There is thus provided a means of estimating the percentages of carbon bisulphide and thiophen, for the quantity of the former is proportional to the difference between the freezing points of the mixture before and after its removal, while that of the latter is proportional to the difference between the freezing points of the mixture before and after the subsequent removal of the thiophen.

The remaining liquid is a mixture of benzene, toluene, and paraffin. The amount by which its freezing point is below that of pure benzene ( $5.48^\circ\text{C}.$ ) is a measure of the sum of the percentages of toluene and paraffin, i.e.,

$$\delta\theta = k_1 \times \text{toluene \%} + k_2 \times \text{paraffin \%},$$

where  $k_1$  and  $k_2$  are the freezing point depressions produced by 1% of toluene and paraffin respectively.

One equation, however, is insufficient to evaluate the two unknowns, toluene % and paraffin %. Another equation is required, and this is provided by the specific gravity. The amount by which the sp. gr. is lower than that of pure benzene (0.8848 at  $15.5^\circ/15.5^\circ\text{C}.$ ) is also a measure of the sum of the quantities of toluene and paraffin, i.e.,

$$\delta \text{ (s.g.)} = g_1 \times \text{toluene \%} + g_2 \times \text{paraffin \%},$$

where  $g_1$  and  $g_2$  are amounts by which the specific gravity of pure benzene is lowered by the presence of 1% of toluene and paraffin respectively;  $g_2$ , which is not truly constant, cannot be calculated from a knowledge of the specific gravities of benzene and paraffin, for the volume produced by mixing benzene and paraffin is greater than the sum of the separate volumes, the magnitude of the expansion depending upon the relative proportions. It may, however, be taken as constant for small percentages, and can easily be found experimentally. For the four unknowns, therefore, there are available four equations involving six determinable constants, and four observed quantities.

1.  $\Delta\theta = c_1 \times \text{carbon bisulphide \%}$ .
2.  $\Delta\theta = c_2 \times \text{thiophen \%}$ .
3.  $\delta\theta = k_1 \times \text{toluene \%} + k_2 \times \text{paraffin \%}$ .
4.  $\delta \text{ (s.g.)} = g_1 \times \text{toluene \%} + g_2 \times \text{paraffin \%}$ .

These equations can be solved graphically, and for this purpose a graph has been constructed giving the volume percentages of the four solutes in terms, not of temperature and specific gravity differences, but of observed temperatures and specific gravity.

*Preparation of pure materials.* The purest materials available for the determination of the constants were:—

*Benzene*, b. pt. (corr.)  $80.3^\circ\text{C}.$ , sp. gr. ( $15.5^\circ/15.5^\circ$ ) 0.8846, prepared from chosen samples of commercially pure benzol in the following way. The carbon bisulphide was first removed by means of alcoholic potash, the alcohol being thoroughly washed out

with water. The residual benzene was then shaken with strong sulphuric acid until all traces of thiophen had been removed. It was then distilled through a dephlegmator, the portion distilling at a constant temperature being collected. It contained about 0.1% of paraffin as the only impurity.

*Carbon bisulphide*, b. pt. (corr.)  $46.25^\circ\text{C}.$ , sp. gr. ( $15.5^\circ/15.5^\circ$ ) 1.2713, prepared from "A.R." material by shaking with mercury, followed by distillation.

*Thiophen*, b. pt.  $84^\circ\text{--}84.5^\circ\text{C}.$ , sp. gr. ( $15.5^\circ/15.5^\circ$ ) 1.0703, prepared from Kahlbaum's pure thiophen by redistillation.

*Toluene*, b. pt. (corr.)  $110.8^\circ\text{C}.$ , sp. gr. ( $15.5^\circ/15.5^\circ$ ) 0.8720, prepared from chosen pure toluol samples by a method similar to that used in the case of benzene.

*Paraffin*, b. pt.  $93^\circ\text{--}97^\circ\text{C}.$ , sp. gr. ( $15.5^\circ/15.5^\circ$ ) 0.7231, prepared from a sample of pure benzol from a gasworks tar by exhaustive treatment first with approximately 5% oleum, and finally, in order to avoid the possibility of attacking the paraffin, with cold 100% sulphuric acid.

*Determination of the constants.* Mixtures of benzene with small quantities of the four solutes were carefully made up by weighing, and the freezing points determined. As a matter of interest, the molecular weights of the solutes were calculated. Those of carbon bisulphide and toluene averaged slightly higher than the formula weights. That of the paraffin appears to be about 105. Thiophen, as is well known, does not separate in the pure state, but as a solid solution of constant composition. The molecular weight of 131—133 was calculated from the data (formula wt. = 84).

The following figures were finally obtained, and were used in drawing the graph:—

Substance.	Depression of f. pt. of benzene caused by the presence of 1% vol./vol.	Depression of sp. gr. of pure benzene produced by presence of 1% vol./vol.
Carbon bisulphide ..	$0.94^\circ\text{C}.$ ( $C_1$ )	—
Thiophen ..	$0.465^\circ\text{C}.$ ( $C_2$ )	—
Toluene ..	$0.52^\circ\text{C}.$ ( $k_1$ )	0.0001 ( $g_1$ )
Paraffin ..	$0.425^\circ\text{C}.$ ( $k_2$ )	0.0018 ( $g_2$ )

*Nature of impurities.* The impurities in a well-washed sample of commercially pure benzol occur with one possible exception only in very small quantities. Unsaturated compounds should be negligible in amount or altogether absent; toluene, carbon bisulphide, and thiophen may be present to the extent of a few tenths per cent. "Paraffin," however, may vary from a small trace to as much as 3% in somewhat rare cases. It is highly probable that this paraffin varies in composition according to the circumstances under which it is produced (temperature of carbonisation, type of retort or oven, etc.). In one particular case, the paraffin occurring in the pure benzol prepared from a gas tar was isolated, and was found to have sp. gr. 0.7231 ( $15.5^\circ/15.5^\circ$ ), and to boil over a range of  $4^\circ\text{C}.$  from  $93^\circ\text{C}.$  to  $97^\circ\text{C}.$  From its physical and chemical properties it appears to consist of a mixture of hydrocarbons of the methane series with cyclic hydrocarbons of the polymethylene series. In spite of its relatively high boiling point, as much as 2.5—3% of paraffin may occur in a pure benzol having a distilling range (5% to 95%) of  $0.5^\circ\text{C}.$  In this case the major part of the paraffin must distil with the benzene as a constant boiling mixture.

*Application of the method.* It is evident, therefore, that a method of analysis such as that described above can be applied to the case of commercial pure benzols. Up to the present time, in order to carry out the complete analysis of a sample of "pure" benzol, it would have been necessary to estimate the carbon bisulphide and the thiophen by gravimetric methods; the toluene by the fractionation of a large bulk of the sample to obtain a residue rich in toluene (the composition of which could be found from its distillation characteristics), and the paraffin either from the consideration of the



specific gravity of the sample after the removal of carbon bisulphide and thiophen, or by the removal of aromatic hydrocarbons by exhaustive treatment with sulphuric acid.

The proposed method has the advantage that by its aid an analysis can be completed within one day by the employment of simple operations. By suitable arrangement of the work, this rate is considerably increased when several analyses are carried on together.

*The freezing point measurements.* The following provides a means whereby small quantities of carbon bisulphide, thiophen, toluene, and paraffin may be estimated with little trouble, and with a fair approach to accuracy, the measurements required being three freezing points and one specific gravity. The freezing points are determined in the usual way, by means of any thermometer capable of giving readings accurately to  $0.01^{\circ}\text{C}$ . over the range  $0-5.5^{\circ}\text{C}$ . Since, on an average, a difference of  $0.01^{\circ}\text{C}$ . corresponds with about 0.02% of impurity (0.01% in the case of carbon bisulphide), this is the theoretical accuracy of the method. Should the best thermometer available be one divided in  $0.1^{\circ}\text{C}$ . readable to  $0.05^{\circ}\text{C}$ ., results can be obtained accurately only to 0.1% (0.05 in the case of carbon bisulphide). In this case of course the thermometer corrections must be known to  $0.05^{\circ}\text{C}$ .

*The method of analysis.* The ice points of the thermometer having been determined, the freezing point (F.P.1) of the sample is taken. The carbon bisulphide is then completely removed by treatment for not more than one hour with alcoholic potash, avoiding the use of an undue excess of alcohol, followed by three washings with water. The freezing point of the residual benzene free from carbon bisulphide is determined (F.P.2). The thiophen is then completely removed from the carbon bisulphide-free benzene, according to a method privately communicated by Dr. S. P. Schotz by shaking continuously for 2-3 hours with twice its volume of a cold solution of basic mercuric sulphate (Denigès' reagent), prepared by dissolving 5 grms. of mercuric oxide in a solution of 20 c.c. of concentrated sulphuric acid in 100 c.c. of distilled water and filtering the solution. The residual benzene is washed twice with cold water in a separating funnel, the water removed, the benzene filtered from suspended solid through a small filter, and its freezing point (F.P.3) and specific gravity at  $15.5^{\circ}/15.5^{\circ}\text{C}$ . are determined. The specific gravity must be measured accurately to within 5 in the fifth decimal place, a degree of accuracy obtainable without difficulty if a good type of pycnometer is used, and if the temperature of the bath is known to  $0.05^{\circ}\text{C}$ . In practice it is convenient to take 60-70 c.c. of the sample and remove the carbon bisulphide. The residual liquid is then divided into two portions, 20-30 c.c. being reserved for the determination of F.P.2. The thiophen is removed from the remaining 40 c.c. It is advisable in all cases to prove the absence of thiophen from the final residue by the isatin and sulphuric acid test.

*Employment of the graph.* The volume percentage (c.c. per 100 c.c.) of carbon bisulphide is obtained from the graph by reference to F.P.1 and F.P.2; the thiophen from F.P.2 and F.P.3; and the paraffin and toluene from F.P.3 and the specific gravity. Theoretically it is necessary to apply corrections to all the values obtained, to allow for the increased concentration of the other impurities incident on the removal of carbon bisulphide and of thiophen, but in practice the smallness of the quantities present and the limits of accuracy of the method render this unnecessary.

*The effect of water.* The material does not need to be dried after the various extractions, for water appears to be sufficiently insoluble in benzene, at temperatures near  $5^{\circ}\text{C}$ ., not to affect the freezing point. The liquid of which the specific gravity is

to be determined should, however, be dried with a pellet of calcium chloride.

*Effect of other impurities.* The condition essential to the successful application of the method is that impurities other than the above named shall be either absent altogether, or present in not more than the most minute quantity. The sulphuric acid test (90 c.c. of the sample shaken for exactly 5 minutes with 10 c.c. of 90% sulphuric acid) must not show more than a pure lemon-yellow colour; this indicates that the analysis may be performed directly on the sample as received. Should the acid test show a deeper colour, the sample must be washed for 5 minutes with 5% of its volume of 90% sulphuric acid, followed by treatment with caustic soda solution, and several thorough washings with water before proceeding with the analysis. The presence of undesirable impurities is also indicated by the appearance during the treatment with alcoholic potash of a strong yellow colour that appears also in the extracted benzene. Care must be taken that the alcohol used is sufficiently pure, for should it contain aldehydes, coloured resinous substances are produced, and these ultimately contaminate the benzene, which should remain colourless after treatment. For the same reason it is inadvisable to leave the benzene and alcoholic potash in contact for more than an hour. The effect of the impurities in an insufficiently washed sample is to increase the apparent quantity of carbon bisulphide. The thiophen estimation may also be affected to a smaller extent.

*Results.* The method has been tested on mixtures of known composition made up from the same materials (with the exception of the benzene) as those used to obtain the figures for drawing the graph.

(1) A known mixture of benzene, carbon bisulphide, thiophen, toluene, and paraffin (the benzene used contained 0.1% of paraffin).

	Taken	Found
F.pt.1 = $4.43^{\circ}\text{C}$ . Carbon bisulphide	0.206	0.21
F.pt.2 = $4.63^{\circ}\text{C}$ . Thiophen	0.499	0.47
F.pt.3 = $4.84^{\circ}\text{C}$ . Toluene	0.129	0.15
Sp.gr. = 0.8821 Paraffin	1.29 (1.19 + 0.1)	1.3

(2) A known mixture similar to (1), but containing in addition a small quantity of hexylene. The mixture was divided into two portions, one of which was analysed directly, while the other received a preliminary acid washing, as already described. The benzene used contained 0.33% of paraffin.

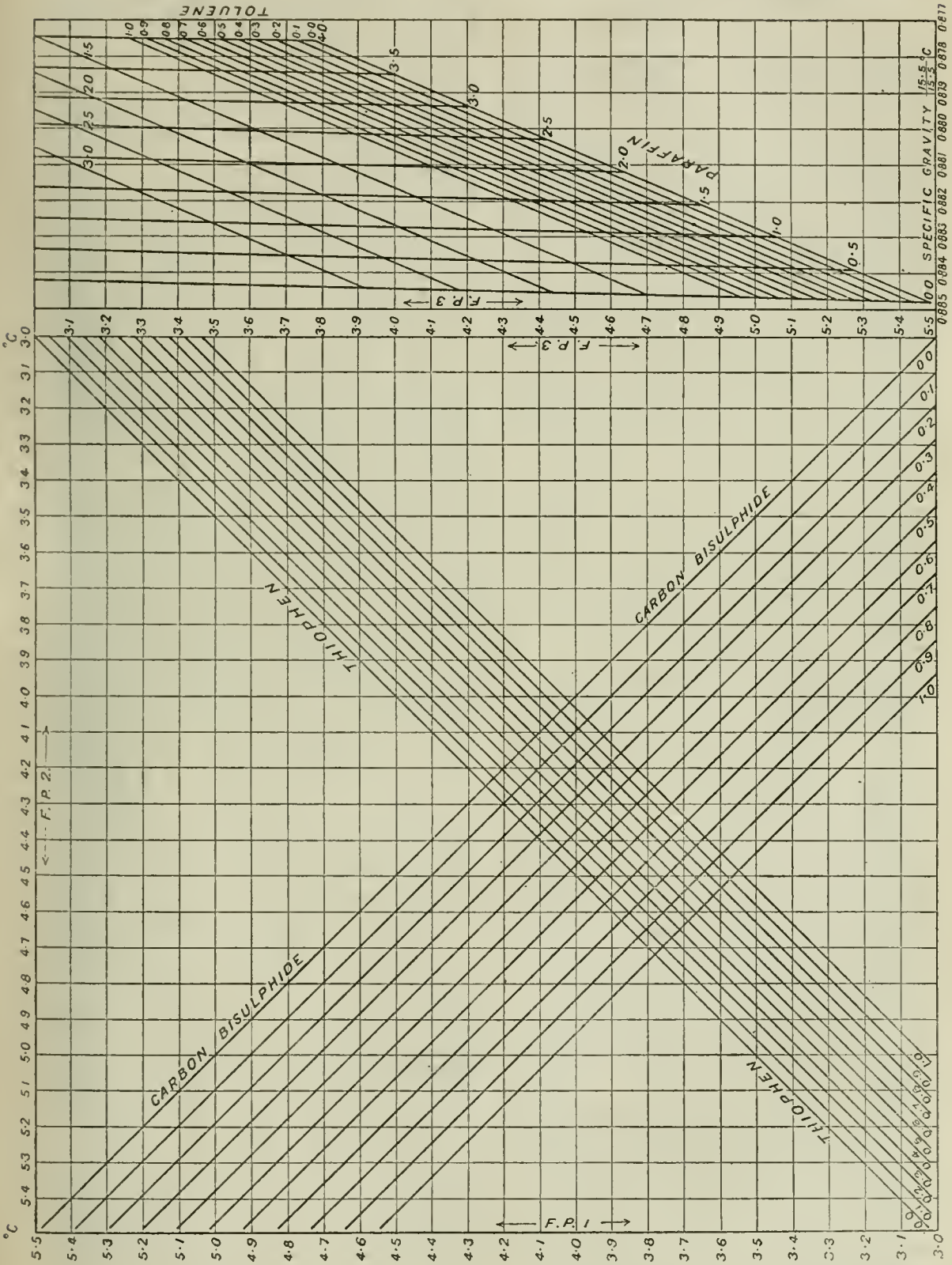
	Not acid washed	Acid washed		Taken	Found	
					Not acid washed	Acid washed
F.pt.1	3.95° C.	3.99° C.	Carbon bisulphide	0.367	0.43	0.36
F.pt.2	4.35° C.	4.32° C.	Thiophen	0.565	0.46	0.50
F.pt.3	4.56° C.	4.55° C.	Toluene	0.115	0.11	0.12
Sp.gr.	0.88115	0.88115	Paraffin	1.97	2.0	2.0
				(1.64 + 0.33)		

(3) *Sample of pure benzol.*

Sp.gr. = 0.8862	..	Distillation range (5% to 95%)	= $0.4^{\circ}\text{C}$ .
		Sulphuric acid test	= yellow.
F.pt.1 = $5.06^{\circ}\text{C}$ .	..	Carbon bisulphide	= 0.03%
F.pt.2 = $5.09^{\circ}\text{C}$ .	..	Thiophen	= 0.69%
F.pt.3 = $5.37^{\circ}\text{C}$ .	..	Toluene	= 0.10%
Sp.gr. = 0.8845	..	Paraffin	= 0.15%

The method can also be applied in certain cases to well-washed commercial benzols, and for this reason the toluene graph has been extended to 3%; but in such a case the accuracy of the method is diminished. Should the quantity of toluene exceed about 3%, the method of analysis devised by Spielmann and Wheeler (this J., April 15, 1916, p. 396) is more widely and more easily applicable.

I wish to record with pleasure my thanks to Dr. Percy E. Spielmann for his continued interest and for the valuable advice which he has given during the preparation of this paper.





## THE ANALYSIS OF ALUMINIUM ALLOYS AND METALLIC ALUMINIUM.

BY J. J. FOX, D.SC., F.I.C., E. W. SKELTON, B.SC., A.I.C.,  
AND F. R. ENNOS, B.A.(CANTAB.), B.SC.

The extended use of aluminium alloys has directed attention to the methods of analysis of these alloys and several communications dealing with the subject have appeared recently. A paper by Withey (Institute of Metals, March, 1916) gives a fairly complete account of the methods for the determination of the important constituents and impurities in aluminium and its alloys. Stansbie (this J., 1917, 802) describes a method which is suitable for works but its use is limited, as will be shown later. Recently another paper has been published on the subject by Collett and Regan (this J., 1918, 91); the process described by these authors is suitable for dealing with certain alloys made according to known specifications from pure materials, minor impurities being either absent or negligible in amount. The alloys with which we have had to deal, however, have been of varied composition and on this account we have had to select or devise methods by which all the constituents present can be determined both accurately and rapidly. We have therefore thought it desirable to give an account of the methods which we have found most suitable for *general* work, after considerable experience of the matter.

In those cases where only the more important constituents are to be determined, the processes described by us have no disadvantage, either in speed or accuracy, compared with the processes referred to above. On the contrary, a common alloy containing about 3% of copper, 13% of zinc, and free from manganese, nickel, and tin, can be examined by Method II. much more rapidly than by any process requiring precipitation of zinc sulphide. Where manganese, nickel, etc., are present the method is at least as rapid as any of the processes hitherto published.

It may be noted that the analysis of aluminium alloys containing tin has received little attention in the literature of the subject. A large proportion of the alloys examined by us have contained up to 2% of tin and occasionally more, while in others it is often present in small amounts, such as 0.2%. We have also found lead to be a very common impurity, generally in alloys containing a large proportion of zinc.

As will be seen, we prefer to determine copper electrolytically. In this connexion we should like to emphasise the importance of the use of hydrogen peroxide\* in place of nitric acid during the electrolysis of copper in the analysis of alloys containing a large amount of zinc. By this means a bright copper deposit is obtained and the resulting solution contains no nitrates or ammonia, thus permitting of the subsequent electrolytic determination of zinc and iron in soda solution.

During our work we soon came to the conclusion that in order to obtain the best results the method to be used must be specially adapted for the particular type of aluminium alloy under examination, and for the purpose of analysis we distinguish three types.

**Type 1.** Metallic aluminium, i.e. unalloyed aluminium. The examination in this case is for all metallic impurities and for silicon. Since any single impurity rarely exceeds 1% in metallic aluminium, the main impurities should be determined to within  $\pm 0.05\%$  and the determination of the lesser impurities should be subject to a much smaller error.

**Type 2.** Alloys containing up to 13% of copper, and zinc up to 3%. Tin, if present, is usually below

3%, and manganese, nickel, and magnesium may occur. All these constituents should be determined to at least within  $\pm 0.1\%$ , and generally more accurately. Impurities and constituents such as iron, lead, and silicon should be determined to at least 0.05%.

**Type 3.** Alloys containing up to 20% of zinc, copper up to 5%, and all other constituents not exceeding 1%. The zinc in such cases should certainly be determined within 0.2% and generally within  $\pm 0.1\%$ .

These limits may appear rather wide in some cases, but it is our experience that none of the methods hitherto proposed leads to any greater (or as great) accuracy, especially with the zinc.

Before proceeding to the determination of the constituents of an unknown aluminium alloy, it is advisable to make a rapid qualitative test in order to determine to which of the three types the metal belongs. If the metal is in bulk, a very fair indication can be obtained from the nature of the drillings. Unalloyed aluminium is difficult to drill with  $\frac{1}{8}$ " drills. It tears away and clogs the drill. Alloys with a high copper and low zinc content drill fairly short and clean and the drillings have a bluish tinge. Alloys with high zinc content generally show some slight tendency to tear and the drillings are long and white. Alloys containing magnesium give long white drillings but there is no tendency to tear.

The qualitative test is carried out most expeditiously according to the following scheme. About a gram of the alloy is allowed to react with 40 c.c. of pure 10% sodium hydroxide solution, the action being regulated by warming or cooling the solution. When evolution of gas has ceased the clear solution, which contains the bulk of the aluminium and zinc, is decanted from the brown residual metal into another beaker, and a few c.c. of sodium sulphide solution are added to it. From the bulk of the precipitate a rough estimate of the zinc content can be formed. The residual metals in the first beaker are washed by decantation with distilled water and then dissolved in a little nitric acid. The resulting solution is neutralised with a little ammonia, acidified with hydrochloric acid, and the copper (the presence or absence of which is by this time evident) and other Group II. metals removed by the passage of hydrogen sulphide. Group II. metallic sulphides are filtered off, the filtrate is boiled to remove hydrogen sulphide, and then oxidised with a little bromine water and the iron and aluminium precipitated with ammonia and filtered off. Nickel if present yields a blue solution at this stage, and it must be removed by dimethylglyoxime. Ammonium sulphide is added to remove any zinc and the filtrate from the zinc sulphide tested for magnesium. It is unnecessary to examine specially for lead, manganese, and tin, as these are found in the ordinary course of analysis. Nickel should be tested for carefully, because if its presence is unsuspected, all results after Group II. in the main analysis will be vitiated unless special precautions are observed. Magnesium is present in most samples, but generally as a small harmless impurity, and its estimation is frequently not required. In very few cases have we found nickel in alloys of the third class, i.e. those containing a large amount of zinc.

The recorded methods for the analysis of aluminium alloys describe two ways of attacking the metal: (1) complete dissolution by means of acid, (2) partial attack by means of sodium hydroxide solution whereby the zinc and aluminium are stated to be dissolved. The second procedure is certainly advantageous, inasmuch as most (but not all) of the zinc and aluminium are separated from the bulk of the copper and iron. Tin if present is partly dissolved. Traces of iron and

\* Mr. W. Brash, B.Sc., A.R.C.S., has informed the authors that he has employed hydrogen peroxide when determining copper by electrolysis in brass and bronze.

manganese may also pass into solution, as is indicated by the dark coloured precipitate sometimes obtained when sodium sulphide solution is added to the solution of sodium aluminate and zincate. We have made a series of experiments with aluminium alloys containing from 2 to 15% of zinc, many of the metals being in the form of fine sawings as recommended by Stansbie (*loc. cit.*). The alloys were boiled with 10% sodium hydroxide for periods varying from one half to six hours, and it was found that from 0.2% of zinc in the low zinc alloys to as much as 1.6% in the high zinc alloys, remained with the undissolved portion of the metal. We desire to call particular attention to the fact that any process for zinc relying solely on the extraction with soda will give low results, unless the zinc in the undissolved metal is determined. Moreover, if tin is a constituent of the alloy, electrolysis of the soda solution leads to deposition of tin with zinc. For this reason, we are of opinion that the most satisfactory general method of attacking the alloy is by complete dissolution of the metal.

None of the methods hitherto described gives any indication that there is serious difficulty in obtaining an accurate determination of tin. The process usually recommended is to dissolve the metal in nitric acid, evaporate to dryness, filter the insoluble matter, and weigh the tin oxide; but there are several difficulties in this procedure which call for comment. In the first place, aluminium alloys containing small proportions of copper and zinc are not easily dissolved by nitric acid and the evaporation to dryness of aluminium nitrate is not easy and requires a good deal of attention. Besides this, the resulting tin oxide is usually contaminated with silica and with iron and aluminium oxides. It cannot be assumed to consist of tin oxide alone, even approximately. Furthermore in the case of some alloys it is practically impossible to render the tin oxide insoluble by merely evaporating the solution to dryness. The determination of tin by conversion into oxide of the stannic sulphide precipitated in Group II. after electrolysis of the copper in sulphuric acid solution, has been found to give results which are satisfactory but very slightly high. Under the conditions of acidity prevailing in our method of analysis, no tin sulphate is found to hydrolyse out with the silica, nor is any tin deposited with the copper during electrolysis.

The methods here described are those which we have found to be most generally useful. The reagents required are (a) a 10% solution of pure sodium hydroxide, (b) nitro-sulphuric acid made by mixing 300 c.c. of concentrated sulphuric acid with 300 c.c. of water, cooling, and adding 200 c.c. of pure nitric acid (sp. gr. 1.42).

METHOD I. For unalloyed aluminium and for alloys with low zinc (type 2).

**Lead.** Two grms. of the metallic drillings is weighed out into a 300 c.c. tall beaker, covered with about 70 c.c. of 10% sodium hydroxide solution, and allowed to dissolve till evolution of gas practically ceases. The caustic soda solution is then decanted off through a prepared Gooch crucible and the insoluble portion washed with water. It is then dissolved in a little nitric acid and the solution evaporated to fuming with 10 c.c. of concentrated sulphuric acid on a hot plate. When cool, the liquid is diluted to about 50 c.c. with water and allowed to stand until the lead sulphate and some silica have separated completely. The precipitate is filtered off on a Gooch crucible and washed with dilute (1 in 6) sulphuric acid. The contents of the Gooch crucible containing the lead sulphate (and silica) are extracted with hot 30% ammonium acetate solution; the extract is acidified with hydrochloric acid, and the lead removed as sulphide by passing hydrogen sulphide

through the solution. The sulphide is filtered off, dissolved in nitric acid, evaporated with a small quantity of sulphuric acid, and estimated as lead sulphate in the ordinary way. If there is not much silica with the lead sulphate, as generally happens, it is accurate to ignite the mixture on the Gooch crucible to a dull red heat, cool, weigh, extract with hot 30% ammonium acetate, ignite the crucible, and take the loss in weight as lead sulphate.

**Manganese.** The filtrate from the first precipitate of lead sulphate is treated with about 20 c.c. of strong nitric acid, cooled, and oxidised with sodium bismuthate in the cold. In this way, the presence or absence of manganese is determined. If the colour of the permanganic acid indicates only a few hundredths per cent. of manganese, the solution is filtered through a Gooch crucible packed with asbestos and titrated with N/20 ferrous ammonium sulphate in the usual way. Should the manganese exceed 0.1% it is best to determine it on another portion, since if present in more than slight traces, it tends to dissolve in the soda. For an accurate estimation of manganese 1.1 grms. of alloy is treated with 30 c.c. of 10% soda as above till evolution of gas ceases, and nitric acid added till the solution is acid and all the metal has dissolved. Then another 20 c.c. of concentrated nitric acid is added, the solution is boiled and cooled, and the manganese determined after oxidation with bismuthate.

**Main analysis.** While the lead estimation is being carried out, the main analysis is started. One gm. of the metal drillings, preferably not too fine, is weighed out into a 400 c.c. tall beaker without a lip. 10 c.c. of the nitro-sulphuric acid solution is added and the beaker covered with a well-fitting clock glass. The dissolution of the metal is started by placing the beaker on the hot plate. When the action has begun the beaker is removed from the plate and reaction allowed to proceed. The action becomes rather violent and then subsides somewhat. At this stage a further 15 c.c. of the nitro-sulphuric acid is added and the dissolution is allowed to proceed fairly rapidly, keeping the beaker well covered and warming if the action shows signs of slackening. The last pieces of metal are brought into solution by gently boiling the liquid. During this part of the operation the boiling should not be so vigorous as to remove all the nitrous fumes from the beaker. When solution is complete, the cover glass is moved slightly to one side of the beaker and evaporation continued fairly rapidly until most of the water and nitric acid has been expelled. The cover glass is then removed and the evaporation continued without boiling until fumes of sulphuric anhydride form and the liquid begins to become turbid. At this point a clean glass rod is introduced and the liquid is stirred, the beaker being moved about on the plate at the same time, to avoid superheating at any point and prevent bumping. When the contents are nearly solid, the beaker is removed from the hot plate and the stirring continued in order to break up the cake of sulphates and ensure complete solidification. The beaker is then replaced on the hot plate and allowed to fume for a few minutes to render silica insoluble, after which it is taken off, covered with its original cover glass, and allowed to cool.

The dissolution and evaporation are not so difficult to carry out as might at first appear. With a little practice the exact time to begin the stirring can easily be recognised. If stirred too early the mass will not solidify quickly, but will show a great tendency to bump, while if evaporation is carried too far, the mass grows solid very suddenly and bumps violently. The condition to be aimed at is a certain degree of super-saturation before stir-



ring. Four samples may be dissolved and evaporated to this stage in half an hour.

**Silicon.** When the beaker has cooled somewhat the cover glass and sides of the beaker are washed down with water and the mixed sulphates are stirred to a paste. About 60 c.c. of water is added altogether, and the sulphates are then dissolved by boiling for a few minutes on the hot plate. The solution is filtered through an ashless filter into a 250 c.c. squat beaker and the silica and silicon (together with some lead sulphate, if any lead is present in the alloy) on the filter are washed several times with hot water. The precipitate is ignited *gently* in a platinum crucible and weighed. The silica is volatilised (a) by means of a few drops of hydrofluoric acid and sulphuric acid, and the residue is ignited and weighed, the loss of silica being calculated to silicon. If the contents of the crucible indicate silicon, generally in the form of a black coating near the bottom of the crucible, the residue is treated with a few drops of hydrofluoric acid, nitric acid, and sulphuric acid, evaporated to dryness, again ignited, and weighed. This second loss is taken as silicon and the result added to the silicon first volatilised at (a) as silica. If the metal contains much lead the ignition of the silicon, silica, and lead sulphate should of course be conducted very carefully, not only to prevent damage to the crucible, but to avoid any possible conversion of the lead sulphate and silica into lead silicate.

**Copper.** To the filtrate from the silica, etc., in the 250 c.c. squat beaker 25 c.c. of nitric acid (sp. gr. 1.42) is added and the solution subjected to electrolysis, using a rotating cathode consisting of a weighed platinum gauze about 2.5 cm. in diameter and 3 cm. long, and a plain platinum disc about one inch square for anode. We use a current of 2–3 amperes. When the copper has been removed, *i.e.*, the solution has become colourless, the sides of the beaker and the cover are washed down with a little distilled water and electrolysis continued for another ten minutes. The cathode is then removed as quickly as possible and washed, the wash water falling into the beaker used in the electrolysis. It is then dipped in clean alcohol, dried in the steam oven and weighed, the weight of the copper deposit giving the copper content of the metal. Any lead or manganese oxides on the anode are also gently washed with water to avoid any loss of solution, but the deposit on the anode is rejected. It is not needed further, because lead and manganese have been separately determined as above.

**Tin.** The solution after electrolysis is saturated with hydrogen sulphide. The character of the precipitate reveals the presence or absence of tin. The precipitate is filtered off, catching the filtrate in a 400 c.c. tall beaker, and washed with acidified hydrogen sulphide water containing a little ammonium nitrate. If tin is absent the precipitate is ignited and the copper in it estimated by dissolving in a little strong nitric acid, adding excess of ammonia, and matching the blue colour produced by means of a standard copper solution containing one gm. of copper per litre. The "copper traces" thus estimated should not exceed 0.05%, corresponding to 0.5 c.c. of the standard copper solution. If tin is present, the precipitate, after washing as above, is extracted on the filter with a warm dilute sodium sulphide solution to remove the tin sulphide, and the small residue of black copper and lead sulphides washed with a little hot water. This residue is then ignited and the "copper traces" determined colorimetrically, the result being added to the copper determined electrolytically. The solution of sodium thioannate is acidified carefully with hydrochloric acid, saturated with hydrogen sulphide, and allowed to stand on the steam bath to

settle. The tin sulphide is filtered off and washed with slightly acid ammonium nitrate solution until free from chlorides. It is then transferred with the filter to a weighed porcelain crucible 4.5 cm. in diameter (wide form) and treated with about 1 c.c. of nitric acid (sp. gr. 1.42), and the mass evaporated to dryness on the steam bath. The residue is then ignited gently to burn the charred filter, and finally strongly, to convert it into tin dioxide, in which form it is weighed. It is found that a part of the "copper trace" is often dissolved by the sodium sulphide owing to the formation of polysulphide with the sulphur in the precipitate, but this copper which contaminates the tin oxide is easily determined colorimetrically as above, the object of the sodium sulphide treatment being to free the tin sulphide from lead sulphide, which remains wholly undissolved. If lead has been proved to be absent, the sodium sulphide treatment may be dispensed with.

To the filtrate from Group II. sulphides, 7 grms. of pure tartaric acid is added; the solution is then made alkaline with ammonia, and about 10 c.c. of ammonium sulphide solution introduced. The beaker is covered, and the precipitate allowed to settle on the steam bath. If nickel is absent from the metal, the precipitate consisting of zinc, iron, and manganese sulphides is filtered off and washed till free from tartaric acid, with an ammonium nitrate solution containing a little ammonium sulphide.

**Iron.** The mixed sulphides are dissolved in hot dilute hydrochloric acid, oxidised with bromine, and the iron and manganese precipitated from the solution by bromine and ammonia. A double precipitation is of course necessary. After this, a basic acetate separation should be carried out to separate the iron and manganese from one another, and then the basic iron acetate dissolved and reprecipitated from ammoniacal tartrate solution with ammonium sulphide. The ferrous sulphide thus obtained is ignited and weighed as ferric oxide.

**Zinc.** The solution containing the zinc should be rendered faintly acid with hydrochloric acid, and some ammonium chloride and about one gm. of microcosmic salt added to precipitate the zinc as zinc ammonium phosphate, which is filtered off, washed with water, ignited to zinc pyrophosphate, and weighed. If, however, the zinc is less than 1%, we consider it preferable to precipitate the solution with ammonium sulphide, filter off the zinc sulphide, ignite at the lowest possible temperature, and weigh as zinc oxide.

**Magnesium.** To the filtrate from the zinc and iron sulphides a solution containing 5 grms. of microcosmic salt is added, with vigorous stirring; ammonia is then added, with further stirring. The solution is then allowed to stand, preferably overnight, to precipitate any magnesium as magnesium ammonium phosphate. The precipitate is filtered off, redissolved in a little dilute hydrochloric acid, a little tartaric acid and microcosmic salt are added, and the magnesium reprecipitated by gradually adding ammonia and stirring; then ammonia in excess is introduced and the liquid put aside for about 2 hours, after which the precipitate is filtered off, ignited to magnesium pyrophosphate, and weighed as such.

It should be noted, however, that in this process, manganese if present to any extent is liable to contaminate the magnesium, and possibly the zinc precipitate. If this is found to be the case the manganese content of each ignited precipitate is determined by means of sodium bismuthate, and the necessary correction applied after calculating to  $Mn_2P_2O_7$  in the case of phosphate precipitates, or  $Mn_2O_3$  in the case of the zinc oxide precipitate.

If the alloy contains nickel, the procedure must be modified somewhat. Part of the nickel sulphide is precipitated with the alkaline sulphides, *i.e.*,

with the iron, zinc, and manganese, and to dissolve the precipitate completely, a little nitric acid as well as hydrochloric acid must be poured over the filter. The iron and manganese are separated from the zinc and nickel as before, with bromine and ammonia, and the nickel is separated from the zinc by means of 1% alcoholic dimethylglyoxime, in slightly ammoniacal solution. The nickel precipitate is filtered off, washed, and discarded, and the zinc determined as above. The other portion of the nickel will be found in the filtrate from the mixed alkaline sulphides. This is acidified faintly with acetic acid, hydrogen sulphide is boiled off, and a little dimethylglyoxime added to the solution, which is rendered faintly alkaline with ammonia and allowed to stand in a warm place for about 20 minutes. The precipitate of sulphur, nickel sulphide, and nickel dimethylglyoxime is filtered off and discarded. The filtrate is then made alkaline with about 20 c.c. of strong ammonia, and 5 grms. of microcosmic salt added as before in order to obtain the magnesium.

**Nickel.** The nickel is best determined on one gram. of the alloy by dissolving out the bulk of the aluminium and zinc with 40 c.c. of 10% soda, filtering off the insoluble metallic residue, and dissolving this in nitric acid. Group II. metals are removed by means of hydrogen sulphide, the excess of which is expelled by boiling. Four grms. of tartaric acid is then added to the solution, which is heated to 60° C., and an excess of dimethylglyoxime (1% alcoholic solution) added, followed by sufficient ammonia to render the liquid slightly alkaline. The precipitated nickel dimethylglyoxime is filtered off on a freshly prepared Gooch crucible packed with asbestos, washed with hot water, dried to constant weight in the steam oven, and the nickel estimated on the assumption that the precipitate contains 20.32% of nickel.

The whole process described above may appear lengthy, but it must be remembered that manganese and nickel are absent in the majority of cases, while magnesium is only present in traces except in special alloys; so that the time required for the analysis is considerably shortened. Nevertheless, the scheme is designed to cover all the metals likely to be found in types (1) and (2), and after going through the process it is quite safe to take aluminium by difference, as is usually done.

The method is applicable to every type of aluminium metal and the results are accurate. The chief difficulty, however, arises with metals containing a large proportion of zinc (type 3). In this case, the method occupies 16 to 20 hours. The reason for this is the time taken in the settling, filtration, and washing of the mixed sulphides of iron and zinc. If plenty of iron is present, the mixed sulphide does not filter badly, but it is slow work at best; while, if the iron content is low and the precipitate of a light grey colour it is most difficult to retain it on the filter during washing. The time required to filter and wash such a precipitate satisfactorily is about four hours.

We have therefore devised another method for alloys containing much zinc. Titration methods were not very promising and electrolytic methods were therefore resorted to. The only electrolyte that appeared suitable for the separation of aluminium and zinc was alkali hydroxide, containing aluminate and zincate. From such a solution, using a rotating cathode, it was found possible to precipitate about 0.2 gm. of zinc free from aluminium and only leave traces of zinc—less than a milligram—in the solution. Sulphates in the solution did not interfere with the zinc electrolysis, but nitrates or ammonium salts prevented the deposition. In applying this method to aluminium alloys, various difficulties were at first encountered. As nitrates or substances likely

to generate ammonia must be excluded, the addition of 25 c.c. of nitric acid to the solution as recommended above for types (1) and (2), before electrolysis of copper is inadmissible. The first trials for the complete analysis of aluminium alloys using the electrolytic method for zinc involved the removal of all Group II. metals by hydrogen sulphide. Another difficulty was the estimation of the iron. This was finally accomplished by boiling off the hydrogen sulphide after removing Group II. metals, oxidising the solution with a little bromine water, adding about one gm. of tartaric acid and then excess of sodium hydroxide solution. Under these conditions the iron, aluminium, and zinc remain in solution, and when the liquid is subjected to electrolysis, the iron and zinc are deposited together as a clean layer. Magnesium has no deleterious effect, although in this case the electrolysis is carried out with magnesium hydroxide suspended in the liquid. Quantities of magnesium up to 1% do not contaminate the zinc deposit. Manganese, however, shows a decided, and nickel a slight, tendency to be deposited with the zinc. These metals rarely occur in alloys of type (3), however, and even if the deposit is contaminated with them, the degree of contamination can easily be determined as shown below, and the necessary corrections applied. The iron-zinc deposit can be dissolved off the cathode in dilute sulphuric acid and the ferrous iron titrated with *N*/20 permanganate.

Numerous experiments were made in order to find the most suitable cathode for the zinc electrolysis. Platinum cannot be used as the surface is permanently spoiled by the zinc, while the use of copper makes it impossible to determine the iron by titration after dissolving the iron-zinc deposit in dilute sulphuric acid. A silver cathode overcomes this last difficulty, but the electrode retains a little of the zinc and cannot be ignited before use. Finally, a gold plated platinum electrode was used; this was in the form of the platinum gauze cylinder, 2.5 cm. in diameter and 3 cm. long, used for the copper electrolysis and upon which a coating of about 0.1 gm. of gold was deposited from a cyanide solution. The coating extended for about 1 cm. up the axial stem, i.e. above the top of the gauze. The iron-zinc deposit dissolves off this cathode more completely than off the other metallic electrodes, and seldom leaves more than 2 mgrms. of zinc, in which no iron can be detected. The electrode, after cleaning in dilute sulphuric acid, is slightly dark, but can be completely freed from the last traces of adherent zinc by immersing in hot nitric acid (sp. gr. 1.2) for a short time. Before use, these gilded electrodes are gently ignited and then weighed. They have proved very satisfactory in use.

In the course of investigations into the electrolysis of copper from sulphuric acid solutions containing no nitrates, or urea or other substances likely to generate ammonia during the subsequent zinc electrolysis, it was found that the copper deposit was of a dark brown or black colour and the result was generally about 0.1% higher than if nitric acid were present in the solution. It was noticed, however, that the blackening of the deposit only occurred at those parts of the cathode that were wholly immersed in the solution during the revolution of the cathode. The portion of the copper deposited on the upper part of the cathode, which was partly out of the solution during its rotation, remained bright. This led us to think that the atmospheric oxygen was keeping the deposit bright, and an experiment was performed with the anode placed beneath the rotating cathode to observe the effect of the anodic oxygen on the deposit. The result was that the copper deposit was bright at the top of the cathode, black in the middle, and dull red at the bottom, showing clearly that a mild oxidising agent was necessary to keep



the deposit bright. Moreover, as it was found that a trace of sulphur dioxide or a small crystal of sodium sulphite added to the solution greatly intensified the blackening of the copper, sometimes with the deposition of sulphur in the solution, it was concluded that the blackening of the copper and the high results during electrolysis in pure dilute sulphuric acid were due to the reduction of sulphuric acid and the deposition of a film of copper sulphide. After various trials, hydrogen peroxide was found to be the most suitable oxidising agent to use in order to get a bright copper deposit and at the same time avoid the presence of nitrates. With hydrogen peroxide the copper remains bright unless the time of electrolysis is prolonged unduly beyond that required for complete deposition of the copper. When the copper has all been deposited, the cathode hydrogen begins to reduce the hydrogen peroxide, and as soon as this is all destroyed, the solution becomes full of gas bubbles. There is then the usual tendency for the copper deposit to blacken. If this free evolution of gas is observed, the copper can be kept bright by the further addition of one or two c.c. of hydrogen peroxide solution.

*Method II.* The observations recorded above enabled us to devise a new and rapid method for the analysis of aluminium alloys, especially those with high zinc content. The process is as follows:—

*Lead and manganese.* Two grms. of the metal is treated with caustic soda and the residual metals examined for lead and manganese as described in the first method above.

*Nickel and magnesium.* One gm. of the metal is treated with 10% sodium hydroxide solution till action ceases, the insoluble metals are filtered off, and dissolved in nitric acid. Group II. metals are removed by hydrogen sulphide, the excess of which is boiled off. The nickel, if present, is precipitated from an ammoniacal tartrate solution by means of dimethylglyoxime, and is filtered off on a Gooch crucible and weighed. The filtrate from the nickel is precipitated with ammonium sulphide and any iron and zinc sulphides filtered off and rejected. The filtrate from the zinc sulphide is examined for magnesium, by adding microcosmic salt, as in the general method already described. Generally nickel is absent, and magnesium is not present except as traces.

*Main analysis.* One gm. of the metal is dissolved, as described in the general method above, in nitro-sulphuric acid and the silica and silicon filtered off. To the filtrate, occupying about 100 c.c. in volume, 7 c.c. of "20-volume" pure hydrogen peroxide is added, the liquid is well mixed, and the cool solution subjected to electrolysis, using the rotating gauze cathode previously referred to and a current of 2 ampères. Electrolysis is continued for 10 minutes after the solution becomes colourless, adding a little more hydrogen peroxide if necessary, to prevent evolution of gas. The cathode is washed down with distilled water, dipped in clean alcohol, dried at 100° C., and weighed. The liquid is saturated with hydrogen sulphide, filtered into a 400 c.c. squat beaker, and the sulphide precipitate carefully washed with warm water (ammonium nitrate solution must not be used), and then examined for traces of copper and for tin, by the method already described. The filtrate from the Group II. sulphides is now boiled to expel hydrogen sulphide, and a little bromine water added to oxidise any sulphur and ferrous iron. The solution is cooled, about one gm. of pure tartaric acid is added, followed by a 50% solution of pure sodium hydroxide,\* until the

alumina and zinc oxide at first precipitated have just redissolved. A further 20 c.c. of the 50% soda solution is added, the solution cooled to room temperature and made up to about 300 c.c. If the concentration is too great or the temperature too low, salts tend to crystallise out. At this stage magnesium hydroxide is precipitated and this forms a delicate test for minute quantities of magnesium in the alloy, since 1 mgrm. shows readily. The liquid is electrolysed, using the weighed gilt platinum rotating cathode, for half-an-hour with a current of 1.7 ampères (3 to 4 volts), then for half-an-hour at 2 ampères. Under these conditions the solution does not become very warm, and if the electrode is well immersed in the electrolyte, the deposit is of a light bluish grey colour with a small dark ring round the stem where the latter enters the solution. At the end of an hour the cathode is quickly washed with several changes of distilled water, dipped in alcohol, dried in the steam oven, and weighed. The weight of the deposit gives the total iron and zinc, together with a little nickel and manganese if these are present in the original metal. To analyse the deposit, the cathode is immersed in warm 30% sulphuric acid, the deposit dissolved off, and the clean cathode removed. If the alloy contains no manganese the resulting solution is titrated with N/20 potassium permanganate solution and the weight of iron in the deposit obtained. On the other hand, if manganese is present, the cathode solution is divided into two portions, the ferrous iron being titrated with N/20 potassium permanganate in one part, and the manganese determined in the other by addition of nitric acid, cooling, oxidising with sodium bismuthate to permanganic acid, and titrating with N/20 ferrous salt solution as usual. If the metal contains nickel, the portion of the cathode solution used for the iron titration is treated with about a gm. of tartaric acid, a few c.c. of dimethylglyoxime solution are added, and the solution made faintly alkaline with ammonia. The nickel dimethylglyoxime precipitate is filtered off, dried, and weighed, and the nickel on the cathode is thus obtained. The true weight of zinc in the cathode deposit is obtained by subtracting the combined weight of iron, nickel, and manganese from the weight of the deposit. We find, however, that nickel and manganese are very rarely present in the cathode deposit.

The alkaline solution left after the zinc-iron electrolysis is treated with a few c.c. of sodium sulphide solution, when traces of iron and zinc which may not have been deposited, soon show as sulphide. Should the sulphide precipitate amount to more than traces, it is filtered off, redissolved in dilute hydrochloric acid, and the iron and zinc separated and weighed. In most cases, however, the amount of zinc and iron not electrolysed is extremely small, much less than 0.1%. This is especially the case if the zinc is largely in excess of the iron, as is usual with metals of type 3, where the iron seldom exceeds 1% and the zinc is generally 9% or more. Recently, however, the authors have tried to apply the method to metals of type 2, when zinc is present as a constituent. In the majority of cases the electrolysis has succeeded in removing practically all the iron and zinc from the solution. Sometimes, however, from some unknown cause, the method fails with these alloys. The cathode becomes coated with a deposit containing a little zinc and all the iron, after which no further deposit of zinc can be obtained, but the rest of the zinc can be deposited on a fresh cathode. Alternatively the remainder of the zinc in this case may be precipitated by means of sodium sulphide.

So far as accuracy is concerned the processes here described leave little to be desired. The copper

\* Certain samples of "pure" sodium hydroxide have been found to contain a little zinc.

determined by electrolysis in the presence of hydrogen peroxide has been generally found to agree exactly with the results obtained with nitric acid in the solution and seldom differ by more than  $\pm 0.03\%$  on a 3% copper content. With reference to the iron-zinc determination, we consider that the results are better than those obtained by the ordinary separation of iron and zinc. The cathode deposit is almost identical in weight with the combined weight of iron and zinc determined by the method given for metals of type 2, but after carrying out the permanganate titration for the iron in the cathode deposit, the percentage of zinc is slightly higher, and of the iron slightly lower (generally of the order  $0.05-0.1\%$ ) than that obtained by the usual chemical separation and weighing. This may be due partly to the great difficulty of separating zinc completely from iron by chemical means, as is made plain by Withey's experiments (*loc. cit.*), or it may be due to slight oxidation of the ferrous iron during solution of the cathode deposit. To test this latter point 0.1718 gm. of pure zinc oxide, corresponding to 0.1380 gm. of zinc, was dissolved in dilute sulphuric acid, and 20 c.c. of a ferrous sulphate solution (containing 0.0101 gm. of iron per 20 c.c., as determined by reduction with sulphur dioxide, boiling off excess gas, and titrating with  $N/20$  permanganate) was added. After oxidation of the iron with bromine and making the solution to correct strength with tartaric acid and caustic soda, the iron and zinc were deposited by electrolysis. The deposit weighed 0.1486 gm., and the weight of iron, as determined by dissolving off in dilute sulphuric acid and titration with  $N/20$  potassium permanganate, was 0.0102 gm., leaving 0.1384 gm. as the weight of the zinc deposit. These figures, which are typical of many results, confirm the accuracy of the method of estimation. Moreover, when duplicate analyses of an alloy have been carried out, the combined weight of zinc and iron in the two cases has been found to be within 0.5 mgrm. on deposits weighing about 0.13 gm. Where the iron content has been determined in one deposit after solution in sulphuric acid by titration with standard permanganate, and in the other by precipitating the iron once as hydroxide from ammonia and ammonium chloride solution, twice as basic acetate, and once again as hydroxide, as recommended by Withey (*loc. cit.*), the two iron results are found to be in very close agreement.

We have made a number of experiments to determine the completeness of the precipitation of magnesium in the presence of zinc and aluminium by means of sodium hydroxide and tartrate, and find that as little as 1 mgrm. of magnesium is wholly precipitated under the conditions described above. The magnesium hydroxide may be filtered off before or after the electrolysis of the zinc solution, redissolved, and reprecipitated as magnesium ammonium phosphate from an ammoniacal tartrate solution. In the absence of nickel this method of determining magnesium is accurate.

We desire to express our indebtedness to Sir J. J. Dobbie, F.R.S., for permission to publish the methods and results in this paper.

#### DISCUSSION.

Mr. C. E. BARRS agreed with the authors that lead was often present in aluminium, more particularly so if the alloy contained zinc. The suggestion to use a platinum electrode coated with gold led him to inquire why a gold electrode could not be used and so save the expensive platinum. He also asked whether the authors had made any experiments on the direct estimation of aluminium.

Dr. R. SELIGMAN said that the specifications to which they were required to work nowadays demanded the absence of appreciable quantities of lead in alloys of the type under discussion, and

it therefore could not be assumed that the alloys would be free from this material. On the contrary, the vast majority of alloys which were made up with ordinary spelter contained the proportion of lead which the spelter carried, and it was only alloys which were made up from modern electrolytic zinc which were free from lead. It was, therefore, essential that these alloys should be tested for lead.

Dr. Fox, replying to the discussion, agreed with Dr. Seligman's remarks. A gold electrode could, of course, be used but platinum was more rigid. He knew of no short method for the direct estimation of aluminium; the only procedure was to estimate it in the usual way by going through the separations. The result was usually  $0.3\%$  or  $0.4\%$  too high.

## Newcastle Section.

Meeting held at Bolbee Hall on November 20th, 1918.

PROFESSOR P. PHILLIPS BEDSON IN THE CHAIR.

### THE RELATION BETWEEN MOLECULAR STRUCTURE AND THE ACTIVITY TOWARDS HYDROGEN SULPHIDE OF OXIDE OF IRON.

BY G. WEYMAN.

In recent years almost the whole of the purification of coal gas from hydrogen sulphide has been effected by oxide of iron. The sulphides of iron with which such purification is concerned are the sesquisulphide,  $\text{Fe}_2\text{S}_3$ , which is oxidised by atmospheric air to free sulphur and ferric oxide; the monosulphide,  $\text{FeS}$ , which is also oxidised to free sulphur and ferric oxide, and which at higher temperatures may combine with free sulphur to form the disulphide,  $\text{FeS}_2$ . It seems somewhat uncertain whether the disulphide when prepared in this way will further oxidise in air to give ferrous sulphate. Ferrous sulphate, however, is always present in the unoxidised sulphides, although at low temperatures the amount may be small. After oxidation the amount is certainly increased.

Purification of coal gas is carried on at temperatures somewhat above the normal, under which conditions the sesquisulphide is the chief product. L. T. Wright (J. Chem. Soc., 1883, 156) found that by treating the hydrate precipitated by ammonia from ferric chloride with hydrogen sulphide and treating the product with carbon bisulphide out of contact with the air, a variable amount of free sulphur was produced, which bore no relation to the total quantity of free sulphur obtained after oxidation.

Free sulphur is always present from one source or another, and disulphide is produced by local heating. Subsequent oxidation might account for part of the ferrous sulphate found. This reaction also results in the liberation of sulphur dioxide. In those cases where there is no excess of alkali present the oxide quickly becomes acid, and the activity falls off with remarkable abruptness. If the oxide is maintained in an alkaline condition, the ferrous sulphate formed is immediately decomposed, with the result that the full amount of ferric hydrate is liberated, and available for further use. Even in an alkaline condition the activity of the oxide falls off in course of time. In an acid condition the amount of iron rendered inert by the formation of ferric and ferrous salts, or as the disulphide, is not sufficient to account for the almost complete inactivity of the whole body of the oxide.



Gedel\* has pointed out the remarkable influence of traces of ammonia or acid on the absorption of hydrogen sulphide by oxide of iron, and has found that in the latter case the monosulphide (and later the disulphide) was formed; in the former case only the sesquisulphide. He could not find any monosulphide in ordinary works oxide, a fact he attributed to the presence of ammonia.

Although Lewis Thompson, in his "Chemistry of Gas Lighting," states that the activity of iron oxide depends on molecular structure rather than on hydration, it is generally considered that only hydrated oxide is active, probably owing to the fact that strongly ignited oxide is certainly inactive.

In order to determine the precise temperature at which hydrated oxide became inactive, differently prepared pure samples were heated in an open silica tube to various temperatures measured by a thermocouple inserted in the oxide, for two to three hours, and then cooled and examined for their absorptive power towards hydrogen sulphide. The latter operation consisted in passing dry hydrogen sulphide over a weighed quantity of the oxide in a weighed tube of which the outlet was connected to a weighed calcium chloride tube. The air was displaced before and after the experiment by dry carbon dioxide. Three hours was given for the absorption, by which time it was practically complete. No additional water was added in any case.

In all cases, except one, the oxide obtained after heating to 100°, 300°, 350°, 400°, 450°, 500°, 550°, 600°, and 650° C. gave in the cold practically theoretical absorption (62.63%, theoretical = 63.7%) calculated on the content of ferric oxide. The oxide obtained by heating to 650° C. did not turn black till 5 minutes after the hydrogen sulphide was passing at the outlet; it then became black suddenly, with considerable heat effect. Oxide heated to 700° C. only gave a partial absorption, the extent of which depended on the time of heating and ranged from 10% to 32% of hydrogen sulphide. The oxide heated to 800° C. absorbed less than 1%. In the exception noted above, the hydrate dealt with was obtained by precipitation of a fresh solution of ferric chloride by ammonia, and drying at 100° C. Dried at this temperature it had the maximum activity, but when heated to 250° C., just after losing the bulk of the water, it became incandescent, and left on cooling a black, lustrous, hard residue which was inactive. On further heating the colour changed to puce, but the oxide was still inactive. In all cases most, at any rate, of the water of hydration was given off below 300° C.

It is evident then that ferric hydrates dehydrated at as high a temperature as 650° C. may still retain their activity in the cold. At about 700° C. a change sets in which may also be effected at lower temperatures by very prolonged heating. The sulphide formed in the above cases was entirely ferric sulphide, and oxidised in air with sufficient rapidity to set fire to the liberated sulphur. A determination of the free sulphur in one case in the unoxidised sulphide gave only a trace of sulphur when the quantity taken was 10 grms. This shows that under certain conditions, and with particular varieties of oxide, the absorption of hydrogen sulphide may take place in the comparative absence of water in accordance with the equation  $\text{Fe}_2\text{O}_3 + 3\text{H}_2\text{S} = \text{Fe}_2\text{S}_3 + 3\text{H}_2\text{O}$ .

The absorptions noted above were made at atmospheric temperature, but if the oxide is treated with hydrogen sulphide at higher temperatures the reaction takes a different course. In this case, the sulphide or sulphides formed are only attacked

by dilute acid when heated to boiling point, and then liberate hydrogen sulphide. No ferric oxide is formed on oxidation but only ferrous sulphate; the latter compound is also present in the unoxidised powder.

Even the oxide heated to 800° C. and which was inactive in the cold absorbed the gas with avidity at the higher temperatures. This indicated that the change in structure which inhibits the action of hydrogen sulphide in the cold must take place on cooling, and that at 800° C. the oxide molecules are in such a mobile state that slow cooling gives them an opportunity of arranging themselves in a more condensed state. To obtain further evidence of this, some oxide was heated for three hours to 800° C. and all but a check portion was plunged suddenly into cold water. The check sample, which was cooled slowly, absorbed less than 1% of hydrogen sulphide in the cold, while after drying the quickly cooled sample absorbed 43%. With a better cooling arrangement it would seem probable that the absorption would be more nearly theoretical.

An attempt to demonstrate a heat effect by cooling and heating curves failed.

The specific gravities of the powders obtained by heating to various temperatures and cooling slowly were determined in pure toluene. They showed a progressive rise; for instance after heating to 300° C. sp. gr. = 4.207; to 510° C., 4.444; to 700° C., 4.485.

To avoid possible confusion it might be mentioned here that "burnt oxide" from the manufacture of sulphuric acid is in a similar condition to the oxide heated above 700° C. It has been and is used for gas purification—not so much on account of the ferric oxide, as by reason of a varying content of ferrous sulphate, which is converted into ferric hydrate by addition of an alkali and subsequent oxidation before use. Unfortunately the burning of the material is often carried to such a point that little or no ferrous sulphate is left, and the material is worthless from this point of view. It is true also that the amount of ferrous sulphate necessary to make it of value is relatively small because with revivification *in situ*, which is now universally the practice, the same oxide may be alternately fouled and oxidised many more times than used to be commercially feasible.

The above considerations lead to the idea that the chemical activity of iron oxide is dependent primarily on molecular structure, and not on any particular degree of hydration. At the same time the molecular structure is evidently derived in almost all cases from some form of hydrate.

Passing, then, to the question of the constitution of the ferric hydrates, I hope to show that the conclusions arrived at by former investigators admit of a very close application and find some confirmation in the study of gas purification.

Péan de St. Gilles,\* studying the action of boiling water on hydrated ferric oxide, found what he considered to be a soluble modification. Graham, later, subjected solutions of ferric salts to dialysis and succeeded in getting a similar result, namely a solution containing very little else except ferric oxide. Magnier de la Source and others prepared solutions containing even less of the acid radicle, but still a small quantity was invariably left. Bechamp,† after pursuing the matter still further and taking into consideration the facility with which ferric oxide dissolves in ferric chloride, decided that there were formed true compounds of ferric oxide and hydrochloric acid of a more condensed type, which he considered were basic oxychlorides. Lewis T. Wright,‡ in preparing

\* Ann. Chim. et Phys., 1856, [3], 46, 47.

† Ann. Chim. et Phys., 1859 [3], 57, 296.

‡ Loc. cit.

\* J. Gasbeleucht., 1905, 48, 412, 428; this J., 1905, 668, 678.

ferric hydroxide in order to study the action of hydrogen sulphide upon it, noticed the difficulty of getting the precipitate free from chloride, and found that after washing practically free from chloride a further quantity was obtained after drying; moreover, under certain conditions, the ferric oxide became so finely divided that it passed through all filter papers but yet was not in true solution. Wright observed the different colours of ferric hydrate prepared in different ways, and quotes Tommasi,\* who obtained a soluble modification by keeping ferric hydrate (presumably containing some chloride) under water for a year. Tommasi also found that at the same time 30% of his sample was insoluble in dilute acid, some was unaffected by hydrogen sulphide, and some again was only altered by this gas after ten minutes' contact when it suddenly "flashed black." Wright suggested that the soluble modifications were only basic salts, and states that ferric hydrate when washed and dried at 100° C. becomes converted into an inactive form. Further, by treatment of the hydrate suspended in water with hydrogen sulphide and subsequently dissolving out the sulphide with potassium cyanide, Wright was able to estimate the proportion of the two types. The study by Wyrouboff and Verneuil† of the oxides of the rare earths and the subsequent application of their results by Nicolardot‡ to the ferric hydrates shed a new light on the matter.

Nicolardot pointed out that the solutions of the ferric hydrates in ferric chloride are strongly acid and decompose carbonates. They can therefore hardly be basic chlorides. He succeeded in preparing a number of the compounds and in drying them over sulphuric acid to constant weight, in spite of the fact that Van Bemmelen did not thus succeed, and proved instead by the vapour pressure curves that no definite hydrates could exist. Van Bemmelen, however, was working with a mixture of very similar substances, each of which there seems no reason to doubt would be quite definite if it could be isolated.

Nicolardot, after careful and persistent working, showed that the substance precipitated from ferric solutions may vary very largely according to the conditions, such as the freshness of the ferric solutions, the temperature, and the manner of adding the reagents. Such a precipitate contains varying quantities of different polymerised compounds, some more strongly condensed than others, each molecule containing a number of ferric oxide molecules bound together and acting as a unit combining with one equivalent of hydrochloric acid. These compounds may undergo further condensation on heating or simply on standing, and the condensed molecules in some cases may become depolymerised. Hydrates prepared in other ways, such as are formed when solutions of ferrous salts are precipitated by an alkali and allowed to oxidise in air, give other forms with substantially different properties. The condensation changes give well-defined heat effects. In short, the condensed hydrates are considered as derived either from the normal sesquioxide or from two different anhydrides in two different ways. Some are analogous to the pyro and meta acids, being condensed with loss of water, and are of one of the following types:  $[\text{Fe}_2(\text{OH})_4]_m - n\text{H}_2\text{O}$ ;  $[\text{Fe}_2\text{O}(\text{OH})_2]_m - n\text{H}_2\text{O}$ ;  $[\text{Fe}_2\text{O}_2(\text{OH})_2]_m - n\text{H}_2\text{O}$ . The others are condensed polymers without the elimination of water. They are of the types:  $[\text{Fe}_2\text{O}(\text{OH})_4]_p$  and  $[\text{Fe}_2\text{O}_2(\text{OH})_2]_p$ . Combining with different acids, the first give rise with elimination

of water to complex compounds—ethers, etc. In the latter a monovalent acid group is simply substituted for an hydroxyl group. Only the first and last terms of any series of the compounds are definite compounds. In the first the ratio of ferric oxide to monovalent acid is unity. The intermediate terms are mixtures of the first and last.

Nicolardot further describes six modifications which he isolated and examined, and gives their methods of preparation and properties. This work seems to be of very great importance, for not only is the formation of these condensed products substantiated by his experimental work, but it explains the observations of previous investigators and opens a new phase in the study of the process of gas purification.

The difficulty of freeing ferric hydrate from chlorides meets with an explanation in the formation of acid salts, and the so-called soluble modifications are nothing more than solutions of the salts of very condensed hydrates. Since the hydrates undergo polymerisation so easily and some of the modifications are inactive towards hydrogen sulphide, the trouble found in obtaining anything like a theoretical absorption is clear. The salts of the polymerised hydrates seem to behave with hydrogen sulphide in a similar manner to other iron salts, that is, they will form sulphides provided alkali is present to neutralise the acid set free. Wright's active and inactive oxides are simply different phases of condensation. Moreover, the different phases of condensation would be expected to influence the mode of chemical action, and it is quite possible that one type of compound which is especially susceptible to reduction would give ferrous sulphide on treatment with hydrogen sulphide, while another type would result in ferric sulphide being formed. It is curious that from a solution of a salt the product is uniformly ferrous sulphide. In the purifiers the oxide is, of course, in a solid condition, with, say, 30% of water. If the mass is allowed to become acid, the salt of a polymerised form would be capable of passing into solution, but in an alkaline condition the insoluble hydrate would not do so. Now Gedel found that under acid conditions only ferrous sulphide is formed, while I have pointed out above that when there was no water present and the oxide was neutral only ferric sulphide was produced. On the other hand Wright, working with a hydrate containing some chlorine, but which was dried at 100° C. and contained some water of hydration and was presumably neutral, obtained a mixture of the two sulphides. The acid conditions of Gedel, of course, presupposed that there would be sufficient acid to form a salt of a polymerised base, but not of an unpolymerised base. The action in this case is first one of depolymerisation, followed by reduction and sulphiding. The ferric sulphide would result from depolymerisation and then sulphiding.

The various types of hydrates all seem to approximate to a content of water represented by the formula  $\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$ , but vary 1% or so each way. I have never succeeded in drying any form exactly to that composition at 100° C., as Linder did. There is no doubt that changes in molecular condition slowly occur at 100° C., and this must account for the variation.

The behaviour of some of the different types of hydrate has already been dealt with, and it will be noted that they are not the same; in fact the difference in structure prevails at least up to 700° C., at which it seems that there is a tendency of the different types to merge into one another. Nicolardot points out, however, that some give off chlorine and some hydrochloric acid, and that the last traces of water may not be expelled even at 1000° C. At any rate most of the varieties at 700° C. have reached such a state that they cool to an oxide inactive in the cold towards hydrogen sulphide.

\* Bull. Soc. Chim., [2], 38, 152.

† Bull. Soc. Chim., 1899, 21, 137; Ann. Chim. et Phys., 1905 [8], 6, 141.

‡ Ann. Chim. et Phys., 1905 [1], 6, 331.



In further study it is evident that precautions should be taken to ascertain the type and degree of polymerisation of the oxide used. The hydrate produced by oxidation of ferric sulphide is not one of the crimson, reddish-brown, or dark brown varieties precipitated by alkalis from solutions of ferric salts; these as a rule become incandescent at about 280° C., and afterwards give a black, inactive, crystalline mass: this hydrate is a yellow or light brown substance similar to that formed by atmospheric oxidation of the hydrates precipitated from solutions of ferrous salts by alkalis. It is fully as active as the red varieties. Consideration of the formation of the natural ores used in gas purification (yellow ochre, bog iron ores, etc.) will show that these have been formed in a similar manner by precipitation under reducing conditions and subsequent oxidation. Further, the red hydrates after the first fouling and oxidation give a similar brownish-yellow type. This transformation seems to be accompanied by liberation of heat, which is added on to the heat of reaction. This means that the absorption is more rapid, but the amount of hydrogen sulphide absorbed in the end is the same.

It is evident, therefore, that gas purification is concerned with these yellow-brown types of hydrates, and it seems to me that further study on these lines will reveal important practical and theoretical results.

In conclusion I must express my gratitude to Mr. F. H. Walker for his help in preparing this paper.

#### DISCUSSION.

Mr. T. HARDIE said that, in his experience, the difficulties met with in purifying gas by means of natural bog ore were much less than when artificial oxides were used, especially with regard to reduction of efficiency due to acid. When using natural ore it was not difficult to revivify completely *in situ* so that the oxide showed up to 50% sulphur with a single change, whereas with artificial oxide it was difficult to get 50% even with four changes.

Dr. J. T. DUNN remarked on the interesting fact observed by Mr. Weyman that when the oxide was heated to 800° C. and allowed to cool slowly it absorbed practically no hydrogen sulphide, whereas if cooled by dropping into cold water it absorbed 40%. That appeared to be contrary to what one would have expected.

Mr. C. DRU DRURY said that, like Mr. Hardie, he had found considerable differences in the behaviour of the natural and artificial oxides, and his preference was certainly for the former. Artificial oxide was very active in the early stages, but there was considerable difficulty in "fouling up" to sufficient sulphur content in two changes, which they liked to do. The artificial oxide also became very hard and gave back pressure in the purifiers. He asked what percentage of moisture was recommended with artificial oxide to give the greatest activity. It appeared to him that the drier the oxide was, the more active it would be, but it was usual to moisten it slightly to prevent it falling through the grid.

Mr. F. H. WALKER, jun., thought that Mr. Weyman's paper showed the importance of keeping the purifying material in the boxes alkaline. The falling-off of efficiency might be due in the case of artificial oxide to the fact that a small quantity of lime was added to keep the material alkaline; the lime formed calcium sulphate, which caked the mass together, producing the back pressure to which Mr. Drury referred. The observation as to the behaviour of oxide heated to 800° C. and quenched was of very great interest, because, he thought, by that means "burnt oxide" might be made available for gas purification.

Dr. J. H. PATERSON remarked that magnesia heated in an arc furnace and cooled slowly was

inert to certain chemical reagents, whereas if dropped into paraffin and cooled rapidly, it was attacked readily by a number of substances which had no effect upon it in the ordinary condition.

#### ESTIMATION OF SULPHUR IN SPENT OXIDE.

BY WM. DIAMOND.

The estimation of the amount of sulphur present in spent oxide is customarily carried out by extraction with carbon bisulphide, and upon the figures obtained by this method are based the selling price of the oxide and also the calculation of the efficiency of working sulphuric acid chambers. It is obvious that the results obtained by this process of extraction are misleading, since carbon bisulphide extracts from the spent oxide other impurities, including cyanogen compounds, ammonia, tar, and oil, all of which are weighed and returned as "sulphur."

A method of removing the tarry matter from the oxide before extraction by treatment with sulphuric acid has been suggested, but this appears to be unsatisfactory in several respects. Particulars of a series of experiments on this method of acid treatment have been communicated to me. The results were as follows: Dried spent oxide was heated on a water bath overnight with sulphuric acid of various strengths, up to 40%; the products were not satisfactory. When the pure acid (100%) was used, the product was clean but not entirely satisfactory; also it remained moist, and the subsequent extraction with carbon bisulphide was incomplete. The use of sulphuric acid containing 10% of free SO<sub>3</sub> resulted in the production of sulphur of satisfactory colour. To obviate the difficulty due to the presence of free acid after this treatment, the most satisfactory procedure seems to be to moisten the material, add magnesium oxide, and dry before extraction.

It has been suggested that when oxide is treated with acid, high sulphur figures are obtained owing to the decomposition of thiocyanates, but experiment showed that this was not the case. An attempt was also made to render the tarry matter insoluble by treatment with sulphur dioxide, but the material became so wet that the figures obtained on subsequent extraction with carbon bisulphide were very low.

*Proposed method.* According to Mendeléeff, sulphur is soluble in carbon bisulphide in practically all proportions, but is only slightly soluble in cold benzene. It occurred to me that by washing dry spent oxide with several portions of benzene, all the tar and oil would be extracted, leaving only the sulphur to be extracted by carbon bisulphide. I therefore had several samples treated in this manner. About 10 grms. of the oxide is placed in an extraction thimble, and nine successive portions of 20 c.c. of cold benzene are poured over the sample, each portion being allowed to filter through the oxide before adding a further portion. A number of tests showed that nine extractions allowed an ample margin. The whole extract is divided into two portions: the first is evaporated to dryness, and the residue weighed; the second is evaporated to dryness, boiled down with nitric acid, water is added, and the sulphur precipitated by barium chloride, and weighed as barium sulphate. The first estimation gives total tar, oil, and soluble sulphur compounds, the second represents the sulphur only; therefore if the second is now necessary to extract the sulphur from the represent tar, oil, and soluble organic matters. It is now necessary to extract the sulphur from the same sample with carbon bisulphide in a Soxhlet extraction apparatus as usual. The weighings will give: 1. Tar, oil, and organic matter soluble in benzene. 2. Sulphur compounds soluble in benzene. 3. Sulphur extracted by carbon bisulphide.

In order that there should not be any dispute as to allocating the soluble sulphur, it is suggested that the proportion (thus found) be added to the sulphur found by extraction with carbon bisulphide.

In one test of a spent oxide, extraction with benzene gave residues of 14.18 and 13.37% in duplicate tests; the amounts of sulphur found by weighing as barium sulphate were 9.17 and 8.22%; thus the percentages of tar and other soluble matter were 5.01 and 5.14%, average 5.07%. The amounts extracted by carbon bisulphide from the original oxide were 49.87 and 49.79%, average 49.83%; so that the actual sulphur content of the sample was 44.76%.

#### DISCUSSION.

Dr. J. T. DUNN said that he thought Mr. Diamond was working on the right lines. The plan they had always adopted in his laboratory to determine the sulphur had been, after the extraction by carbon bisulphide, to oxidise a part of the extracted sulphur with nitric acid and determine the sulphate produced as barium sulphate, but his assistant, Mr. Redpath, had some time ago begun work to try to find a solvent that would take out the tarry matters and leave the sulphur. Benzene, as Mr. Diamond had shown, did take out these other substances but it also extracted some of the sulphur. Taking eighteen determinations as to the relative constancy of the proportion of impurity contained in the sulphur extracted, in the highest of these, the pure sulphur formed 98% of the total extracted by carbon bisulphide; the lowest, which was quite exceptional, was 93%, but, in the great bulk of them, the percentage of pure sulphur in the carbon bisulphide extract ranged between 96 and 97% and the average of the whole 18 was 96.4%. From these figures, it would seem that, if the oxide were extracted with carbon bisulphide, and then it was assumed that the total sulphur was 95% of that extract, one would, in nearly all cases, be very near the truth. Still, it would be more satisfactory to have a method of extracting the pure sulphur directly.

Mr. A. TROBRIDGE said that some years ago he had carried on some experiments on the estimation of sulphur by combustion and found that that method gave results which were, on the average 4% lower than by the carbon bisulphide method. He had always considered that the tarry matter led to the use of excessive amounts of sodium nitrate in the sulphuric acid process.

Mr. J. H. WALKER, jun., said that he had found that a considerable amount of sulphur dioxide was produced by roasting the residue after extraction of spent oxide by carbon bisulphide.

The CHAIRMAN remarked that the modifications of sulphur differed very materially in their solubility in carbon bisulphide, some unstable forms not being readily dissolved. The combustion method appeared preferable for the estimation of sulphur in spent oxide.

### Sydney Section.

*Meeting held on May 15th, 1918.*

MR. B. J. SMART IN THE CHAIR.

#### SOME ADVANTAGES TO BE DERIVED BY THE UTILISATION OF WASTE ON THE SOUTH COAST OF NEW SOUTH WALES.

BY R. W. CHALLINOR.

(ABSTRACT.)

The greater part of the coking industry in New South Wales is concentrated on the South Coast; of the 952 coke ovens in operation in the colony in

1916, 552 are in the South Coast District (Illawarra), 252 in the Northern district (Newcastle, Wallsend, and Singleton) and the remainder in the Western district (Lithgow).

Nearly all the coal is coked in beehive ovens, the only by-product installation being one of 66 ovens at the Broken Hill Proprietary Steel Works. Of the beehive ovens, forty, erected at Corral in 1912, are utilising the waste gases under boilers; from the remainder, all the by-products are wasted.

The total production of coke in New South Wales in 1915 was 417,753 tons, of which about 20% was made at the Broken Hill works.

The author pleads for the substitution of modern by-product ovens for the present wasteful beehive ovens, giving figures showing the amounts of the various products which might be recovered. Greig-Smith in 1916 estimated the value of the coke produced in New South Wales in 1913 at £209,000, and that of the by-products which might have been recovered at £344,000. The author, making an independent calculation, estimated the loss of by-products through non-recovery at £1,000,000 in three years.

The installation of by-product coking plant in New South Wales would result in the recovery of ammonia, tar and its products, and render possible the establishment of the manufacture of dyes, synthetic drugs, and perfumes, whilst the coal and coke dust might be saved by briquetting. The waste heat at the South Coast works might be utilised profitably for the evaporation of sea water with a view to producing various salts and bromine. The waste heat from the quenching of the coke should also be utilised. By converting the surplus gas into electrical energy, possibilities are opened up of establishing electro-chemical industries such as the manufacture of alkali, bleaching powder, and chlorinated organic compounds, or of making nitric acid from the atmosphere and ammonium nitrate from this acid and the ammonia recovered from the coking process.

### Yorkshire Section.

*Meeting held at Queen's Hotel, Leeds, on October 21st, 1918.*

MR. W. MCD. MACKEY IN THE CHAIR.

#### SOME ASPECTS OF THE SCIENTIFIC GLASSWARE INDUSTRY.

BY F. W. BRANSON.

In a previous communication\* I gave in detail some analytical data as to the composition of practically all the types of important chemical glassware imported prior to the declaration of war and gave preference to a zinc-aluminium borosilicate glass, taking as a starting point for further investigations a Jena glass beaker obtained direct shortly before the declaration of war, the composition of which was:—SiO<sub>2</sub> 64.66%, Al<sub>2</sub>O<sub>3</sub> 6.74%, ZnO 10.12%, CaO 0.08%, MgO 0.13%, K<sub>2</sub>O nil, Na<sub>2</sub>O 7.21%, B<sub>2</sub>O<sub>3</sub> 11.14%, Fe<sub>2</sub>O<sub>3</sub> 0.10%.

Numerous analyses and experimental meltings made with proportions of zinc oxide varying from 4% to 10% indicate that a percentage of alumina equal to about 6.5% of Al<sub>2</sub>O<sub>3</sub>, and a percentage of ZnO of 7.5%—8.5% and of boric oxide (B<sub>2</sub>O<sub>3</sub>) about 7% will give a good glass if perfectly

\* This J., 1915, 471.



annealed, equal to all reasonable requirements for scientific work, provided the silica and alkali (or alkalis) are present in the best proportions. With regard to the presence of calcium oxide in glass similar to the above it has been suggested that if zinc oxide is reduced in a formula for resistance glass, lime is the best substitute.\* A good deal of experience has been obtained in manufacturing on a large scale in which the percentage of alumina used was similar to that in the general formula given for a good resistance glass, and it is clear that a proportion of about 6% of alumina gives increased strength to glass and, as pointed out by Wood, the presence of alumina in a batch mixture diminishes considerably the action of melted glass on the pots.†

The earliest satisfactory output on a manufacturing scale was obtained, so far as my knowledge goes, by the York Glass Co., Ltd., who repeated my laboratory experiments in their works, using cryolite as a part source of alumina. A zinc-aluminium boro-silicate glass obtained by the use of a good formula is not an expensive glass to make and does not devitrify. It has good working properties and is therefore a favourite one with workmen owing to its good plasticity over a wide range of temperature. It makes excellent glass tubing (especially thin glass tubing) for the manufacture of glass apparatus and ampoules. In addition to the above, a considerable quantity of aluminium boro-silicate and other types of scientific glassware of excellent quality is being made by several firms. Analyses published in 1917 of all the British glasses then made, by W. E. S. Turner and his colleagues (J. Soc. Glass Tech., 1917, 1, 196) show that all are better than any pre-war imported glassware and that three of the series are superior to the other British glasses; two of these appear to have been made from the same or a very similar formula to that previously referred to, whilst the third has a lower zinc content and lime is added in place of a proportion of the zinc.

A great aid to the industry would be the standardisation of hollow scientific glassware such as beakers and flasks as to shape, contents, weight, etc. There is at present no lead as to what the total content of say a nominal litre (or other size) beaker or flask should be, nor is there any official recommendation as to the range of sizes. Travers has suggested definite thicknesses and weights for certain apparatus,‡ and I have advocated that steps should be taken to give an official lead to the trade. I have in conjunction with Messrs. Douglas H. Baird and Frank Wood presented a report accompanied by a set of scale drawings of a complete series of beakers and flasks, which by permission of the Department of Scientific and Industrial Research (Sub-Committee on Glassware Testing and Standardising Glass and Optical Instruments) is available for publication, with a view to adoption by all British manufacturers. To give a single example as to unnecessary sizes:—40 plain beakers were listed by the principal British and Continental manufacturers in the years 1914 and 1915. This number has been reduced in the Report referred to, to 21, namely:—30, 50, 75, 100, 125, 150, 175, 200, 250, 300, 400, 500, 600, 800, 1000, 1250, 1500, 2000, 3000, 4000, 5000 c.c. In addition, spouted beakers were reduced from 33 sizes to 22, flasks (round or flat bottom) from 26 to 24, beaker flasks from 11 to 9, and distilling flasks from 19 to 13. If we are to resist successfully foreign competition after the war there should be no redundant sizes; the abolition of such would mean economy to manufacturers, distributors, and users; lower freight

charges would also be attained, and indeed economy secured in every direction.

I would suggest that a committee be formed to include one nominee each from the Society of Chemical Industry, The British Chemical Ware Manufacturers' Association, Ltd., The British Laboratory Ware Association, Ltd., and the Society of Glass Technology, and one other to represent the actual workers in the scientific glassware industry, to draw up a report showing what sizes are unnecessary or to make such alterations as may be necessary, with power to co-opt other members (not exceeding two) if thought necessary. At a later stage graduated and other instruments could with advantage be taken in hand by this committee. One problem, namely, the question of a standard taper for stoppers for burettes, gas analysis apparatus, etc., has been carefully considered by A. M. Rowland, who suggests an angle of about 9° (see J. Soc. Glass Tech., 1917, 1, 103).

With regard to graduated scientific glassware it is interesting to note that at the National Physical Laboratory steps have been taken to extend greatly the existing facilities for testing, stamping, and granting of certificates for approved instruments. If a nominal charge for testing British graduated glassware could be made, the manufacture and use of verified instruments would be greatly extended. The Ministry of Munitions has ordered that all clinical thermometers sold after Nov. 21, 1918, must bear the approved test mark of the Laboratory. Unless full use is made of the resources of the National Physical Laboratory it is feared that inaccurate apparatus will be manufactured (or perhaps imported) and supplied, the result being dissatisfaction.

In addition to the Class A tests (such as are used for clinical thermometers), a Class B grade of commercial accuracy (say with an increased tolerance of 50% compared with Class A) has been adopted, and if in the future it be found possible to have sub-stations in the provinces for testing the Class B instruments, a very much larger number of pieces of apparatus can be dealt with at a minimum of cost. That a necessity urgently exists for such tests is obvious from the inaccurate graduated instruments exhibited.

An interesting section of my research was that which related to annealing, as in the early stages of the manufacture of British chemical glassware considerable difficulties were encountered. I cannot emphasise too strongly the importance of adequate annealing as I have repeatedly found that glassware made from the same formula by different firms varied greatly as regards internal stress. A careful determination was therefore made as to the exact temperature for annealing without deformation a glass which approximated in composition to the general formula already given, and this was found to be 580° C. by means of an accurate pyrometer and Hilger's strain viewer.

Twyman\* has determined the variation of the viscosity of glass with temperature in the case of a variety of glasses and gives a formula which applies throughout the ranges of temperature of interest for purposes of annealing. Combining this formula with the expressions given by Maxwell for the disappearance of stress in a viscous body, it is possible (by measuring the viscosity of the glass at any convenient temperature by means of the apparatus) to find the annealing temperature of any sample of glass. Repeated trials show that the results so obtained are entirely satisfactory.† Having ascertained the correct

\* J. Soc. Glass Tech., 1917, 1, 16.

† J. Soc. Glass Tech., 1918, 2, 80.

‡ This J., 1918, 238 T.

\* J. Soc. Glass Tech., 1917, 1, 61.

† See also English and Turner, J. Soc. Glass Tech., 1918, 2, 90.

annealing point, an experiment was made to find whether any improvement resulted by re-annealing, and I wish to express my indebtedness to Messrs. John Moncrieff, Ltd., of Perth, for making a batch of glass on a manufacturing scale and annealing beakers made from it at a temperature of  $550^{\circ}\text{C}$ ., and after cooling, re-annealing them at the same temperature. These beakers were carefully tested for stress by the method already referred to and found to be "Well annealed" and "Perfectly annealed" respectively. Mr. A. Chaston Chapman reported that the re-annealed beaker was more resistant to chemical reagents than the "single" annealed beaker. Further research is desirable to decide if re-annealing is of any practical value.

In both peace and war the scientific glassware industry is of such great importance, as so many other industries are directly dependent on it and as it is certain that but for the stocks of merchants at the time of the declaration of war a very serious crisis would have occurred, that it should, I think, be regarded as a "Key industry" and receive state assistance to the fullest possible extent; liberal special grants being allocated to the National Physical Laboratory, Departments of Universities such as the Glass Technology Department of the Sheffield University, Research Departments of organised trades such as the scientific glassware industry, and technical institutions in which glass technology is a subject.

I wish to express my indebtedness to Mr. F. H. Branson for his assistance with numerous analytical and experimental operations.

#### DISCUSSION.

The CHAIRMAN said that in the main he agreed with Mr. Branson as regards standardisation, but there was always the possibility that if once they began to depend on other people for standardising their instruments the habit of standardising—and it ought to be a habit—might easily be lost. Personally he thought it was important that every worker should standardise his own instruments. With regard to clinical thermometers, it must not be overlooked that they were apt to change when in use. So long as it did not interfere with a man taking the responsibility of his own instruments he did not find serious fault with the suggestion, but personally he was rather against subsidisation by the Government.

Dr. W. B. DAVIDSON agreed with the Chairman that every laboratory should standardise its own instruments, whether of glass or other material, and no graduated instrument should be accepted without testing. It was their business to eliminate all sources of error as far as they could, and it was quite possible with care to get down to as low an error as 1 in 5000. He was quite in agreement on the question of stoppers—as to the taper or angle. That was one of the weaknesses of the glass industry at present—that it could not turn out stoppers in sufficient numbers which did not give a great deal of trouble. A good deal of trouble also arose from bad annealing, especially in regard to large glass tubes, funnels, etc. On the other hand, British glass manufacturers deserved credit for the excellent quality of the material they had turned out during the war.

Mr. F. W. RICHARDSON suggested that makers of pipettes should be urged to put only a small graduation mark on these instruments, which would not look so unsightly as the heavy marks at present used when supplemented by the analyst's own graduation marks.

Dr. W. E. S. TURNER said that he supposed no one would be averse to purchasing an accurate

instrument if it could always be relied on. It was because of their experience that manufacturers' marks were sometimes inaccurate that they had found it necessary to calibrate measuring vessels for themselves. Mr. English, whose method of rapidly calibrating pipettes was mentioned by Mr. Branson, was engaged in completing a similar method applicable to burettes. The method for pipettes was one intended to combine speed and accuracy.

Prof. J. W. COMB said that if the apparatus that Mr. English had devised would enable scientific workers to be supplied with instruments that did not need re-calibration, it would at any rate save time; but even so he considered that chemists should carefully test the calibrations supplied before finally accepting them as correct. Although British glass-makers had achieved considerable success during the war and a large amount of research had been carried out in this country, the difficulties of glass manufacture had not yet been completely overcome, and a great deal of experimental work would be required both in the laboratory and in the works before the manufacture could be said to have been put on the desired footing. With reference to some of the tests that had been carried out in relation to resistance of glass to reagents, he recalled an experience of his own some time ago in determining soluble silicates after heating certain mixtures, when he had come to the conclusion that neither the acid nor the alkali test was trustworthy alone and that it was necessary in order to obtain trustworthy results to follow the one by the other.

Dr. W. E. S. TURNER, referring to the composition of glass, said it appeared that the zinc-aluminium boro-silicate glass was one of the best on the market and emphasised the value of zinc as a constituent. This did not mean, however, that a glass equally good but not containing zinc could not be obtained. Under some conditions a much simpler glass could be employed, and he had in mind in particular a specimen of a simple kind imported from America which was as good in point of resistance to water and acid as anything we had. With regard to the difference in chemical resistance between partly annealed and well annealed glass, he would certainly have expected such difference to exist, if only by analogy with metals in the hardened and annealed states (although he was aware the analogy was imperfect). But all the results obtained in his own laboratory and all the results from other sources which had come to his notice failed to reveal any difference in resistant power due to re-annealing. He was afraid he could not attach much weight to the figures quoted by Mr. Branson in this connexion, and Mr. Branson had rightly advised caution. The Jena factory, where re-annealing was carried out with flue-gases containing sulphur dioxide, held that there was a considerable improvement in resistance to water. Tests with acid and alkali, however, carried out at the National Physical Laboratory, failed to bring to light any difference between the ordinary and the re-annealed glass. The question of standardisation of sizes of glass articles had been raised by Dr. Travers at the Annual Meeting of the Society at Bristol last July (this J., 1918, 238 T); but even before that the matter had received considerable attention by a sub-committee of the Department of Scientific and Industrial Research. Perhaps something would now be done to ascertain what sizes users generally thought most convenient.

Mr. BRANSON, in reply, expressed his agreement with Dr. Davidson with regard to the personal checking of important graduated instruments. With reference to English's method, the inventor



had informed him that with careful practice the working error was very small. He thought that no difficulty would occur in adopting Mr. Richardson's suggestion. With Prof. Cobb's remarks he was wholly in accord, as although a large amount of work had been done under difficult conditions, a wide field yet remained unbroken. Dr. Turner's remark on the new glass was another indication as to the value of the work of the department which he so well conducted.

## Communication.

### THE AGEING OF VULCANISED PLANTATION RUBBER.

#### PART II.

BY HENRY P. STEVENS.

In a previous paper (this J., 1918, 305 T) I dealt with ageing experiments on ordinary sheet and so-called "slab" rubber. The present communication contains the results of an exactly similar series of experiments on smoked sheet, smoked "slab," ordinary pale crêpe, and smoked crêpe. Reference should be made to the first paper for the scheme of the experiments.

The figures for the coefficients of vulcanisation determined at the commencement and conclusion of the ageing experiments were as follows:—

Hours vulcanised	(1) Smoked Sheet.		(2) Smoked Slab.		(3) Pale Crêpe.		(4) Smoked Crêpe.	
	Beginning of ageing period	End of ageing period	Beginning of ageing period	End of ageing period	Beginning of ageing period	End of ageing period	Beginning of ageing period	End of ageing period
2	2.66	2.57	2.82	2.92	2.01	2.26	2.11	2.25
3	3.06	3.04	3.37	3.53	2.86	2.66	2.75	2.80
3½	3.72	—	4.14	4.42	3.62	3.58	3.45	—
4	4.33	4.38	4.84	5.63	4.27	4.28	4.17	4.21
4½	4.87	5.51	5.54	6.16	4.86	—	4.92	5.11
5	5.68	6.34	6.27	6.80	5.94	—	5.57	5.88

These figures confirm the previous conclusion, namely, that the increase with age in the coefficient is more noticeable in the over-vulcanised samples. Thus, an analysis of the above figures and those given in the previous paper gives the following results:—

Average coefficients between	Average increase in coefficient during ageing period.
2 and 3 = 2.52	0.02
3 and 4 = 3.43	0.10
4 and 5 = 4.55	0.25
5 and 6 = 5.69	0.51

The curves for breaking strain and elongation are similar to those published in the previous paper. The maximum breaking strains are those obtained from tests made shortly after vulcanisation and correspond to coefficients of 4 to 5. Tabulating the results as above, we have

Average coefficients between	Maximum breaking strain.
2 and 3 = 2.52	1560
3 and 4 = 3.43	1930
4 and 5 = 4.55	2090
5 and 6 = 5.69	1790
over 6 = 6.56	1580

These figures show clearly that the maximum breaking strain is not attained without considerably overcuring the rubber. The diagrams show that all types of rubber, when cured to give a coefficient of

4—5, attain the maximum breaking strain within 10 or 15 weeks of vulcanisation, after which they rapidly lose strength. In a year or so they become hardened and perished. On the other hand, if the vulcanisation be carried only so far as corresponds to a coefficient of 2—3, there is obtained with all types of rubber a product which, although only of medium strength when freshly cured, improves on an average for about a year and then only very slowly loses strength, so that at the end of two years the rubber is still stronger than when first vulcanised.

In the first paper figures were given showing the correlation of the coefficient and the period during which increase in breaking strain takes place. If these figures are analysed and the averages calculated as above the following are obtained:—

Average coefficients between	Average period (weeks) during which increase in breaking strain takes place.
2 and 3 = 2.52	54
3 and 4 = 3.43	33
4 and 5 = 4.55	10
5 and 6 = 5.69	3
over 6 = 6.56	nil

If the various diagrams be compared, it will be noted that the position of the curves is dependent on the coefficient and not necessarily on the time of cure, showing that the coefficient is the essential index of the degree of vulcanisation.

The curves for the same coefficient for different types of rubber do not necessarily coincide, for it has been shown in a previous paper (this J., 1918, 280 T) that the load required to produce a given elongation in sheet rubber is greater than in crêpe rubber when both types are cured to give the same coefficient. The curves for elongation all slope downwards, showing a gradual reduction in the final length with the period of ageing. With low coefficients the reduction is very gradual; with coefficients over 5 the vulcanised specimens are perished before the full period of ageing is completed. The average figures are as follows:—

Average coefficients between	Percentage decrease in final length.
2 and 3 = 2.52	7.7
3 and 4 = 3.43	13.9
4 and 5 = 4.55	39.9
over 5 = 6.02	nearly 100

The elongations corresponding to low coefficients for ordinary sheet rubber given in the first paper show an upward tendency during the first few weeks. This was not noticed with the curves for the unrolled sheet, nor is it found in the elongation curves here reproduced. In fact the reverse tendency is noticeable in the curves for smoked slab and pale crêpe, the curves corresponding to the low coefficients falling a trifle more rapidly during the first few weeks than at a later stage. It is probable that these fluctuations are due to variations in temperature during the period of ageing, and that at a constant temperature the curves for low coefficients would slope uniformly from the commencement of the ageing period. The curves with coefficients under 3 are practically straight lines within the period of ageing. With higher coefficients the curve is steeper and the reduction in final length is accelerated in relation to the ageing period.

#### Conclusions.

These experiments illustrate the general instability of vulcanised rubber in contrast to the raw material. I have found that plantation rubber may be stored for ten years without showing any change in feel or appearance and, when vulcanised, gives results comparable with those yielded by the freshly

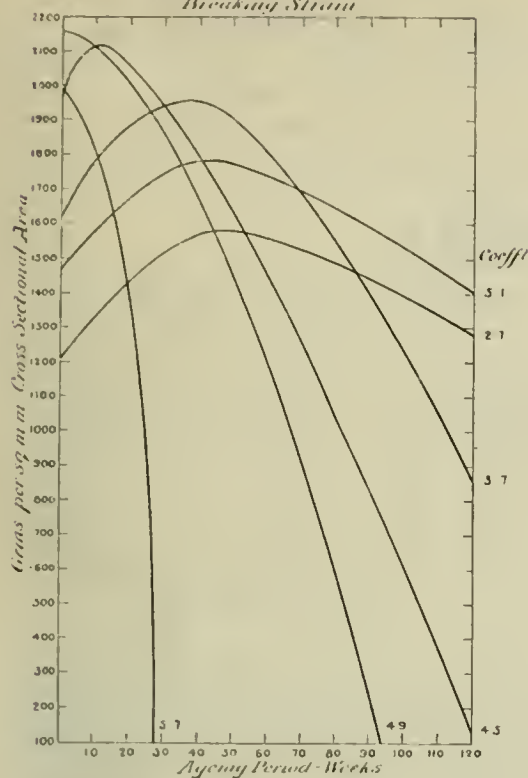
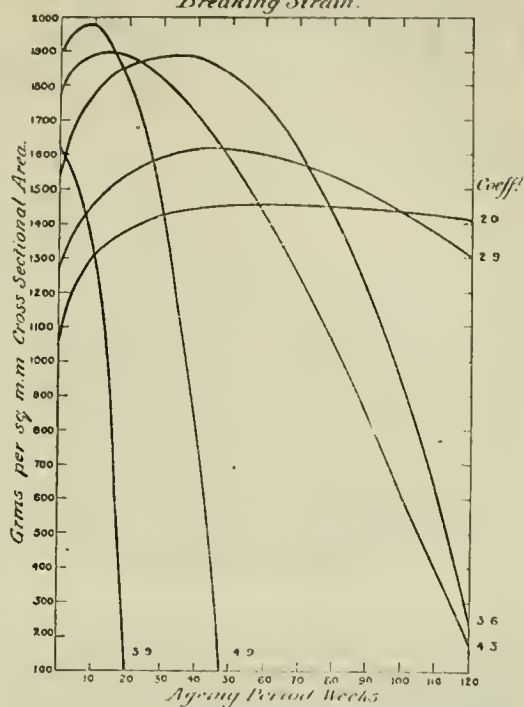
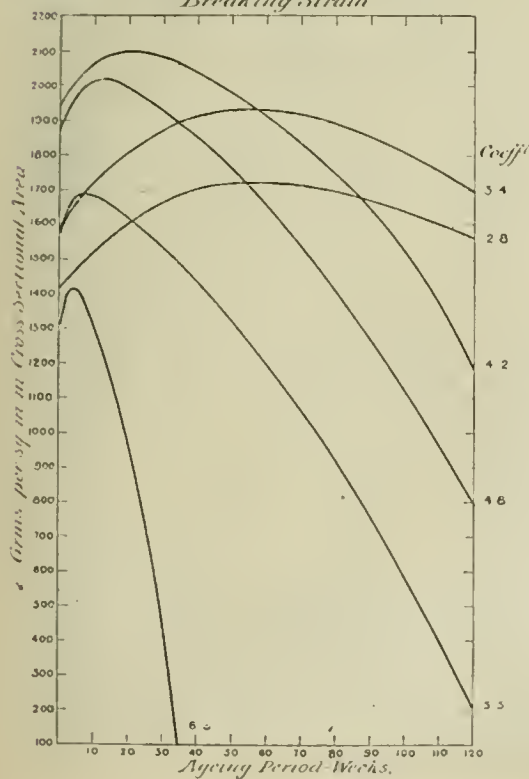
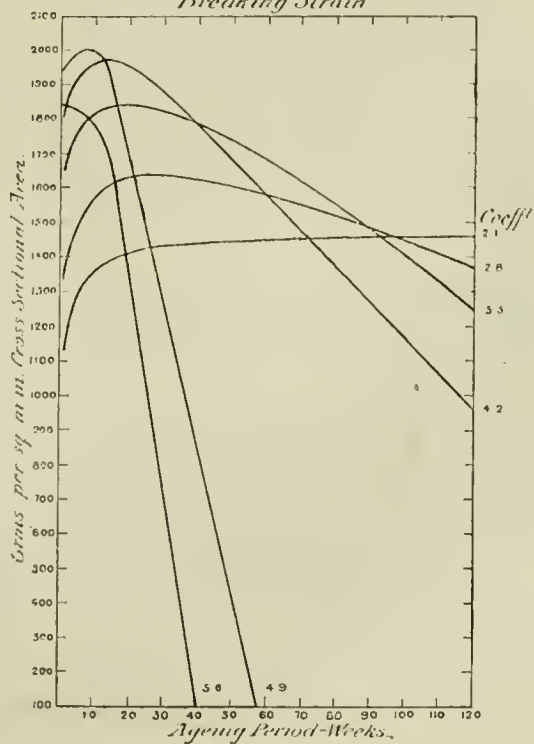
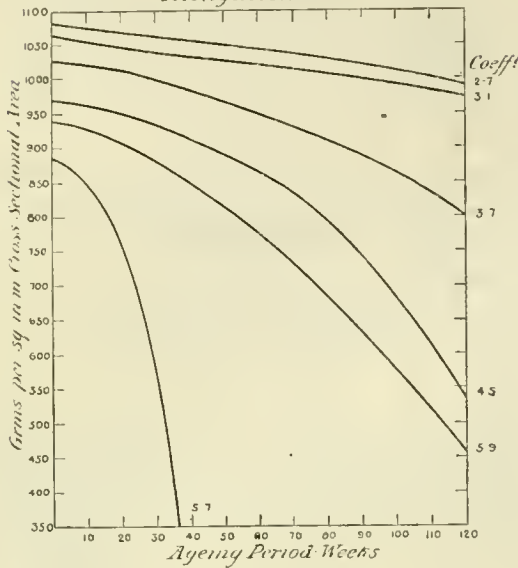
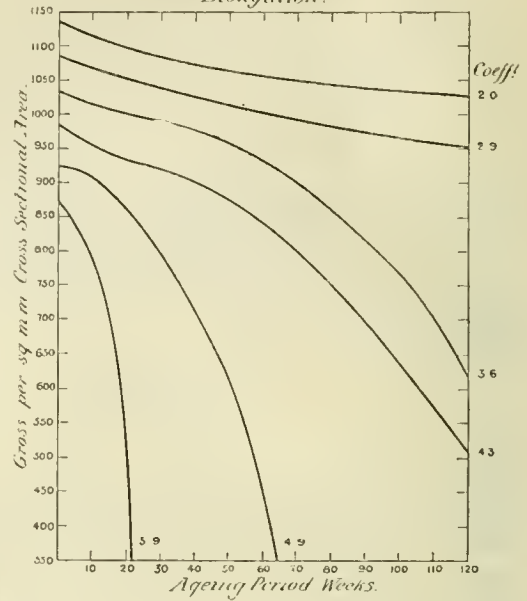
FIG 1 A Smoked Sheet  
Breaking StrainFIG. 3 A. Pale Crepe  
Breaking Strain.FIG 2 A Smoked Slab  
Breaking StrainFIG. 4 A. Smoked Crepe  
Breaking Strain

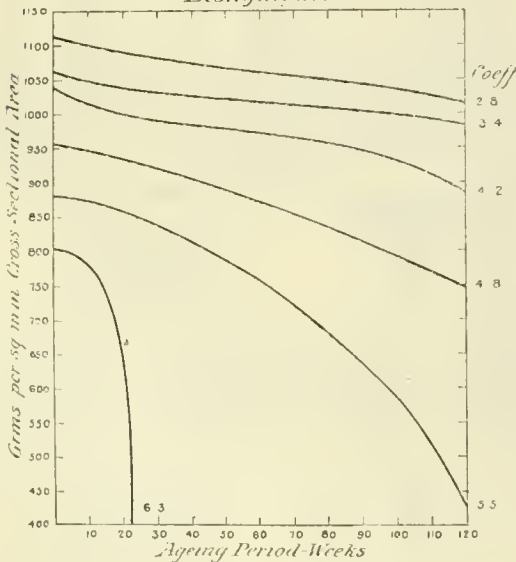
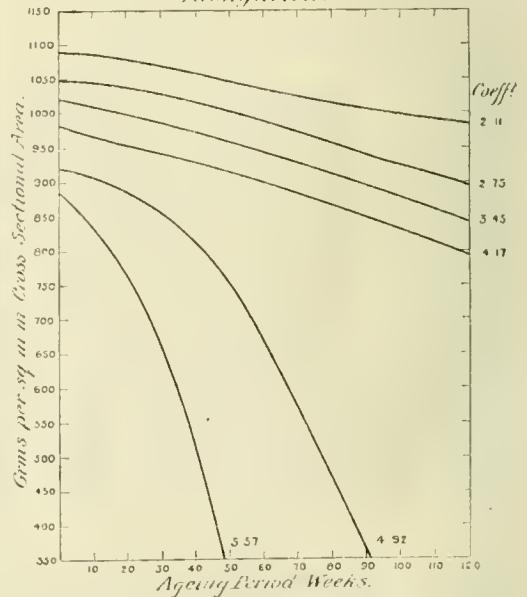


FIG 1.B. *Smoked Sheet Elongation.*FIG 3.B. *Pale Crepe Elongation.*

harvested raw material. On the other hand, vulcanised rubber, however carefully vulcanised, commences to change in physical properties from the moment the vulcanisation process is completed. When the original coefficient exceeds 3, appreciable deterioration takes place in 2 years. This may result in a loss of 50% of the tensile strength when the coefficient reaches 4, and complete "perishing" with a coefficient of 5 or more. Under the condi-

tions of the experiments it is apparent that a reasonably permanent product of primary vulcanisation can only be obtained when the coefficient does not exceed 3, and I regard this figure as a suitable standard for vulcanisation when comparing the physical properties of different specimens of vulcanised rubber.

I am indebted to the Rubber Growers' Association for permission to publish these results.

FIG 2.B. *Smoked 'Stub' Elongation.*FIG 4.B. *Smoked Crepe Elongation.*

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## ABSTRACTS

### I.—GENERAL; PLANT; MACHINERY.

#### PATENTS.

*Ammonia compressors [for refrigerating machines].* Apeldoornse Machinefabriek en Metaalgieterij voorheen L. Landaal, Apeldoorn, Holland. Eng. Pat. 111,101, Oct. 11, 1917. (Appl. No. 14,735 of 1917.) Under Int. Conv., Nov. 8, 1916.

IN a water-cooled ammonia compressor the outer frame surrounds part of the horizontal cylinder and supports it on two bearing surfaces, the frame between these points forming the outer wall of the water jacket at the middle portion of the cylinder. The cylinder is also supported in a registering recess near the cylinder cover, which is provided with a stuffing-box. The ammonia is supplied to the cylinder through a closed conduit passing longitudinally through the water jacket space on the upper side, and delivered through a similar conduit on the lower side. These conduits are controlled by valves accessible through openings in the outer frame. The inlet and outlet for the water jacket are arranged at such a height that the level of water in the jacket does not reach the suction conduit and surrounding parts which are cooled by the cold ammonia.—W. F. F.

*Graphite crucibles.* R. W. James, London. From E. Rietz, Sao Paulo, Brazil. Eng. Pat. 111,152, Nov. 11, 1916. (Appl. No. 16,295 of 1916.)

IN making graphite crucibles, 5 to 40% of compounds of the rare earth metals (e.g., thorium, zirconium) is mixed with the graphite. Zirconium compounds, especially the oxide, are found to be particularly suitable. A typical mixture consists of 10% of zircon earth, 70% of graphite, and 20% of clay, kiln brick, and sand. The crucible after moulding is baked at 1000° C.—J. H. P.

*Fire extinguishers; Chemical recharge for —.* G. J. Money, London. Eng. Pat. 111,403, Feb. 28, 1917. (Appl. No. 2969 of 1917.)

A CHEMICAL fire extinguisher comprises a hermetically sealed glass bottle containing carbon tetrachloride and dilute sulphuric acid of the same specific gravity. A more intimate mixture is thus obtained when the bottle is broken and the contents added to alkali in the usual manner.

—W. F. F.

*Furnaces for generating heat.* W. Thomas and A. E. Mainwaring, Nanaimo, B.C., Canada. Eng. Pat. 111,413, Mar. 29, 1917. (Appl. No. 4549 of 1917.)

IN a furnace suitable for burning low-grade or other fuel, such as mine waste, lignite, and the like, for heating steam boilers, bakery ovens, brick and pottery kilns or roasting furnaces, lime and cement kilns, or retorts, etc., the fuel is fed horizontally into one end of the upper part of the furnace chamber, where it is gasified and coked by poorer hot gases from the heated fuel which has settled into the lower wider part of the chamber. An exhaustor mounted above the

furnace draws combustible gas from the top of the furnace and delivers it through a special arrangement of flues in the furnace walls to an adjacent secondary combustion chamber, with which the lower part of the main furnace chamber also communicates directly. A blower, also mounted above the furnace, delivers air for combustion through heating tubes in the uptake from the secondary combustion chamber, and thence through another series of flues in the furnace walls to the secondary combustion chamber, where it is discharged through ports adjacent to the gas ports. A steam generator or the like is arranged above the secondary combustion chamber.

—W. F. F.

*Grinding mills; Separator for —.* E. J. Steckle, Dixon, Ill. U.S. Pat. 1,244,788, Oct. 30, 1917. Date of appl., Feb. 26, 1917.

A CYLINDRICAL separating chamber has a vertical inlet pipe extending into the lower end, through which a blast of air laden with the ground material is introduced. An outlet pipe is fixed in line with the inlet pipe in the upper end of the chamber, and a baffle which can be varied from the shape of a hood to an inverted hood is fixed between the pipes.—J. H. P.

*Grinding-mill.* J. B. Shackey, San Francisco, Cal. U.S. Pat. 1,245,625, Nov. 6, 1917. Date of appl., Mar. 23, 1914.

A GRINDING-MILL comprises a pan having an annular horizontal grinding track and a central vertical rotating shaft. The shaft carries a driving spider having vertical guideways sliding on the shaft, and separate members, carrying antifriction rollers at top and bottom, slide vertically in the guideways. Each member carries a short radial shaft on which a roller is mounted which bears on the grinding track, and the driving spider is supported from these shafts by springs.—W. F. F.

*Liquids used for separating solids of different specific gravity: Process of recovering —.* A. Nagelvoort, New York, Assignor to Virginia Haloid Co., Wilmington, Del. U.S. Pat. 1,244,884, Oct. 30, 1917. Date of appl., June 16, 1916.

THE process relates to the recovery of a liquid which has been used to separate solids of different specific gravities and which has a specific gravity intermediate between those of the solids. The separating liquid is removed by washing with an inert solvent of lower boiling point, at a temperature above the melting point of the separating liquid but below the boiling point of the solvent, and the solvent is evaporated.—J. H. P.

*Separating solids of different specific gravities: Process of —.* A. Nagelvoort, New York. Assignor to Virginia Haloid Co., Wilmington, Del. U.S. Pat. 1,244,885, Oct. 28, 1917. Date of appl., Sep. 1, 1916.

THE solids are separated in a solution of two



liquids of different specific gravities, each greater than that of water, the lighter liquid (e.g., carbon bisulphide) having a substantially lower boiling point than the heavier (e.g., a "relatively heavy salt"). The heavier constituent of the separant is removed from the separated solids by washing with the lighter liquid, and sufficient of the latter is distilled from the solution to leave a residue in the proportions of the original separating mixture.—J. H. P.

*Pulverising molten materials: Method and apparatus for —.* W. Freeman, Tacoma, Wash. U.S. Pat. 1,245,328, Nov. 6, 1917. Date of appl., Sep. 6, 1916.

A hollow tubular jet of gas is projected horizontally from a nozzle, a gap is formed in the upper side of the jet, and the molten material is fed in a freely falling stream into the gap, so that it is pulverised and conveyed by the jet.—W. F. F.

*Mixer; Continuous —.* T. B. Headley, Assignor to G. L. Headley, and Headley Good Roads Co., Philadelphia, Pa. U.S. Pat. 1,246,003, Nov. 6, 1917. Date of appl., Mar. 29, 1917.

FLUIDS to be mixed are fed into a long horizontal cylinder by a mixing nozzle or by supply pipes spaced along the cylinder. A discharge opening is provided at or near one end, and the fluids are fed towards the opening against the opposition of a mechanical conveyor and one or more throttling plates.—W. F. F.

*Furnaces.* London Electric Supply Corporation, Ltd., and A. G. Fox, London, Eng. Pat. 111,320, Nov. 17, 1916. (Appl. No. 16,515 of 1916.)

*Deflocculating solid materials: Method of —.* E. G. Acheson, New York, Eng. Pat. 111,434, May 19, 1917. (Appl. No. 7247 of 1917.) Under Int. Conv., Jan. 17, 1917.

See U.S. Pat. 1,223,350 of 1917; this J., 1917, 586.

*Refrigerating apparatus.* J. C. Bertsch, New York, U.S. Pat. 1,245,179, Nov. 6, 1917. Date of appl., June 24, 1915.

See Eng. Pat. 108,759 of 1916; this J., 1917, 1086.

## 11A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Carbonisation of coal.* F. D. Marshall, Gas. J. 1917, 140, 465.

WHILST the chief aim of high-temperature carbonisation is the production of a maximum yield of gas, the aim of low-temperature carbonisation is the production of an easily ignitable coke burning brightly with practically no smoke. In addition, from coals, cannel, shales, and lignite, quantities of oil may be obtained varying from 16 gallons in the case of colliery refuse to 87 gallons in the case of cannel. From these, petrols, burning oils, Diesel oils, lubricating oils, paraffin wax, tar acids, and a valuable bituminous pitch, can be obtained. The gas is "stripped" to recover additional petrol, and this, together with the petrol obtained by fractionating the crude oils, gives a total of 4 to 8 gallons of petrol per ton of coal. The gas after "stripping" is used as retort fuel. The above results are obtained by carbonising in iron retorts of special construction at a temperature from 900° to 1200° F. (480° to 640° C.) and under a vacuum of 10 to 20 ins. of mercury. The author and his colleagues claim to have established the fact that 5 in. is the maximum thickness of charge for low-temperature processes, and have on hand several large installations, in all of which retorts taking one ton per charge of four hours will be installed.—J. E. C.

*Light oils [benzol]; Recovery of — from coke-oven gas.* W. H. Wright, Amer. Gas Inst. Gas J., 1917, 140, 462.

THERE are at present 43 plants in the United States recovering light oils, with an approximate output of 110 million gallons as against 10 million gallons in 1913. The first attempt to recover benzol was made by Vogel in 1859, fatty oils being used for absorption with the idea of increasing their value for illuminating purposes. A patent was taken out by Carvès in 1884 for the recovery of benzol in connection with his coke ovens, heavy tar oil being used in an intermittent process. The first continuous plant of any size was built by Brunck in 1894. The general type of scrubber used in the United States appears to be of the tower form, from 6 to 24 ft. in diameter, and 40 to 90 ft. high, uniformly filled with wood grids. Experience has shown that it is desirable to have not less than three towers in a series, the wash-oil being circulated in counter-current to the gas, and in sufficient quantity to bring about a saturation of the benzolised oil to about 2 or 3%. Tar oil is used as an absorbent in Europe, and high-boiling petroleum oils in the United States. Plants at which still temperatures of about 135° C. are obtained show better wash-oil results than plants working at lower temperatures. The time of contact of the gas and oil in the majority of plants is from 70 to 100 seconds. The absorbing power of the oil commences to drop off very rapidly at about 25° C., so that it is important to keep the temperatures of gas and wash oil below this figure. In European plants the benzolised oil passes first through an oil-to-oil heat exchanger, then through a vapour-to-oil exchanger, whilst in America this sequence is reversed. Experience has fully demonstrated the desirability of the use of superheated steam. The author suggests the use of the single-pipe refrigeration ammonia cooler for cooling the wash oil, also that the cooling be brought about with the wash oil inside the tubes, and water outside, down to about 40° F. (4.5° C.), and the remainder of the cooling with oil outside and water inside. The reduction in heat value and illuminating power is given as 10 B.Th.U. and 2.3 candles respectively per 0.1 gall. of light oil removed per 1000 cub. ft.—J. E. C.

*Benzol-recovery plant operation; Laboratory methods for —.* P. W. Sperr, jun. Met. and Chem. Eng., 1917, 17, 548—555.

THE author describes the benzol recovery system of the H. Koppers Co., and enumerates the routine tests required in the operation of such a plant. To determine the light oil in the gas, a sample is drawn off from the main through a series of eleven wash-bottles and then through a meter. The connecting tube from the main to the first wash-bottle is heated by a steam-jacket to prevent deposition of naphthalene. The first wash-bottle contains strong caustic soda solution; the remainder contain wash oil and are kept below 20° C. throughout the test. When a suitable quantity of the gas has been passed through, the oil is emptied into a copper still and steam-distilled until about 500 c.c. of water has passed over, the oil in the still being maintained at 170°—180° C. The oil in the distillate is then separated from water, dried, and redistilled, the volume distilling below 200° C. being noted. To estimate the benzene, toluene, and solvent naphtha content, the oil is purified by successive treatment with sulphuric acid, caustic soda solution, and warm water, dried over solid calcium chloride, and fractionally distilled. A weighed sample is transferred to a distilling flask fitted with a fractionating column and distilled at a rate not faster than one drop per sec. to 130° C. (fraction "A"); the collecting flask is then changed and the distillation

resumed to 200° C. ("B"). "A" is then redistilled at the rate of one drop per 2 secs., "C" being the fraction collected to 95° C., and "D" the fraction to 120° C. "C" is again redistilled very slowly to 95° C. ("E"). Each sample is collected in a glass-stoppered Erlenmeyer flask and weighed. "E"—wt. of benzene content; "D" + ("C"—"E")—wt. of toluene content; "B" + ("A"—"C"—"D")—wt. of solvent naphtha content. Percentages by volume may be calculated by taking the sp. gr. at 15° C. of "E" for benzene, "D" for toluene, and "B" for solvent naphtha. The percentage of non-aromatic hydrocarbons in each fraction can also be calculated (Rittman and others: this J., 1915, 1980). This method of analysis of light oils gives good results (to within 1%) if the benzene content is at least 50%, and the toluene content not over 30%; if this is not so, pure benzene may be added to bring the composition of the sample within the limits. Other methods for the analysis of light oils are described briefly. The percentage of wash oil in light oil is determined by distilling 1000 c.c., at first dry to 120° C., and then with the introduction of steam, keeping the oil between 120° and 130° C. The residual oil in the still is cooled and filtered free from naphthalene. A blank experiment is run by distilling similarly 1000 c.c. of wash oil.—L. A. C.

*Solvent gasoline.* C. O. North. J. Ind. Eng. Chem., 1917, 9, 1038—1043.

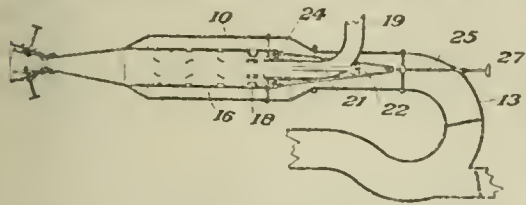
LABORATORY experiments are recorded on the cracking of heavy mineral naphtha, crude oil, and heavy kerosene for a solvent gasoline suitable for rubber manufacture. The oil was distilled under pressure from an iron still of about 1½ gallons capacity and the vapour passed through a cracker consisting of a gas-heated iron tube connected with a reflux condenser fitted with a steam jacket. The heavier vapours were then returned for further cracking, while the lighter passed forward to be condensed and collected. Gases formed were collected in a small holder. The deposition of carbon in the cracker was not excessive at the temperature of working (a dull red heat). Heavy kerosene gave the most satisfactory results. The yield of solvent gasoline (30°—130° C.) was up to 16.4% and of motor gasoline (30°—150° C.) up to 23%.—H. J. H.

*Utilisation of grape marc as fuel.* Matignon and Marchal. See XVIII.

#### PATENTS.

*Mixing powdered fuel and air; Apparatus for* —. H. A. Gill. London. From Powdered Coal Engineering and Equipment Co., Chicago, Ill., U.S.A. Eng. Pat. 111,412, Mar. 26, 1917. (Appl. No. 4322 of 1917.)

POWDERED fuel is supplied by the conduit, 19, to the perforated mixing chamber, 16, and air is supplied by the conduit, 13, to the annular



chamber, 10. The perforations in the chamber, 16, are punched inwards so as to form louvers and produce a number of air jets in all directions in the mixing chamber, and the openings, 18, at the rear end are made larger. When an additional supply of fuel is required, the feed may be aug-

mented by admitting air to the injector nozzle, 21, through the valve, 22. When an additional supply of air is required to produce a longer flame, it may be supplied by opening the annular valve, 24, by means of the screwed rod, 25, and handle, 27.—W. F. F.

*Coke-oven walls; Method of repairing* —. A. H. Chalmers, Fairfield, Ala. U.S. Pat. 1,214,467, Oct. 30, 1917. Date of appl., May 1, 1916.

IN a coke oven having vertical openings through the roof leading to combustion chambers in the walls, a beam is supported upon the two walls adjacent to the one to be repaired. A supporting member, suspended from this beam, passes through one of the aforesaid openings and by means of an attached plate supports the roof during repairs to the wall.—J. E. C.

*Burners for heavy oils for use in oil furnaces.* J. B. Steurs, Seraing-lez-Liège, Belgium. Eng. Pat. 111,249, June 5, 1917. (Appl. No. 8051 of 1917.)

PRIMARY low-pressure air is fed through an adjustable nozzle which passes through the oil supply chamber and regulates the flow of oil. The air and atomised oil pass into a mixing chamber and then through a nozzle into the furnace. Secondary high-pressure air is fed into a chamber surrounding the mixing chamber, and then passes through an annular opening around the nozzle, thus drawing the mixture of low-pressure air and oil into the burner.—L. A. C.

*Oils; Process of transforming — into lighter oils.* I. H. Wheat-roft, Oklahoma, U.S.A. Eng. Pat. 111,317, Nov. 6, 1916. (Appl. No. 15,868 of 1916.)

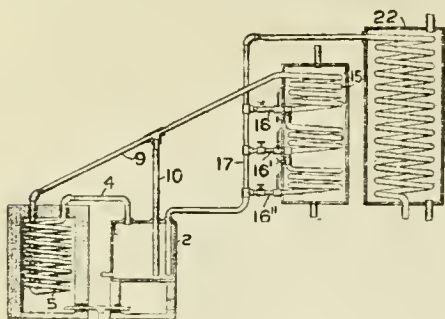
CRUDE petroleum or oil distillate is pumped at high speed through a heating coil of small diameter and great length in a furnace, where it is heated to 400°—450° F. (205°—232° C.), and thence at 100 lb. per sq. in. pressure through a jacketed tube heated to 800°—900° F. (427°—482° C.) by superheated steam from a coil in the furnace. At this point about 10% of superheated steam is injected into the oil. The oil then passes through a drum in the upper part of the furnace, heated to a temperature above 1000° F. (540° C.), and provided with a spiral to give the oil vapour a whirling movement. The heavier vapour is thus forced outwards and exposed to a higher temperature than the lighter vapour at the centre, thus causing a more uniform and complete cracking. The vapour then passes to a drum where carbon settles out, and thence to a cooler where the temperature is reduced to about 300° F. (150° C.). The oil gas and vapour then pass to a separator into which superheated steam is passed to raise the temperature to about 350° F. (177° C.), the resulting vapour is passed to a condenser, and the residue pumped back to the original supply tank for further treatment. The condensed liquid passes through a tank and is forced by a pump through a steam-jacketed heater to a steam still heated to 345° F. (174° C.). The vapour passes to a condenser and thence to a storage tank, and the residue is drawn off to a tank from which it may be passed through the first or second steps again as desired.—W. F. F.

*Still or retort [for cracking oils].* F. E. Wellman, Kansas City, Kans., Assignor to The Kansas City Gasoline Co., Kansas City, Mo. U.S. Pat. 1,245,291, Nov. 6, 1917. Date of appl., Feb. 23, 1915.

IN a low-pressure cracking system, the vapour space of a vaporiser, 2, is connected by a pipe, 1, to a superheating coil, 5, from which vapour passes upwards by an inclined pipe, 9, to the uppermost coil, 15, of a fractional condenser. Valves are provided between the units of the condenser and also on branch pipes, 16, 16', 16'', which



are connected above the condenser valves and to a common vertical vapour discharge pipe, 17, leading to the top of a condenser, 22. A drainage



pipe, 10, discharging into the liquid in the vaporiser, is provided on the pipe, 9, and a second discharge pipe is provided by the downward extension of the pipe, 17, as shown.—W. F. F.

*Cracking oils; Apparatus for* —. C. G. Lambert, Shreveport, La. U.S. Pat. 1,245,930, Nov. 6, 1917. Date of appl., July 7, 1917.

IN an apparatus for cracking oils, a heating pipe coated with heat-insulating material and containing electric heaters passes through the still chamber. A pump within the chamber forces the oil under high pressure through the heating pipe to a series of branch pipes in the upper part of the chamber. The heated oil is sprayed from these pipes by detachable sprayers into the vapour space, and gaseous products are withdrawn. Heavy condensed oil is returned to the still and the lighter vapour then condensed.—W. F. F.

*Coke ovens and the like; Doors for* —. H. Koppers, Essen-Ruhr, Germany. Eng. Pat. 108,306, June 29, 1917. (Appl. No. 9402 of 1917.) Under Int. Conv., July 22, 1916.

*Internal combustion engines; Scavenging of the cylinders of* —. P. Belyavin, Petrograd, and C. G. Robertson, Barrow-in-Furness. Eng. Pat. 111,162, Nov. 16, 1916. (Appl. No. 16,458 of 1916.)

*Furnaces for generating heat.* Eng. Pat. 111,413. See 1.

*Extraction of hydrocarbon products from shales and coals.* U.S. Pat. 1,244,840. See IIb.

## IIb.—DESTRUCTIVE DISTILLATION ; HEATING ; LIGHTING.

### PATENTS.

*Hydrocarbon products; Extraction of* — from shales and coals. D. T. Day, Washington, D.C., Assignor to V. Z. Reed, Denver, Colo. U.S. Pat. 1,244,840, Oct. 30, 1917. Date of appl., Dec. 1, 1916.

VAPOURS generated by the destructive distillation of coal or shale are condensed in a mass of similar coal or shale in a suitable container. The condensed material passes downwards through the mass, exercising a solvent action on the oil therein. The combined solvent and extracted matter is then recovered.—L. A. C.

*Electric arcs; Method of controlling and apparatus for producing* —. D. F. Comstock, and Technicolor Motion Picture Corporation, Boston, Mass., U.S.A. Eng. Pat. 110,982, Dec. 14, 1916. (Appl. No. 17,981 of 1916.)

A STREAM of magnetic flux is created in the region

of the gap between the electrodes, approximately longitudinally of the gap, but displaced angularly with respect to the axis of the gap, whereby the ionic discharge is distributed over the end of one electrode and a steady incandescent area is maintained on the end of the electrode in normal relationship to the axis of the electrode. The magnetic field is arranged so that the lines of force passing through the portions of the incandescent area on the anode furthest removed from and nearest to the cathode pass respectively through the central portion and laterally of the central portion of the emission area of the cathode, whereby the ionic discharge is so distributed over the incandescent area that the anode is consumed uniformly and the position of the incandescent area is maintained constant and asymmetric with respect to the cathode. The invention is specially applicable to the use of an arc carrying a heavy current for the production on one of a pair of electrodes of a steady incandescent area out of alignment with the other electrode, and suitable for projection apparatus and the like.

*Furnaces for generating heat.* Eng. Pat. 111,413. See 1.

## III.—TAR AND TAR PRODUCTS.

*Recovery of light oils [benzol] from coke-oven gas.* Wright. See IIa.

*Laboratory methods for benzol-recovery plant operation.* Sperr, jun. See IIa.

### PATENTS.

*Toluene [and propane from turpentine]; Process of producing* —. R. H. McKee, Crono, Me., Assignor to New Process Gasoline Co., Philadelphia, Pa. U.S. Pat. 1,244,444, Oct. 23, 1917. Date of appl., Jan. 18, 1916.

SPRUCE turpentine and aluminium chloride are heated to the reacting temperature, the liquid is distilled in presence of hydrochloric acid, and the toluene and propane produced are separated from the other reaction products.—F. W. A.

*Phenols; Process of making* —. H. Howard, Brookline, Mass. U.S. Pat. 1,245,343, Nov. 6, 1917. Date of appl., May 22, 1915.

IN a process of making phenols from the corresponding aromatic hydrocarbons, the phenol is liberated from an alkali metal phenolate by the action of an alkali bicarbonate.—F. W. A.

*Phenols; Process of making* —. H. S. Kimball, Newton, Assignor to Merrimac Chemical Co., Boston, Mass. U.S. Pat. 1,245,353, Nov. 6, 1917. Date of appl., July 24, 1915.

IN a process for making phenols from the corresponding aromatic hydrocarbons, the hydrocarbon is sulphonated, the sulphonic acid is neutralised by means of calcium sulphite, the calcium sulphonate is converted into the sodium salt by means of sodium sulphite, and the phenolate produced by treatment with caustic soda is decomposed by the sulphur dioxide liberated in the neutralisation.—F. W. A.

*Aminoalkyl esters and alkylaminoalkyl esters of p-aminobenzoic acid; Production of* —. W. Bader, and Levinstein, Ltd., Manchester. Eng. Pat. 111,328, Nov. 21, 1916. (Appl. No. 16,646 of 1916.)

p-AMINO BENZOIC alkyl esters are treated with an amino-alcohol or alkylamino-alcohol in presence of a small quantity (about 2%) of the aluminium compound of the alcohol. Example: 10 lb. of

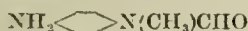
*p*-aminobenzoic ethyl ester, 10 lb. of diethylaminoethanol, and 4—8 oz. of the aluminium compound of diethylaminoethanol are heated to 150°—180° C., all the ethyl alcohol formed being allowed to distil off; the excess of diethylaminoethanol is removed by distillation at 27—28 ins. vacuum until the temperature of the liquor has risen to 170° C., when the residue is poured into cold water and neutralised with hydrochloric acid; any unconverted aminobenzoic ethyl ester remains undissolved and is removed by filtration. The diethylaminoethyl ester of *p*-aminobenzoic acid may be precipitated by caustic soda, and the hydrochloride forms a pure white salt. Instead of adding the aluminium compound, it may be formed in the mixture, e.g., by adding 2% of aluminium amalgam.—F. W. A.

#### IV.—COLOURING MATTERS AND DYES.

##### PATENTS.

*Intermediate products [methylformanilide derivatives] suitable for the manufacture of colouring matters; Manufacture of* —. H. Levinstein, Manchester, and G. T. Morgan, London. Eng. Pat. 111,321, Nov. 17, 1916. (Appl. No. 16,520 of 1916.)

*p*-NITROMETHYLFORMANILIDE (m. pt. 119°—120° C.), its substitution products (other than nitro derivatives), such as halogen or alkyloxy derivatives, and homologues, are produced by nitration at —5° to 20° C. of methylformanilide or its corresponding derivative having a free *para*-position to the amino-group. *p*-Aminomethylformanilide (m. pt. 115°—116° C.),



its substitution products and homologues are obtained by reduction of the corresponding nitro-derivatives by means of reducing agents which do not eliminate the formyl group, e.g., iron borings and dilute formic or acetic acid at 85°—100° C. These products may be used with advantage in the manufacture of dyestuffs, as the formyl group may easily be removed.

—F. W. A.

*Dye [compositions].* S. M. Tootal, J. Kershaw, J. Mather, and V. A. Tootal, Blackpool. Eng. Pat. 111,365, Dec. 15, 1916. (Appl. No. 18,025 of 1916.)

A COMPOSITION of a combined dye tint and finish, more particularly suitable for domestic purposes, consists of a mixture of dye with glue or gelatin in the form of a dry circular wafer, zinc oxide, ammonia, or sodium carbonate being added to prevent stickiness.—F. W. A.

*[Triphenylmethane] dye; Violet cotton* —. M. Weiler, Elberfeld, Germany, Assignor to Synthetic Patents Co., New York. U.S. Pat. 1,244,149, Oct. 23, 1917. Date of appl., Sep. 22, 1915.

DYESTUFFS are claimed, produced by sulphonation of a triphenylmethane compound and of the general formula:  $\text{RR}_1\text{C}(\text{R}_2)\text{NH}\cdot\text{R}_3$ , in which R and R<sub>1</sub> are aryl radicals of an aromatic hydroxycarboxylic acid, e.g., hydroxycarboxytoluene, R<sub>2</sub> is a divalent aryl radical of the benzene series, and R<sub>3</sub> is a sulphonated aryl radical. The products give violet shades when printed on cotton in conjunction with a chrome mordant.—F. W. A.

*[Sulphur] dyestuffs; Process for the production of* —. Z. Suzuki, Los Angeles, Cal. U.S. Pat. (A) 1,244,795 and (B) 1,244,796, Oct. 30, 1917. Date of appl., Oct. 30, 1916.

SULPHUR-CONTAINING dyes are obtained by heating (A) rice hulls or (B) tobacco stems with water and caustic alkali, adding sulphur and sodium sulphide, and continuing the heating until hydrogen sulphide has been evolved and until a dry mass is obtained.—F. W. A.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Sulphite pulps; Method of staining to distinguish between bleached and unbleached* —. C. G. Bright, Tech. Assoc. Pulp and Paper Ind., May, 1917. J. Ind. Eng. Chem., 1917, 9, 1044—1045.

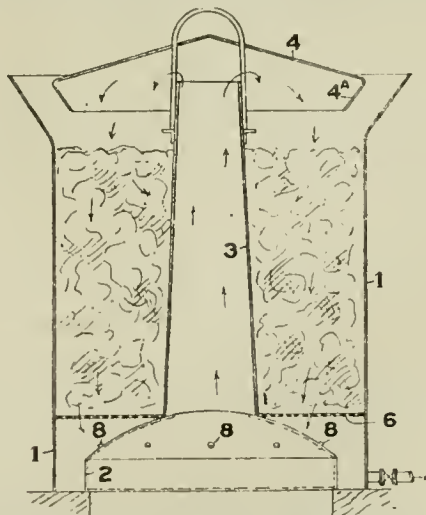
THE fibres are first stained with Cross and Bevan's ferric ferriyanide reagent which colours unbleached sulphite green on account of contained lignin, leaving the bleached pulp colourless. The contrast is further increased by subsequently staining with a red substantive dyestuff, when the green changes to a very pure blue and the bleached fibres are coloured red. The ferric ferriyanide reagent is produced by mixing equal volumes of *N*/10 ferric chloride and *N*/10 potassium ferriyanide. The slide carrying the fibres is dipped into this mixture for 15 minutes at 35° C. The red stain is prepared by stirring slowly into hot distilled water 0.4 gm. of Benzopurpurin 4 B. extra and 0.1 gm. of Oxamine Brilliant Red B.X. The immersion lasts 5 minutes. Fresh solutions are used in all cases.

—H. J. H.

##### PATENTS.

*Cotton waste or other fibrous material; Apparatus for cleansing* —. A. Poulson, Widnes, and W. C. A. Mate, Garstang. Eng. Pat. 110,691, Mar. 6, 1917. (Appl. No. 3276 of 1917.)

AN apparatus for cleansing cotton waste or other fibrous material, especially for use in the manufacture of explosives, consists of an outer tank, 1,



forming with a chamber, 2, channels for collecting the separated dirt. The cleansing liquid on heating circulates upward through tubes, 3, fitted at the top with a deflector plate, 4, having a return flange, 4a, and returns to the chamber, 2, by perforations, 6, in a diaphragm on which rests the material to be cleaned, and the projecting tubular nozzles, 8.—F. W. A.



*Cellulosic material; Preparation and treatment of — to render it suitable for use as a substitute for cork and the like.* G. E. Heyl, London. Eng. Pat. 110,941, Nov. 8, 1916. (Appl. No. 16,011 of 1916.)

FINELY divided material such as spent hops, ground wood, and other cellulose, with or without finely divided cork, is treated with a fireproofing salt, such as borax or a tungstate, and dried; it is then sprayed with a solution of rubber in naphtha or other solvent and moulded into slabs by passing between heated rollers.—J. F. B.

*Paper; Calenders or machines for imparting a tin-facced or like finish to —.* Grove Mill Paper Co., Ltd., and W. Porritt, Stockport, Cheshire. Eng. Pat. 111,024, Mar. 8, 1917. (Appl. No. 3411 of 1917.)

THE calender consists of two bowls, separately adjustable, with means for quickly raising and lowering the lower pressure bowl, and two endless linen or fabric bands passing partly over the bowls in the same direction. The endless bands are supported, one by rollers below the stillage and by rollers on vertical supports at some distance behind, and the other by rollers on horizontal supports above. Both the vertical and horizontal supports carry means for adjusting the tension of the bands, and scroll-rollers are situated near the calender bowls for opening out the upper and the lower bands. After the paper has passed through the calender between the two bands, it is conducted over steam-heated drying rollers and reeled up.—J. F. B.

*Paper and method of making the same.* E. F. Millard, Watertown, Mass. U.S. Pat. 1,244,116, Oct. 23, 1917. Date of appl., June 7, 1916.

PAPER consisting substantially of short (thin raw wood fibres is prepared mechanically from raw wood, and in a separate operation a pulp of relatively longer fibres is also prepared mechanically from raw wood. The two pulps are mixed in equal proportions to form a pulp mixture containing sufficient long fibres to loosen up the short fibres and facilitate the running of the pulp, combined with sufficient short fibres to impart strength and finish to the paper.—J. F. B.

*Paper pulp; Process of manufacturing —.* T. Marusawa, Fukuoka, Japan. U.S. Pat. 1,241,525, Oct. 30, 1917. Date of appl., Aug. 2, 1917.

VEGETABLE fibrous material is boiled with ammonium bisulphite, or a mixture of ammonium bisulphite with sulphurous acid or a soluble sulphite, an acidity of 0.1 to 0.8-N being maintained. The boiling may be carried out in two stages.—F. SP.

*Pulp and other material; Presses for expressing liquids and forming articles from —.* W. J. Norris, London. Eng. Pat. 111,327, Nov. 18, 1916. (Appl. No. 16,574 of 1916.)

*Artificial silk and like threads; Apparatus for treating —.* H. de Chardonnet, Paris. U.S. Pat. 1,245,690, Nov. 6, 1917. Date of appl., Mar. 3, 1917.

SEE Eng. Pat. 10,858 of 1915; this J., 1916, 320.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Cloth dyeing, bleaching, and like machines.* R. Jackson, Chinley. Eng. Pat. 110,715, May 24, 1917. (Appl. No. 7480 of 1917.)

IN a cloth dyeing, bleaching, or like machine, the

cloth, whilst held taut, is passed at a uniform speed below the surface of the liquor by means of two guiding rollers, and squeezed across its entire width before and after being passed through the liquor by means of squeezing rollers, one above each guiding roller; the motion for winding and unwinding is obtained from the squeezing rollers.

—F. W. A.

*Dye [compositions].* Eng. Pat. 111,365. See IV.

*Soapy liquors from laundries; Method of treating waste —.* E. W. Lucas, Ealing, Assignor to H. P. Denison, Syracuse, N.Y. U.S. Pat. 1,245,605, Nov. 6, 1917. Date of appl., Apr. 17, 1914.

SEE Eng. Pat. 10,280 of 1913; this J., 1913, 941.

*Fast tints; Process for producing —.* G. Engi, Assignor to Society of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,245,694, Nov. 6, 1917. Date of appl., Nov. 4, 1915.

SEE Eng. Pat. 13,204 of 1915; this J., 1916, 449.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid; Concentration of —.* P. Pipereaut and Helbronner, Monit. Scient., 1917, 61, 265—269.

THE concentration of dilute sulphuric acid, as usually carried out by means of hot flue gases passed in counter-current over the acid in a series of volvic stone vessels, has the disadvantage of causing considerable loss of acid in the vapours. This is due to the comminution of the acid and the formation, under the action of the hot blast, of a considerable amount of anhydride from the concentrated acid, which is only condensed with difficulty in water or weak acid. The authors therefore propose to reverse the usual process and pass the acid in a shallow undisturbed stream, through a series of twelve vessels of special design, in the same direction as the flue gases. Each vessel is in the form of a rectangular box, 1 metre long, 1.5 m. wide, and 0.5 m. high, provided with circular inlet and outlet, the latter at a lower level than the former, and keyed together. Sodium silicate is used to make the joints tight, and the whole is covered with sheet lead 10 mm. thick. The acid to be concentrated is introduced into the first vessel through a pipe in the side. Every alternate vessel is provided with a pipe through which any desired proportion of vapour may be led away for condensation. Gases at 1200° C. can be employed directly on acid of 53° B., and the proportions should be adjusted so that (a) the concentrated acid is not heated above 300° C., at which temperature anhydride formation begins, and (b) the gases leaving the system are still considerably below the point of saturation with water vapour. The advantages claimed for the system are economy of fuel, low acidity of the condensed vapours, reduced atmospheric pollution, and smaller wear and tear of the plant.—F. SP.

*Chlorides; McLean-Van Slyke iodometric method for the titration of small amounts of halides, in its application to —.* R. F. McCracken and M. D. Walsh. J. Amer. Chem. Soc., 1917, 39, 2501—2506. (See this J., 1915, 608, 1033.)

VARIATIONS in the amount of citrate used, from 10% below to 21% above that prescribed by McLean and Van Slyke (see this J., 1915, 608; the sodium citrate referred to there is the salt with 5½ mols. of water), do not seriously affect the end-point in the titration with potassium iodide solution provided this is completed without delay. When the titration with potassium iodide is carried out very slowly a blue coloration, which might be mistaken for the end-point, sometimes

develops before the titration is complete. This coloration disappears as the end-point is reached. There is a possibility of slight errors due to adsorption of silver nitrate by the silver iodide formed, if the latter is allowed to agglomerate; this may be avoided by addition of 10 c.c. of 1% starch solution immediately before titration, whereby the silver iodide is retained in solution. The method, applied to amounts of pure sodium chloride up to 100 mgrms., gave a maximum error of 0.24 mgrm.—J. H. L.

*Hydrogen peroxide; Action of—on the neutral salts of lead.* V. Zotier. Bull. Soc. Chim., 1917, 21, 241—243.

NEUTRAL lead salts exert a more or less marked catalytic action on hydrogen peroxide (100 vols.), the catalysis being intense with soluble organic salts and feeble with insoluble organic salts or mineral salts. With a salt such as lead acetate the ultimate result is the complete decomposition of the hydrogen peroxide, and the lead acetate contains no residual lead peroxide (see also J. Chem. Soc., Jan., 1918).—W. G.

*Lead; Some compounds of—.* [Detection of hydrogen peroxide and of lead; preparation of red lead.] V. Zotier. Bull. Soc. Chim., 1917, 21, 241—246.

By the addition of hydrogen peroxide to a solution of lead nitrate in 20% sodium hydroxide, a 40% yield of pure lead peroxide is obtained. Hydrogen peroxide at a dilution of 0.006 gm. per litre may be detected by the addition of a trace of an alkaline solution of a lead salt, and, conversely, lead at a dilution of 0.0025 gm. per litre may be detected by neutralising the solution, adding an excess of hydrogen peroxide, and then, drop by drop, a 1% solution of sodium hydroxide. Amorphous red lead is obtained by heating together equal weights of lead nitrate and 50% sodium hydroxide solution at 150° C., but if the amount of lead salt is halved and the temperature raised to 160° C. a microcrystalline red oxide is obtained (see also J. Chem. Soc., Jan., 1918).—W. G.

*Resources of potash suitable for glass making in the United Kingdom.* Boswell. See VIII.

*Recovery of potash as a by-product of the cement industry.* Ross and Merz. See IX.

*Choice of a [zinc] blende roasting furnace.* Chase. See X.

#### PATENT.

*Electric [induction] metallurgical furnaces.* Eng. Pat. 141,120. See X.

### VIII.—GLASS; CERAMICS.

*Potash suitable for glass making; Resources of— in the United Kingdom.* P. G. H. Boswell. Soc. of Glass Technology, Dec. 19, 1917.

Up to the present no sources of soluble potash salts are available in the British Isles, to replace those previously obtained from Stassfurt and Alsace. Among available insoluble minerals may be mentioned felspar, mica, leucite, and glauconite. The most productive source is likely to be found in the various felspars, which are of wide occurrence. Granite cannot be used as a source of felspar as the latter is too small in grain to be picked out. Granite occurs up and down the country in huge masses, and often at the margins and scattered throughout the mass in veins is found felspar. Such veins are usually known as pegmatite and they may vary from 40 ft. in width to a few inches. Pegmatites are a valuable source of felspar, as

the latter is present in large, easily picked pieces, but they are only to be found in the most inaccessible spots—the North-West of Scotland, the North-West of Ireland, and in Cornwall.

The two chief varieties of felspar are known as microcline and orthoclase, and the latter is to be preferred as a source of potash as there is less tendency of this variety to form mixed crystals with soda felspar. Pegmatite consists of felspar and quartz in large pieces and must be hand-picked, the felspar being in crystals 3 to 4 inches long. Sometimes the felspar is intergrown with the quartz and the rock is then known as perthite. Felspar is prone to decomposition, brought about by gases and solution from below and by atmospheric action. The alkalis are leached out, leaving the silica and alumina as clay. When the leaching is effected from below the final product is very pure china clay. For pottery, felspar should be low in quartz, low in soda (to prevent crystallisation), and low in iron (to obtain good colour). The same features are necessary for glass-making and in addition there ought to be constancy of composition. Unfortunately this can never be realised. A potash content of 10 to 12% is desirable for glass-making felspar and the soda content should not be more than 3%, the iron content not more than 0.1%. All the British felspars are of the microcline type except the Roche deposit in Cornwall, which is orthoclase. The chief deposits in Scotland are in the vicinity of Loch Laxford, Durness, and Overseaig. In Ireland, felspar is found at Belleek, Belmullet, and Glenties, whilst good felsite is obtainable in Waterford and Wicklow, although its iron content is high. There seems little prospect of British felspar being placed on the market as cheaply as the pre-war Scandinavian supplies, owing to the cost of transport in this country and also the higher price and different conditions of labour.

Of late, many methods have been patented for the extraction and recovery of potash. It is hoped that a rich source of potash may be found in the flues of cement kilns and blast furnaces. Possibly it would be found expedient to increase the amount of potash in the flues of cement kilns by using felspar in the manufacture of cement.

W. G. Fearnside said that sedimentary rocks, being the disintegration products of igneous rocks (which all contain potash), might conceivably be a source of potash. Sands and limestones contain very little and so would not pay for extraction, but clays might very well do so.

C. J. Peddle pointed out that felspar was regarded by glass-makers as being important as much for the alumina as for the alkali it contained. It was a cheaper method of giving glass all the beneficial results conferred by alumina, than by using the calcined oxide, and was far more suitable than china clay for this purpose.

*Silver films; Deposition of— on glass.* A. Silverman and R. M. Howe. J. Ind. and Eng. Chem., 1917, 9, 1032—1035.

THE factors governing the deposition of silver on glass by the action of reducing agents on solutions of silver nitrate have been studied—the influence of temperature, time, concentration, and proportions of reagents on the quality of mirror and the quantitative efficiency of the deposition for a variety of reducing agents, namely, potassium tartrate, tartaric acid, lactose, sucrose, formaldehyde. The effect of the addition of alcohol was included. Working at high temperatures (80° C.) sucrose was found to give the best results. The tartrates are less satisfactory. For cold processes using formaldehyde as reducing agent, it was found that low concentration of this lengthened the time of deposition with advantage. Low concentration of the silver nitrate favoured high percentage depositions. The



addition of sugar controls the action of formaldehyde so as to produce uniform mirrors, and also retards the action. The addition of alcohol increases the efficiency of deposition. In view of these principles the following mixing is recommended for cold slow work: 16.5 c.c. of 0.037 molar silver nitrate, 1.0 c.c. of 1.0 molar sucrose, 0.5 c.c. of 80 % methyl alcohol, 2.0 c.c. of 0.8 % formaldehyde. After 40 minutes this deposits 20 % of the silver present. For rapid cold work is recommended the addition of 0.5 c.c. of 40 % formaldehyde to a mixture of 20 c.c. of 0.2 molar silver nitrate with 0.5 c.c. of 80 % methyl alcohol. The results of cold deposition are superior owing to the greater temperature uniformity of reagents and glass and also because of the exclusion of temperature changes after deposition which, owing to different coefficients of expansion of glass and silver, cause a loosening of the mirror.—H. J. H.

#### PATENTS.

*Crucible smelting furnaces [for glass].* C. M. Stein et Cie., Paris. Eng. Pat. 109,245, Nov. 24, 1916. (Appl. No. 16,891 of 1916.) Under Int. Conv., Aug. 29, 1916.

A CRUCIBLE smelting furnace, specially intended for glass or crystal melting. The regenerators are situated outside the main body of the furnace, and glass pits are provided in the base of the central burner and of the flues to protect the regenerators from contact with the glass. Removable plugs are fitted in the glass pits for cleaning purposes.—T. H. R.

*Refractory composition [from slag from manufacture of ferrochromium].* F. M. Becket, Niagara Falls, N.Y., Assignor to Electro Metallurgical Co., New York. U.S. Pat. 1,244,688, Oct. 30, 1917. Date of appl., May 31, 1917.

A REFRACTORY composition contains as an essential component particles of slag derived from the manufacture of high-carbon ferrochromium.  
—W. F. F.

*Glass-blowing machines.* A. E. White, London. From The Westlake European Machine Co., Toledo, Ohio, U.S.A. Eng. Pat. 111,122, Sept. 12, 1916. (Appl. No. 12,895 of 1916.)

*Clay and other substances or articles; Process and apparatus for drying*—F. A. Secord, St. John, New Brunswick, Canada. Eng. Pat. 111,166, Nov. 18, 1916. (Appl. No 16,580 of 1916.)  
SEE U.S. Pat. 1,221,383 of 1917; this J., 1917, 551.

#### IX.—BUILDING MATERIALS.

*Silica bricks; Manufacture of*—H. Le Chatelier and B. Bogitch. Comptes rend., 1917, 165, 742—748. (See also this J., 1917, 963, 964.)

In the manufacture of silica bricks it is the general practice to have present a certain proportion of large grains, sometimes up to 1 cm. in diameter. The object of these grains, it is considered, is to prevent the growth of cracks during firing, but as the grains themselves expand during the accompanying recrystallisation, they tend to exert a disruptive action which increases with the size of grain. For this reason a moderate limit should be set to the size, viz., 0.5 cm. On the other hand, a considerable proportion of impalpable powder is also necessary, whereby the recrystallisation of the quartz as tridymite from the viscous magma is accelerated. During the normal period of firing the solution and recrystallisation can only penetrate to a depth of about 0.01 mm.; hence grains greater than a 0.05 mm. mesh are practically inert and only those below 0.03 mm. are completely changed and are thus the most active in the production of the network frame of recrystallised tridymite in the brick. Exceedingly finely divided silica is necessary, then, to produce this network and in considerable proportion also if the network is to be so far developed as to ensure the stability of the brick at 1700° C. If 10% only of impalpable silica were present, then the lime (2 % of the total silica) would represent 20 % of this and would suffice to cause the whole of it to fuse at the high temperatures of steel furnaces. To confirm these views, experiments were made on the crushing strength at varying temperatures of bricks made up in different ways, with different proportions of coarse and fine grains and different grades of fineness. As impalpable silica the

No.	Mixture.	Linear dilatation. %	Density.		Crushing strength in kilos.		
			Apparent.	True.	Cold.		1600° C.
					After drying.	Fired.	
1	75 raw quartzite 25 impalpable 2 lime	5.2	1.63	2.35	15	165	30
2	75 raw quartzite 25 fines 2 lime	—	—	2.33	10	60	8
3	25 raw quartzite 75 impalpable 2 lime	3.0	1.36	2.35	9	135	10
4	25 raw quartzite 75 fines 2 lime	—	—	2.33	6	52	3
5	75 quartzite baked 25 impalpable 2 lime	3.2	1.57	2.33	10	120	25
6	25 quartzite baked 75 impalpable 2 lime	3.0	1.35	2.34	8	180	15
7	25 quartzite baked 75 impalpable 6 clay	5.0	1.40	2.35	9	150	9.5
8	25 raw quartzite 75 impalpable 6 clay	5.2	1.43	2.36	9.5	160	6
9	25 raw quartzite 75 fines 6 clay	—	—	2.34	7	55	2
10	75 raw quartzite 25 impalpable 6 clay	5.0	1.50	2.34	19	110	18

slimes from gold ore was used after purification with acid. As fine material were used the smaller particles (less than 0.1 mm.) resulting from breaking down a suitable quartz to 4 mm. size. The latter was used as the coarse raw material. The impalpable grains were of the order of 0.01 mm. in diameter. The bricks were fired 5 days up to cone 19.

The crushing strengths are in kilos. per sq. cm., and the values at 1600° C. are those necessary to crush after 1 hour's heating at that temperature. The results (Nos. 1—4) show the value of admixture of impalpable silica (25%) in increasing the mechanical strength at furnace temperatures. Similar experiments made with a quartz, not so easily transformed in baking, gave a like result when impalpable silica was incorporated. At the same time it was observed that although pressing increased the strength of the bricks when cold, no advantage was apparent at 1600° C. when compared with hand made bricks of the same mixture. Experiments (Nos. 5 and 6) made, using quartzite which had been fired in place of the same in the unfired state, showed that where the quartz has undergone a preliminary transformation, it still gives a brick satisfactory at 1600° C., although decidedly inferior to those made from the raw quartz. Where the quartz is readily transformed, there is then no advantage in baking the raw material before manufacture. Experiments made when the 2 parts of lime was replaced by 6 parts of clay containing 2 parts of bases (alumina, iron and potash), showed that the product was inferior to that made with lime (experiments 7—10). These bricks differed also in suffering a progressive deformation under load at 1600° C. instead of a sudden collapse characteristic of bricks with the lime binder. It is concluded that the best composition is 75% of coarse quartz, 25% of impalpable quartz—grain size as given, bound with 2% of lime.—H. J. H.

*Potash; Recovery of*—as by-product of the cement industry. W. H. Ross and A. R. Merz. J. Ind. and Eng. Chem., 1917, 9, 1035—1038. (See this J., 1917, 612.)

The process described previously has been applied to the flue dust from cement kilns. The potash present is liberated and volatilised completely by heating feldspar mixed with lime for 1 hour, on a laboratory scale. A similar reaction takes place in the cement kiln between the constituents of the charge containing lime and potash, and a greater or less proportion of the latter is volatilised, appearing in the flue dust. This, when fresh, consists of sulphates, sulphides, and chlorides of potash, soda, and lime. The proportion of sulphides is greater in oil- or gas-fired kilns. Free lime is present and the extract is alkaline. Analyses of the raw mix and cement clinker from 113 works in America permitted an estimation of the amount of potash recoverable. The raw mix contained from 0.2% to 1.16% of  $K_2O$  and the analyses showed that the weight of potash volatilised per barrel of cement varied from 0.35 to 5.34 lb. Omitting plants using blast-furnace slag, the average of 99 plants was 2.09 lb., which means an annual production for the United States of about 87,000 tons of  $K_2O$  volatilised. By electrical precipitation (Cottrell process) 99% of the flue dust can be precipitated, but as the lighter portions are richer in potash, only 90% of the potash is thus recoverable, i.e., 78,000 tons on the above basis. When the kilns are coal-fired, the proportion of soluble potash in the dust is lowered—59.6% in one case, against 91.6% in an oil-fired kiln. Laboratory experiments show that the process described above—digestion with lime water under pressure—renders 90% of the potash soluble in 4 hours at 100 lb. pressure and 95% after 8 hours. Assuming a 90% yield of soluble potash, the

annual production would be 70,000 tons or, excluding plants volatilising less than 1 lb.  $K_2O$  per barrel, 67,000 tons. This however could be materially increased by increasing the proportion of potash in the raw mix. Where aluminiferous cements are made, feldspar alone can be added, whilst for silicious cements, feldspar and a suitable proportion of iron ore would be necessary. In one case the proportion of  $K_2O$  volatilised was increased from 38% to 65% by the addition of salt to the raw mix. The percentage of total alkali in the finished cement was thereby reduced. In another works calcium fluoride was used and the potash driven off as potassium fluoride. On leaching the dust with hot water the calcium and potassium compounds reacted, regenerating the calcium fluoride in the insoluble residue and leaving the potash in solution as sulphate or carbonate. The insoluble residue was returned to the furnace and in this way the proportion of potash volatilised was increased from 60% to 90%. In some works the proportion of potash volatilised can be increased by raising the temperature of the kiln. The percentage volatilisation varies from 25% to 96% and it is considered that by rational working the lower figures could be much increased. If an average yield of soluble potash equal to about 50% could be attained—and experience shows this is possible—then an annual production of 100,000 tons of  $K_2O$  could be realised. The treatment of cement flue dust by electrical precipitation and recovery of soluble potash is being rapidly extended in American plants.—H. J. H.

*Reinforced concrete; Deteriorating action of salt and brine on*—H. J. M. Creighton. J. Franklin Inst., 1917, 184, 689—704.

THE author emphasises the injurious action of salt solutions, especially of chlorides, on cement, and indicates the special danger to concrete reinforced with iron. In contact with brine the latter becomes converted into oxides of iron either by local electrolytic action or otherwise, for the concrete in practice is never completely impervious to air and water. The oxides or hydroxides of iron are more voluminous than the corresponding metallic iron and therefore exert a powerful disruptive action on the concrete in which the iron is embedded. The effects of this are illustrated by 10 photographs of reinforced concrete structures which have suffered owing to the rusting of the iron skeleton. In all cases the concrete had been exposed to the action of brine. The course of the deterioration is as follows: The concrete develops cracks with iron-coloured stains parallel to the reinforcements; the cracks widen and lengthen until a layer of concrete falls away, baring the iron below.—H. J. H.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Pig iron; Electric*—in war-times. R. Turnbull. Amer. Electrochem. Soc., Oct., 1917. [Advance copy.] 5 pages.

THE recent high prices of pig iron have failed to stimulate its production from ore in electric furnaces. This is attributed mainly to the shortage of power, and not to the inherent difficulties of the process. No advantage is found in the use of shaft furnaces as compared with the ordinary types of electric furnace. The war, however, has given an impulse to the manufacture of pig iron from shell scrap and turnings in electric furnaces, and owing to the purity of the scrap, a product of high quality is obtained without much refining. The principal feature is its low phosphorus content. And account is given of the working of a 6-ton furnace in Canada. It is said that in the United



States steel turnings are being added with the ore charged into blast furnaces. The author doubts whether these processes will be feasible generally after the war, but regards them as possible in Canada. Attention is drawn to the importance of correct fit of the coolers placed (round the electrodes) on the roofs of electric furnaces. The coolers should be cast to the exact form of the brickwork, on which they should rest without intervening packing. This is to ensure uniformity of cooling effect over the whole of the brickwork, whereby its life is materially prolonged.—H. J. H.

*Electric furnace: The — in the Norwegian iron industry.* H. Styri. Amer. Electrochem. Soc., Oct., 1917. [Advance copy.] 11 pages.

IN Norway high-grade charcoal irons were formerly manufactured, but the industry declined during the nineteenth century. From 1909 onwards a number of attempts were made to smelt pig iron in electric furnaces, but with only limited success. Such plants as were working at the commencement of the war have been very successful owing to the failure of imported supplies of pig iron. None of these concerns has converted the latter into steel, but—especially since the outbreak of war—a number of plants have melted steel scrap and manufactured special steels of various kinds. The Rennerfelt type of furnace is most commonly employed, but the author considers that it shows no marked advantages over the Héroult furnace. In late years a strong desire has been evident in Norway to make the country more self-supporting in the matter of steels for general purposes, and the war has accentuated this feeling. The Government has helped in the establishment of steel plant—open-hearth and Tropenas converter furnaces—to meet this want. The prospects of a steel industry are discussed. Coal is not plentiful and is remotely placed near the Russian border. Spitzbergen coal is good for steam raising but poor for coking. Iron ores are generally of low grade and remotely placed. It is considered that the future of structural steels made by the older processes is very doubtful and that the prospects are best for electric furnace products—made from scrap and perhaps from ore—which should command a market owing to their high grade.—H. J. H.

*Pure [iron] alloys; Preparation of — for magnetic purposes.* T. D. Yensen. Amer. Electrochem. Soc., Oct., 1917. [Advance copy.] 18 pages.

THE elimination of impurities has a marked beneficial effect on the magnetic permeability and hysteresis loss of iron. It is considered that the magnetic properties at low and medium flux densities depend largely on the thickness and quantity of the intercrystalline material. It is known that a joint reduces the permeability of two contiguous pieces of iron enormously. Carbon, phosphorus, and sulphur are usually present in iron as the corresponding compounds of iron, at the boundaries of adjacent grains of ferrite, and these compounds are known to possess low permeabilities. Where these compounds are practically absent very high permeabilities may be attained. Elements like silicon and aluminium, which are present in the iron in solid solution, do not materially affect the permeability but increase the resistance. At high flux densities all additions, except nickel and cobalt, lower the permeability; cobalt up to 34.5% increases the saturation capacity. The most reliable sources and methods of preparation of pure metals are tabulated. Electrolytic iron prepared by the method of Burgess (this J., 1904, 667) and cobalt by the method of Kalmus (this J., 1914, 261) are recommended. The metals and alloys are melted in crucibles made of magnesia which has been calcined at 1000°C., fused in an air furnace, ground to pass a 20-mesh sieve, bound with

magnesium hydroxide, and pressed to shape in steel moulds under 25 tons pressure. Electric furnaces with inert atmosphere or working under a vacuum are used. The resulting alloys may be worked or forged, but it so they should be annealed again, as this conduces to growth of crystal grain which favours increased permeability.—H. J. H.

*Manganese and some special manganese steels; Magnetic properties of —.* R. Hadfield, C. Chéneveau, and C. Gêneau. Roy. Soc. Proc., 1917, A, 94, 65—87.

THE relation between the magnetic susceptibility of manganese and manganese steels and their chemical constitution was investigated by means of a Curie and Chéneveau magnetic torsion balance, in which the metal is suspended from a torsion wire and a permanent horseshoe magnet is moved horizontally in a direction at right angles to the line joining the poles so as to vary the field. Anhydrous cobalt sulphate having a mass susceptibility of  $+58 \times 10^{-6}$  at 20°C. was used as a standard of comparison, and magnetic fields of 320 and 1600 gauss were used. The samples were enclosed in thin-walled glass tubes, the effect of which was negligible, and the effect of temperature variations was also found to be comparatively small. Powdered manganese was found to be paramagnetic and to have a mean mass susceptibility of  $11.0 \times 10^{-6}$ . Cast manganese was found to be ferromagnetic, but after repeated heating to redness and washing with hydrochloric acid, the mass susceptibility gradually fell from  $2000 \times 10^{-6}$ , approaching the value for powdered manganese. The ferromagnetic properties were inferred to be due to occluded hydrogen. Manganese steels gave variable and large values which were greatly reduced after washing with hydrochloric acid to remove surface ferriferous impurities, to the order of 75 to  $100 \times 10^{-6}$ . The size of the specimen was found to be immaterial provided the specimen is small, and curves are given showing the effect of varying the relative position of the magnet and the specimen. The presence of manganese has little effect on the magnetic properties of steel; the susceptibility increases with the addition of nickel, copper, chromium, or tungsten, but decreases with the addition of carbon, while silicon makes steel ferromagnetic.—W. F. F.

*Copper tailings; Ammonia leaching of — at Kennerott, Alaska.* H. M. Lawrence. Eng. and Min. J., 1917, 104, 781—787.

THE ores of the Bonanza and Jumbo mines consist of chalcocite, malachite, and azurite in a gangue of grey limestone and dolomite; the proportion of carbonate ore averages 36% of the copper value. The ore is hand-sorted to separate larger pieces of sulphide and carbonate from the poorer material, which is concentrated on jigs and tables; 95% of the sulphide and 60% of the carbonate values are thus recovered. The tailings are treated in the leaching plant (this J., 1917, 966) which has a daily capacity of 300 tons of tailings. The ammoniacal solution in contact with the ore is converted into carbonate, which, however, extracts the copper satisfactorily. The exhausted charge is washed with steam. The solvent is recovered by distillation in evaporators operated intermittently, during which a precipitate containing 70% Cu is obtained; continuous distillation was abandoned owing to clogging and formation of incrustations. The extraction averages 75% of the carbonate copper. The losses of ammonia are stated to be  $\frac{1}{2}$  lb. per ton leached. An 800-ton plant is in course of construction.—W. R. S.

[Copper;] *Recent developments in connection with the use of sulphur dioxide in hydrometallurgy [of —].* E. R. Weidlein. J. Ind. and Eng. Chem., 1917, 9, 1057—1058.

At the experimental 5-ton plant at Thompson,

Neve, carbonate or roasted sulphide ores are leached with sulphuric acid. The solution is neutralised with lime, treated with sulphur dioxide, and heated to 160° C. under a pressure of 100 lb., when metallic copper is precipitated. After remelting it is over 99% pure, the balance being chiefly oxygen. Solution and precipitation are continuous. The plant was started in April, 1916, and is being enlarged.—W. R. S.

*Copper converting; Development of* — M. W. Krejci. Eng. and Min. J., 1917, 104, 669—674.

THE Manhès converter, invented in 1880, was introduced at the Parrot smelter, Butte, in 1883-4. Two types were subsequently evolved: the vertical or Great Falls, and the horizontal or Peirce-Smith type. Tilting was first done by hydraulic power, but this has been almost supplanted by electricity. Converters were originally lined with quartz and clay, the lining combining with ferrous oxide formed by the oxidation of the matte. In later practice silicious ore was thrown into the converter to prolong the life of the lining. In 1910 the magnesite or basic lining was adopted. The lining takes no part in the converting reactions, all the silica being added in the form of ore either through the mouth or the tuyères. At the Great Falls smelter, new magnesite linings used to be first coated with matte by blowing it without silica for a few minutes, and cooling. It was found that the lining so obtained consisted largely of magnetite and was far superior to magnesite as regards resistance to corrosion. This coating is now in regular use, and with proper care the magnesite lining lasts almost indefinitely; the magnetite coating is patched when required, generally once in several months.—W. R. S.

*[Zinc] blende-roasting furnace; Choice of a* — M. F. Chase. Eng. and Min. J., 1917, 104, 698—704.

THE first cost and the working cost of a furnace are of less importance than its ability to produce a roasted ore low in sulphur and free from lumps, and a uniform gas high in sulphur dioxide and poor in dust. The furnace should satisfy the following conditions: (1) the ore must be rabbled continuously; (2) the arches must be low, to ensure thorough removal of sulphur dioxide; (3) the ore must not fall through the ascending gas current; (4) the temperature of the different hearths must be controlled by regulating the air supply; (5) the air must be preheated by passing it over the roasted ore; and (6) the rabbles must be easily accessible for repairs. As no furnace fully fills these conditions and the available data on operating conditions are often unreliable, the installation of the best type of furnace to suit local conditions is largely a matter for experiment. The continuous rabble muffle furnaces (such as the Ridge, Wedge, and Huntington-Heberlein) have decided advantages over other types, since the difficulty of cooling the rabble arms while maintaining the heat of the furnace has been overcome; future improvements will probably take place along the lines developed in these furnaces.—W. R. S.

*Zinc-oxide furnaces.* J. F. Cregan. Eng. and Min. J., 1917, 104, 675—680.

THE furnaces in use are of two distinct types: the Eastern, used chiefly at Palmerton, Pa.; and the Western (Rocky Mountains, etc.). The former consists of a series of (generally six) individual grates leading into a common flue, but controlled separately as regards blast pressure and exhaust. Each grate may be shut off from the main flue at any time. The Western furnace has 8—16 double grates and a main flue under one common arch; the grates have secondary arches and are

separated from each other by side walls, but the exhaust pressure can only be regulated on the furnace as a whole. In furnaces of the Western type, the weight of ore charged is 80—120 lb. per sq. ft. of grate surface; this is mixed with 80—100% of coal, and 10—90% of flux if required. The entire charge is intimately mixed before charging, and is fired for 8 hours. The clinker is removed, and the next charge introduced. The zinc content of the clinker is as low as 1% at certain plants, but much higher in general practice. In the author's opinion the Western furnace is of greater adaptability to varied ores and fluxes.

—W. R. S.

*Brass rolling mill alloys.* R. A. Wood. J. Amer. Inst. Metals, 1917, 11, 181—192.

ANTIMONY and bismuth are most harmful in rolling; the effect of arsenic is much less pronounced. The fuel used for melting down should be as low in sulphur as possible, for copper and its alloys absorb sulphur dioxide and become brittle; the metal should be covered with charcoal during the melting. An alloy containing equal parts of copper and zinc is used for brazing; it can still be rolled or drawn, but the operation is most tedious. Brasses with 57—63% of copper (Muntz metal) are used in the extruding process. Those containing more than 60% of copper can be worked cold. Lead (0.5—4%) is added to brass to facilitate milling, turning, or drilling; it diminishes the cohesion of the metal. Tin produces a close-grained brass of high tensile strength. The trade name "tin-bronze" is given to copper-tin alloys free from zinc; a small amount of phosphorus greatly facilitates casting, and the alloys are then termed phosphor-bronzes. Many manufacturers state that phosphorus in alloys containing zinc produces fine pinholes in the castings, but others consider 0.05% beneficial. Very high tensile strength and density of grain are attained by the addition of iron (0.25—2%); it has a tendency to segregate in small hard nodules; manganese assists in distributing the iron. Brasses to which both tin (0.25—5%) and lead 0.5—3% have been added are free-turning, close-grained and take a high finish. Nickel (2—32%) is used in cutlery and hard spring metal, the usual copper-zinc ratio being 2.5:1; iron (0.5—2%) hardens these alloys considerably. Cupronickel (nickel 5—25%) must be cast at a very high heat, particularly that containing high percentages of nickel. Addition of manganese to cupronickel produces alloys of high electrical resistance.

—W. R. S.

*Antimony; Production of electrolytic — from impure ores.* W. A. Burr. Eng. and Min. J., 1917, 104, 789—790.

THE tests were carried out in a plant capable of producing 1000 lb. of metal per day. The stibnite ore was crushed to pass an 8-mesh sieve in the solvent solution (7% sodium hydroxide and 2% sodium chloride), and the pulp leached in two Douglas fir tanks (diameter 16 ft.), the filter-bottom of which supported one thickness of cocoa matting covered with a seamless woollen filter cloth. The solution, heated with steam to 60° C., was let in beneath the filter-bottom, overflowing into the pregnant-solution tank from which it was decanted into wooden precipitation cells measuring 2½ × 2½ × 15 ft. Each cell contained 80 mild steel sheet electrodes. A current density of 8—9 amp. per sq. ft. of cathode surface, at a tension of 2.25—2.50 volts, gave a coarse crystalline deposit which was scraped off when it had accumulated to ¾ in. on both sides of the cathodes. The precipitate was washed, dried, melted down, and cast into ingots assaying 99.94% Sb. The sulphur in the foul liquors was removed from time to time by carbon dioxide.



The cost of production was 2.2 cents per lb. of "star" metal.—W. R. S.

*Flotation; Chemicals used in ore*——. O. C. Ralston and L. D. Yundt, J. Ind. and Eng. Chem., 1917, 9, 1058—1062.

THE use of flotation agents other than oils is discussed. Sulphuric acid often produces a cleaner froth and an increase in extraction, and sometimes allows of reduction in the proportion of oil used. The cause of the improvement is unknown. With certain partly oxidised copper ores, acid is fatal to good flotation. Copper sulphate (1 lb. per ton of ore) has caused higher recoveries of zinc blende in many cases, though not invariably. Probably the blende particles become coated with copper sulphide, but the reason why the filmed particles float better is not known. Sodium carbonate or hydroxide is used in many mills, but beyond the fact that the results are improved, very few observations on their mode of action have been reported. Sodium silicate and sulphide are also employed. The last named acts by coating oxide ores with a film of sulphide (this J., 1917, 460). Lime is not so desirable as sodium compounds; unlike the latter, it usually flocculates the gangue slime. Tartaric and citric acids are mentioned in Eng. Pat. 17,327 of 1914 (this J., 1915, 910); 0.1 to 0.25 lb. of argol per ton of ore has been used with success on copper ores containing much clayey matter. The use of silicofluorides is claimed in Eng. Pat. 4938 of 1914 (this J., 1915, 431) as a substitute for acids. Sodium manganate and chromate are recommended for differential flotation; they could be made cheaply by fusing local manganese or chromium ores with soda. Sulphur dioxide, and sodium sulphate or thio-sulphate in sufficient amounts inhibit flotation. Chloride of lime is said to effect differential flotation of blende from pyrites. Sodium cyanide has been found deleterious to flotation in most cyanide mills in which tailings have been tested.

—W. R. S.

*Flotation concentrates; Vacuum filtration of*——. R. R. Woolley, Eng. and Min. J., 1917, 104, 875—877.

UP to the present, the most successful method of de-watering flotation concentrates has been vacuum filtration. The pulp is settled in Dorr thickeners and pumped into filter troughs from which it passes into continuous-vacuum filters. Since the drying of the cake is caused by the air passing through it, the amount of the latter must be regulated so as to build a cake and permit the greatest volume of air to pass through it at the least cost. To determine this requires experiment in each individual case. Usually the pumps are designed to pass 0.5 to 1 cub. ft. of air per minute for each sq. ft. of filter surface. The filtering medium used must be adapted to the character of the pulp. With fine compact material the best results are obtained with light twill; with loose granular concentrates, duck-cloth is required. Smaller particles of ore have a greater relative area, hence the amount of moisture remaining in the cake increases as the size of the particles decreases.—W. R. S.

#### PATENTS.

*Steel ingots; Casting*——. Sir W. G. Armstrong, Whitworth and Co., and H. H. Ashdown, Newcastle-upon-Tyne, Eng. Pat. 111,133, Nov. 10, 1916. (Appl. No. 16,142 of 1916.)

To prevent segregation and formation of corner cracks, the sides of the ingot, and the bottom from the base upwards, are cooled during pouring. In the sides of the mould and in the bottom plate are pipes for the circulation of the cooling medium,

so arranged that the bottom plate, or any section of the mould, can be cooled as desired.—T. H. B.

*Engraved steel or other intaglio plate; Method of reproducing an——and an intaglio printing plate produced by said method.* R. W. Barker, London. From American Bank Note Co., New York. Eng. Pat. 111,164. Nov. 17, 1916. (Appl. No. 16,525 of 1916.)

A CAMEO matrix of ductile metal such as lead is formed by hydraulic pressure from an original plate bearing a design formed of a number of shallow depressed lines. Iron or steel is deposited upon this matrix used as a cathode in an electrolyte of iron sulphate solution containing about 1% of vanadium chloride, and having a density of at least 21° B. and preferably at a temperature of 160° F.; nickel sulphate or nickel ammonium sulphate may be used instead of vanadium chloride. A thin coating of copper on the matrix before deposition of the iron produces a better printing surface; the matrix is rotated slowly and is cleared from liberated hydrogen by mechanical means or by reversal of current, and precipitated oxides are removed by constant circulation of the electrolyte through a filter, the salts thus lost being replaced by fresh solution; finally, the matrix is stripped from the deposited plate and the iron or steel hardened or tempered.—T. H. B.

*Electric [induction] metallurgical furnaces.* A. Turner, Greenock. Eng. Pat. 111,120, Aug. 16, 1916. (Appl. No. 11,564 of 1916.)

FOR the direct reduction of iron and steel from the ore, with evolution of cyanogen in the gases, and its fixation in the lime slags, three furnaces are used, two of the reverberatory type, the other a cupola of the retort type. The latter is in the middle, the others on each side of it, one under the base of the cupola hearth, the other on the cupola scaffold, forming a reservoir of any desired capacity, and all the furnaces are connected by channel-ways for fluid molten iron. The cupola has a circular channel, either inside or outside of it, and the channel has branch gates leading in pairs into the shaft of the cupola; beneath these paired gates and the refractory lining, horse-shoe electro-magnets are placed so that each gate has a limb of the magnet beneath it. The production of cyanogen in the reduction of the charge is accelerated by the action of the air which is drawn through the tuyères by the gravitational action of the falling, electrically charged molten iron. The cyanogen is conducted, by means of pipes, from the cupola hearth, over the lime-iron ore fluxes in the reverberatory furnaces. When cyanamide is desired, carbide-producing material is used as a flux.—T. H. B.

*Ores and the like; Dressing*——. W. Mauss, Johannesburg, Transvaal. Eng. Pat. 111,377, Jan. 10, 1917. (Appl. No. 499 of 1917.)

CRYSTALLINE and colloid constituents in ore pulp, mixed with water, are separated from each other by a centrifugal force, not less than fifty times, and usually from one to two hundred times greater than that of gravity.—T. H. B.

*Ferro-phosphorus; Process for producing*——. J. J. Gray, Rockdale, Tenn., U.S.A. Eng. Pat. 111,385, Jan. 19, 1917. (Appl. No. 987 of 1917.)

SEE U.S. Pat. 1,220,416 of 1917; this J., 1917, 555. The requisite high temperature in the fusion zone is maintained by using a blast of air preheated to at least 900° F. (480° C.).

*Zinc smelter; Continuous*——. W. McA. Johnson, Hartford, Conn. U.S. Pat. 1,244,504, Oct. 30, 1917. Date of appl., Feb. 4, 1915.

AN electric furnace is connected with a con-

denser and with a preheating retort. Granulated material to be smelted is fed into the preheating retort which is heated externally by means of a fuel-fired furnace, and the charge then falls through a drop shoot which hermetically connects the retort and electric furnace. The whole of the gases from the electric furnace pass to the condenser.—T. H. B.

[Alloy.] *Composition of matter.* C. L. Jones, Oakland, N.J., Assignor to F. J. Molt, Riverdale, N.J. U.S. Pat. 1,241,742, Oct. 30, 1917. Date of appl., Feb. 8, 1917.

AN alloy of Ni (67.8%), Cu (28.0%), Mn (2.5%), Fe (1.5%), V (0.2%), with or without carbon.—T. H. B.

*Alloy.* F. M. Becket, Assignor to Electro Metallurgical Co., Niagara Falls, N.Y. U.S. Pat. 1,245,552, Nov. 6, 1917. Date of appl., Apr. 10, 1916.

AN alloy which is tough, hard, and resistant to oxidation, consists of Cr 20–35 (25 to 30) %, C 1.5 to 3.0 %, Si nil to 3.0 %, the remainder being principally iron.—T. H. B.

*Copper ores; Process for the treatment of* —. G. C. Westby, Ludwig, Nev., Assignor to Western Process Co. U.S. Pat. 1,244,810, Oct. 30, 1917. Date of appl., Aug. 17, 1915.

OXIDISED copper ore is wetted and subjected to the action of gaseous sulphatising reagents, the resulting sulphates are leached out and treated with metallic iron, and the precipitated metals removed. The solvent is removed and the residue dried and calcined to form a pigment and acid gases, the latter being used again for sulphatising.—T. H. B.

*Ores; Process for the treatment of* —. G. C. Westby, Ludwig, Nev., Assignor to Western Process Co. U.S. Pat. 1,244,811, Oct. 30, 1917. Date of appl., Jan. 12, 1917.

ORE mixtures containing sulphide are treated in an enclosed space with a descending current of a solution of acid nitrate, while a counter current of sulphur dioxide and oxygen regenerates nitric and sulphuric acids. The solution is used repeatedly until the ore body is exhausted, and is then passed through a mass of cleaned sulphide to liberate nitrous gases, which are oxidised and used again.—T. H. B.

*Sintering [flue-dust, etc.]; Process of* —. A. J. Boynton, Lorain, and A. E. Sands, Akron, Ohio. U.S. Pat. 1,245,183, Nov. 6, 1917. Date of appl., Aug. 3, 1917.

FLUE-DUST or other similar combustible material is sintered by forming it into a bed on a porous hearth, igniting the upper horizontal face of the bed, and drawing air through it vertically downwards; the bed is tilted to drain off molten cinder formed during the sintering operation.

*Open-hearth furnace.* T. S. Blair, jun., Chicago, Ill., Assignor to Blair Engineering Co., New York. U.S. Pat. 1,245,555, Nov. 6, 1917. Date of appl., July 20, 1917.

THE upper part of the end wall of the gas down-take has a chrome ore facing, immediately below the entrance of the gas port; sections of the facing rest on a series of water-cooled shelves which keep them in position.—T. H. B.

*Metallurgical filtration; Method of* —. P. A. Boeck, Assignor to Celite Products Co., Los Angeles, Cal. U.S. Pat. 1,245,557, Nov. 6, 1917. Date of appl., Nov. 3, 1914.

A MIXTURE of divided ore and divided inert porous mineral matter (e.g., kieselguhr) is treated with a leaching liquid and then filtered.—T. H. B.

*Blast furnaces; Water circulation of tuyères and coolers of* —. W. J. Foster, Walsall, Eng. Pat. 111,199, Jan. 17, 1917. (Appl. No. 815 of 1917.)

*Atomising metals in a state of fusion; Process for* —. F. C. Ucar, Madrid. U.S. Pat. 1,245,536, Nov. 6, 1917. Date of appl., July 6, 1915.

SEE Eng. Pat. 2113 of 1915; this J., 1916, 425.

*Burnt pyrites and the like; Method for treating [chloridising]* —. E. R. Sutcliffe, Leigh. U.S. Pat. 1,245,631, Nov. 6, 1917. Date of appl., July 11, 1913.

SEE Eng. Pat. 16,450 of 1912; this J., 1913, 1115.

*Refractory composition [from slag from manufacture of ferrochromium].* U.S. Pat. 1,244,688. See VIII.

*Insecticidal composition [from smelter fume dust].* U.S. Pat. 1,244,453. See XIXB.

## XI.—ELECTRO-CHEMISTRY.

### PATENT.

*Electric furnaces; Temperature-controlling device for* —. C. E. Hearson, London. Eng. Pat. 111,207, Feb. 1, 1917. (Appl. No. 1661 of 1917.)

IN a tubular electric resistance furnace designed to be operated at say 700°–800° C., the furnace chamber proper, which may be of silica glass, is enclosed in an outer brass tube which acts as a thermostatic element and is connected at each end to slabs of poor conducting and good fire-resisting material, the slab at one end being free to move away from or towards the slab at the other end as the brass tube expands or contracts. A light steel rod is supported between the end slabs outside the furnace and is connected with a lever in such a way that the variations in the expansion and contraction of the brass tube operate spring contact devices for breaking and making the heating current circuit and so regulate the temperature of the furnace.

## XII.—FATS; OILS; WAXES.

*Emulsions; Stability of* — in the constricted tube and marble device for anaërobiosis. I. C. Hall. J. Phys. Chem., 1917, 21, 609–622.

IN the cultivation of anaërobic bacteria in a constricted tube with a seal of hydrocarbon oil, in which a marble was placed, it was found that when the tube was heated in boiling water and then cooled, the oil below the marble became opaque owing to the formation of a fairly stable emulsion with the water. Emulsions may be prepared in this way with water and various petroleum oils, chloroform, ether, benzene, xylene, carbon bisulphide, olive oil, and aniline. The stability of such emulsions when formed in an open tube by rapid cooling in ice water, is slight as compared with similar emulsions formed below a marble seal, e.g., in the case of water and olive oil less than one day as compared with more than 61 days below the seal. The presence or absence of an air surface above the emulsion does not affect the stability, but a marble or other mechanical seal interferes with diffusion and thus prevents evaporation, and to this must be attributed the relatively greater stability of emulsions formed below such a seal.—C. A. M.

### PATENTS.

*Drying oil; Vegetable* — [and cattle food]. S. Loamnitz, Merida, Mexico, Assignor to J. Joffe, New York. U.S. Pat. 1,244,521, Oct. 30, 1917. Date of appl., Feb. 17, 1916.

A DRYING oil is obtained from the seeds of the



chia plant of the genus *Salvia*, family *Labiatae*, whilst the residue forms a cattle food.—C. A. M.

*Seeds and nuts; Process of treating* — for obtaining oil and other products [tannin]. B. Loomis, Assignor to Burdett Loomis Corporation, Hartford, Conn. U.S. Pat. 1,244,995, Oct. 30, 1917. Date of appl., May 5, 1915.

Hot water and an acid solvent, such as acetic acid, are made to circulate at a temperature between 100° and 170° F. (38° and 71° C.) through the oil-bearing material in a closed tank and the resulting extract of tannin drawn off. The residual material is then treated with water and the acid solvent at an increased temperature to remove oil, waxes, gums, etc., and the extract drawn off into a separating chamber, whilst the residue is subjected to the action of a circulating current of gas at a gradually increasing temperature until free from ammonia or other volatile matter, and is then dried.—C. A. M.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Some compounds of lead.* [Preparation of red lead.] Zotier. See VII.

#### PATENTS.

*Pigments.* L. G. Hill, Birmingham, Eng. Pat. 111,318, Nov. 16, 1916. (Appl. No. 16,121 of 1916.)

Any clay, rock, ore, marl, or the like, free from carbonates, and containing not less than 25% of metallic oxides, of which 80% is aluminium oxide, is deprived of a portion or all of its silicious constituent by means of hot caustic soda solution. For example, china clay is heated under pressure in a strong solution of caustic soda. The undissolved residue when washed and dried is suitable for use as a pigment.—E. W. L.

*Kauri and other like gums; Process for the treatment of* —. F. V. Raymond, Auckland, N.Z. U.S. Pat. 1,245,273, Nov. 6, 1917. Date of appl., Sept. 6, 1916.

KAURI and other like gums are treated by means of sand blasts, water blasts, and drying blasts, operated by compressed air.—E. W. L.

*Resinous compound; Halogenated* —. J. P. A. McCoy, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,245,363, Nov. 6, 1917. Date of appl., Feb. 3, 1916.

A HALOGEN (chlorine) di-substitution product of a resinous coal-tar derivative, containing cumarone, indene, para-cumarone, and/or para-indene, is prepared by passing gaseous chlorine for about 8 hours through a melted coal-tar derivative containing at least one of the above-named substances.—E. W. L.

*Process for the treatment of copper ores.* U.S. Pat. 1,244,810. See X.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

*India-rubber latex; Apparatus for and process of estimating the volume of acid required to coagulate a given volume of* —. T. Cockerill, Colombo, Ceylon, Eng. Pat. 100,591, May 27, 1916. (Appl. No. 7511 of 1916.) Under Int. Conv., May 29, 1915.

The stem of a metal hydrometer of special design is so graduated that, either directly, or by

reference to tables, any of the following values for a given sample of latex into which the hydrometer is introduced may be read off:—specific gravity, ounces of wet rubber per gallon of latex, volume of glacial acetic acid required for coagulation, volume of 4% acetic acid required for coagulation.—E. W. L.

*Product possessing rubber-like properties.* C. Weygang, Maidenhead, Eng. Pat. 111,171, Nov. 22, 1916. (Appl. No. 16,733 of 1916.)

CASEIN or animal glue is digested with carbolic acid or acetic acid at a temperature not exceeding 180° F. (82° C.) until complete solution is effected. Mineral substances such as zinc oxide, zinc carbonate, or calcium hydroxide, and organic substances, such as glycerin, molten pitch, or solutions of resins or of rubber or celluloid may then be added, and the mixture is treated with formaldehyde, formic acid, or tannic acid with energetic mixing until the whole is converted into a tenacious rubber-like mass. If much pitch is used the product may be sufficiently insoluble without the subsequent treatment with formaldehyde or formic or tannic acid. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4343 of 1874, 18,861 of 1896, 15,115 of 1898, 926 of 1901, 27,090 of 1904, 12,277 and 12,278 of 1905, 23,031 of 1907, 4154 and 18,691 of 1909, 26,093 of 1910, and 17,242 of 1914; this J., 1897, 749; 1898, 933; 1905, 812; 1906, 327; 1908, 1075; 1910, 100, 911; 1911, 1398; 1916, 266.)—D. F. T.

*Rubber products; Manufacture of* —. L. E. Barton, New York, and H. A. Gardner, Washington, U.S.A. Eng. Pat. 111,257, June 29, 1917. (Appl. No. 9421 of 1917.) Under Int. Conv., Jan. 2, 1917.

TITANIUM dioxide is applied as a "filler" for rubber, the advantages claimed in the resulting mixing including increased strength and rapidity of vulcanisation whilst the oxide also has exceptional colouring power and is capable of giving remarkably white products. For the production of a vulcanised rubber showing great toughness and therefore applicable to the formation of automobile tyre treads, a mixing of titanium dioxide 450, sulphur 50, and washed rubber 500 parts is recommended. The titanium dioxide need not be pure and can be used with excellent results in the form of "composite titanic oxide pigments" (see this J., 1915 1104; 1917, 91, 604.)—D. F. T.

*Rubber; Compounding of* —. W. C. Geer, Akron, Ohio, Assignor to B. F. Goodrich Co., New York, U.S. Pat. 1,245,700, Nov. 6, 1917. Date of appl., May 26, 1916.

IN order to mix fine powder, such as gas-black, with rubber in a mixing mill, the powder is first moistened, e.g., with kerosene oil, and brought into a dustless, granular condition.—E. W. L.

### XV.—LEATHER; BONE; HORN; GLUE.

#### PATENTS.

*Tanning [with iron salts]; Processes for* —. O. Röhm, Darmstadt, Germany, Eng. Pat. 103,827, Jan. 25, 1917. (Appl. No. 1259 of 1917.) Under Int. Conv., Jan. 28, 1916.

SATISFACTORY tanning may be effected with iron salts when the skins are treated either previously or simultaneously with an aldehyde such as formaldehyde. Example: 100 parts of unhaird hides is filled for 1–2 hours with 200 of water containing 3 of 40% formaldehyde solution and 2 of a sodium bicarbonate. The hides are washed and tanned in a solution of 20 parts of iron chloride

in 150 of water rendered basic by the addition of 1 part of sodium carbonate. After tanning, the leather is washed and neutralised in the same way as chrome leather. If desired the iron chloride bath may contain also chromium or aluminium chloride. After tanning in this way the leather may be treated with precipitating reagents, such as ammonia, alkalis, alkaline salts, phenols, naphthols, organic carboxylic acids, vegetable tannins, soap, sulphides, polysulphides, etc. By alternate treatment with iron salts and ammonia as much as 30% of ferric oxide may be fixed in the leather without destroying its quality.

—J. F. B.

*Tanning.* J. Y. Johnson, London. From Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 111,341, Nov. 11, 1916. (Appl. No. 16,201 of 1916.)

CRYSTALLINE aromatic compounds containing a sulphonic or carboxylic group or both, soluble in water, and capable of precipitating glue and gelatin from solution, are used for tanning, instead of the amorphous materials hitherto used. Several examples are given. For instance, dihydroxy-ditolylmethane is treated with sulphuric acid, the sodium salt of the sulphonic acid produced is precipitated with common salt, and the aqueous solution of the sodium salt used for tanning in the ordinary way after the addition of a little sulphuric acid. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 22,516 of 1894, 287 of 1908, 8512 of 1912, and 8818 of 1914; this J., 1909, 153; 1913, 295; 1915, 1155.)

—F. C. T.

*Adhesive.* G. H. Brabrook, Assignor to A. T. Fletcher, Boston, Mass. U.S. Pat. 1,214,163, Oct. 30, 1917. Date of appl. Apr. 1, 1916.

AN adhesive is made by dissolving casein in a solution of a cuprammonium compound. Lime water may be added.—F. C. T.

*Horny bodies; Process and apparatus for making* — particularly of clear translucent or transparent quality from mealy albuminous masses as casein. A. Kohnert, Berlin. Eng. Pat. 102,375, Nov. 15, 1916. (Appl. No. 16,386 of 1916.)

THE finely-divided material is whirled in a container and moistened by spray or mist from an atomiser, operated by steam or compressed air or both. An addition of 15–20% of water leaves the mass still in the form of meal, and in a suitable condition for moulding under high pressure.

—F. C. T.

*Process of treating seeds and nuts for obtaining oil and other products [lamin].* U.S. Pat. 1,214,995. See XII.

## XVI.—SOILS; FERTILISERS.

*Hygroscopic coefficient [of soil]; Direct determination of the —.* F. J. Alway, M. A. Kline, and G. R. McDole. J. Agric. Res., 1917, 11, 117–166. (See also this J., 1917, 10.)

HILGARD'S method was investigated. A moist chamber was prepared consisting of a paraffined wooden box and lid, with a metal tray fitting in the bottom containing a layer of water, and with blotting paper lining the sides and reaching to the bottom of the metal tray. A galvanised wire table was held in position about an inch above the water and an inch from the sides of the box. On this table the tray containing the layer of soil was placed. The box was placed in a dark cellar at a constant temperature of 22°–24° C. in summer and 17°–20° in winter. The soil was

dried at 100°–110° C. before starting the determination; this did not raise the result over that of an air-dried sample. It was found that the amount of hygroscopic moisture increased with an increase in temperature. A time of exposure to the saturated atmosphere of less than 12 hours was insufficient and of more than 24 hours was unnecessary. A soil holding its maximum hygroscopic moisture loses this very rapidly on being brought into the ordinary atmosphere, but if the samples during a determination are removed rapidly, within 30 seconds, from the moist chamber into the drying tubes no error is caused. The most advantageous materials for the soil tray were found to be copper and aluminium, although glass, graniteware, and vulcanised rubber gave satisfactory results; pasteboard gave low results. Not more than two trays should be placed in a box. A soil containing pebbles or gravel should be broken with an iron pestle to pass through a sieve with a 1 mm. mesh. The method was found to give reliable results.—J. H. J.

*Nitrate-nitrogen accumulation in soil; Some factors affecting —.* P. L. Gainey and L. F. Metzler. J. Agric. Res., 1917, 11, 43–61.

PRELIMINARY experiments were made upon the effect of the variations in the methods of investigation of nitrification in soil upon the results obtained. Ammonium sulphate was used as the source of nitrogen and was added at the rate of 60 mgms. of nitrogen per 100 grms. of soil, which was a silt loam. Calcium carbonate and water were also added, and the soil was kept in a moist chamber for four weeks at the ordinary temperature. Nitric nitrogen was determined by a modified phenoldisulphonic acid method. It was found that the quantity of soil used, the depth of the column of soil, the ratio of surface to volume, and the amount of evaporation had little effect upon nitrification. The compactness of the soil was of importance; if much compressed, nitrification ceased a few inches below the surface. These results indicated that access of air appeared to be the controlling factor, and further experiments were made from this point of view. It was found that as the moisture content of a soil decreased, an increase in its compactness brought about an increase in nitrification. The moisture content varied with the compactness, and was found to be an optimum when it amounted to two-thirds of the amount retainable with the particular degree of compactness. Increase in compactness did not affect aeration to a depth of two feet, provided the moisture did not exceed the optimum. Nitrification took place less rapidly in a pulverised than in an unbroken soil. Even in a soil uncultivated for seven years, aeration was in excess of that necessary to maintain aerobic conditions. The conclusion is drawn that increased aeration is not the cause of the beneficial effect observed to follow cultivation.

—J. H. J.

*Basic slags; Yields of grass from [manuring with] various —.* A. W. Oldershaw. J. Board Agric., 1917, 24, 819–826.

EXPERIMENTS were made upon the application of basic slag of various degrees of solubility to a heavy boulder clay soil in Suffolk for a period of four years, the results being judged by the yields of green grass obtained. The slag was applied at the rate of 40 lb. of phosphoric acid per acre. A plot without manure gave the lowest yield of grass, and the other plots gave increasing yields in the following order: ground Belgian phosphate, slag of low total phosphate and low solubility, slag of low total phosphate and fairly high solubility, slag of high total phosphate and high solubility, and slag of low total phosphate and high solubility. Applied at the rate of 10 cwt. per acre, a slag of low percentage of total phosphate



and high solubility gave more than double the yield of grass given by a slag of low percentage of total phosphate and low solubility. The conclusion is drawn that the citric acid solubility of the phosphate in a slag is a factor of great importance in determining the response of a soil of the above character to treatment with the slag.

—J. H. J.

*Sulphur: Effect of—on different crops and soils.* O. M. Shedd. J. Agric. Res., 1917, 11, 91—103. (See also this J., 1916, 749.)

EXPERIMENTS were made to determine whether the application of sulphur to soils low in sulphur content was beneficial to crops grown on them. Eight different types of soil in Kentucky were used and the seeds planted were soy beans, clover, oats, alfalfa, and wheat. The experiments were done in dried jars, 100 lb. and 200 lb. per acre of flowers of sulphur being added to the soil, together with the requisite amounts of calcium phosphate, potassium nitrate, and calcium carbonate. The weights of the crops obtained were determined. The results were very varied. In the majority of cases there was a gain in weight of crop from the use of sulphur, but with some plants on some soils its use was injurious. Where gains occurred, both the total sulphur and the sulphate sulphur were increased proportionately to the amount applied. The gain of sulphur in the case of clover and alfalfa was in the form of sulphate, while with soy beans it was in some other form, and in this case also the protein was frequently increased. Sulphate was formed during germination of the seeds, except in the case of clover.

—J. H. J.

*Ammonium citrate solutions; Preparation of—, and determination of insoluble phosphoric acid [in fertilisers].* P. McG. Shuey. J. Ind. Eng. Chem., 1917, 9, 1045.

A NEUTRAL solution of ammonium citrate can be quickly prepared by mixing commercial citric acid dissolved in water with the calculated quantity of concentrated ammonia; 4 lb. of citric acid is added to 6961 c.c. of water and 1760 c.c. of ammonia (sp. gr. 0.900 at 15° C.) is stirred in; 9512 c.c. of solution (sp. gr. 1.09 at 20° C.) is obtained and this is neutral to corallin. Extreme precautions to secure neutrality of samples of mixed manure before determining the insoluble phosphoric acid are not regarded as essential. Concordant results can be obtained without them.

—H. J. H.

*Identity of cyanuric acid with the so-called "tetra-carbonimide."* Walters and Wise. See XX.

#### PATENT.

*Soil; Treatment of— for improving its productivity.* The Thames Bank (Blackfriars) Iron Co., Ltd., Thornaby-on-Tees, and S. Hopkins and A. D. Greaves, London. Eng. Pat. 111,254, June 20, 1917. (Appl. No. 8843 of 1917.)

CAVITIES in the soil from which roots have been removed are treated for a few minutes with a jet or spray of hot water between 180° and 210° F. (preferably 190° F.), and the whole area of the root cavities then dug over and treated for several hours with hot water between the above-mentioned limits of temperature. The latter process may also be used alone, though it is preferable to use both treatments.—C. A. M.

### XVII.—SUGARS; STARCHES; GUMS.

*"Norit" (decolorising carbon) process of refining as applied in a beet sugar factory.* W. H. Giffard. Intern. Sugar J., 1917, 19, 506—509. (See also this J., 1915, 370.)

EXPERIENCE in a Dutch beet sugar factory has

shown that by remelting the second and third product sugars and treating the resulting liquor with "Norit," a high-grade sugar is obtained. A mixture of 3 hectol. of a suspension of the decolorising carbon (produced by the addition of 80 kilos. to 40 hectol. of cold water) and 40 hectol. of the sugar liquor was stirred in a circular tank for about 20 minutes, and the temperature gradually raised to 90° C. A little phosphoric acid was added to the liquor, which was boiled and allowed to stand for about 12 hours. It was passed first through filter-presses and next through the ordinary filters, and finally run into tanks to await boiling to grain in the vacuum pans. The "Norit" from the filter-presses was regenerated by boiling for one hour with a solution of soda and then washing with water. When after continued use the ash content of the "Norit" had risen from the original 6% to 12%, revivification was effected by boiling with hydrochloric acid, and subsequently washing well with water. Additional fresh decolorising carbon was added from time to time to maintain the necessary quantity. It is stated that when the process is in thorough working order it is sometimes possible to reduce the amount of dry "Norit" used to 1.8% of the weight of sugar under treatment.—J. P. O.

*White sugar; Report on experiments investigating Weinrich's dry lime process of making—.* J. P. Ogilvie. Intern. Sugar J., 1917, 19, 450—452.

IN experiments recently carried out in the Guanica Centrale, Porto Rico, for the purpose of investigating Weinrich's dry lime process of making white sugar (U.S. Pat. 1,084,772 of 1914; this J., 1914, 212), it was found that after the second carbonatation an almost water-white and perfectly clear juice was obtained, from which a satisfactory white sugar could be recovered by boiling, the press-cakes, moreover, being uniformly hard and of low sugar content. A lime-kiln not being available, gas containing only about 13% CO<sub>2</sub>, obtained from an Otto producer charged with coke to which limestone had been added from time to time, was used. No sulphur was employed, but after the second carbonatation the juice was treated with a small quantity of phosphoric acid. Owing to the low carbon dioxide content of the gases, carbonatation was slow, and a certain amount of glucose (dextrose) was destroyed; the average gain in purity was 3.25%. At times, however, when carbonatation could be operated more rapidly and a low temperature could be maintained, the gain in purity was as high as 7%, while the amount of dextrose destroyed was insignificant. Similarly favourable results were obtained with a first molasses of 55° purity. It is claimed that with a fully-equipped plant the average gain in purity would be 5° without the destruction of any appreciable quantity of the dextrose present, thus obtaining by a comparatively economical and simple process results superior to any so far attained with the ordinary double carbonatation process as carried out in Java and elsewhere.—J. P. O.

### XVIII.—FERMENTATION INDUSTRIES.

*Grape marc; Utilisation of—as fuel.* C. Matignon and G. Marchal. Comptes rend., 1917, 165, 718—721.

VERY large quantities of grape marc are at present not utilised. It is not generally employed as feeding stuff or fertiliser, though it contains about the same proportions of nitrogen, phosphorus, and potash as farmyard manure. The average moisture-content of fresh marc is about 65% and this could without difficulty be reduced to 25—30% by air drying under sheds. Marc

from the Chablis district (Yonne), dried at 115° C., had the following composition:—C, 48.2%; H, 5.45%; N, 2.4%; ash, 11.6%, including CaO, 2.64%; K<sub>2</sub>O, 2.34%; SiO<sub>2</sub>, 2.27%; Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>, 1.77%; P<sub>2</sub>O<sub>5</sub>, 1.08%. From analyses published by Muntz, however, it appears that the average ash-content is nearer 6%. The calorific value of the dried marc, after allowing for the evaporation of the water produced by combustion, was found to be 4400 cal. (7900 B.T.U. per lb.), and the temperature theoretically attainable 1930° C. From these data the authors conclude that grape marc could be utilised as fuel, given suitable plant. The fuel value of the marc produced annually in France is estimated as equivalent to at least 160,000 tons of coal.—J. H. L.

*Attempted biochemical synthesis of diglucosides of polyhydric alcohols. β-Diglucoside of glycol.* Bourquelot and Bridel. See XX.

#### PATENTS.

*Fermented beverages; Method and apparatus for producing dealecoholised* —. G. Ciapetti, Rome. U.S. Pat. 1,243,811, Oct. 23, 1917. Date of appl., May 8, 1912.

THE beverage, previously deprived of the greater part of its ethers and gases, is caused to flow over a succession of heated surfaces in a thin film to drive off a portion of the alcohol with a portion of the water. The alcohol and water thus driven off are separated from one another, the water is cooled and re-introduced into the beverage in separate portions, one immediately after the beverage has passed each heated surface, to maintain the volume substantially constant and to assist in maintaining a low temperature.—J. F. B.

*Beverage.* J. Ashus, Roxbury, Mass., U.S.A. Eng. Pat. 111,225, Mar. 27, 1917. (Appl. No. 4416 of 1917.)

SEE U.S. Pat. 1,226,439 of 1917; this J., 1917, 732.

*Beverage; Process of making a non-alcoholic* —. A. L. Straus, Assignor to Baltimore Process Co., Baltimore, Md. Reissue 14,391, Nov. 6, 1917. U.S. Pat. 1,223,121, Apr. 17, 1917. Date of appl., Aug. 4, 1917.

SEE this J., 1917, 608.

### XIXA.—FOODS.

*Wheat, rice and maize grains; Distribution of the [antineuritic] substance in —, the deficiency of which in a diet causes polyneuritis in birds and beri-beri in man.* H. Chick and E. M. Hume. Proc. Roy. Soc., 1917, B, 90, 41–60.

IN the case of wheat and rice grains, the antineuritic vitamin is concentrated mainly in the germ or embryo; it is also present to a less extent in the bran. Ordinary white (wheaten) flour is deficient in the substance and, if used as an exclusive diet, will induce polyneuritis in pigeons or beri-beri in man. The embryo of maize grains also possesses marked antineuritic properties. The addition of wheat germ to a diet of polished rice in quantity (3 grms. every second day) sufficient to prevent polyneuritis in pigeons, also maintains the weight and general health of the birds; in larger quantities (2 to 3 grms. every day) it leads to great increase in the body-weight. Tinned meats and vegetables are deficient in anti-beri-beri vitamins and a diet consisting of these preserved foods should be supplemented by wheaten bread containing the germ of the grain.

—W. P. S.

*Substance (vitamine) whose deficiency in a diet causes polyneuritis in birds and beri-beri in man; Effect of exposures to temperatures at or above 100° C. on the —.* H. Chick and E. M. Hume. Proc. Roy. Soc., 1917, B, 90, 60–68.

THE antineuritic vitamin present in the embryo of wheat grains is not injuriously affected when the embryo is heated at 100° C. for two hours, but its properties are destroyed rapidly at 120° C. The vitamin contained in yeast extract is also destroyed at 120° C., but not at 100° C.—W. P. S.

*Bread-making; Use of lime in —.* J. Effront. Monit. Scient., 1917, 61, 241–246.

THE acidity of flour and the bad odour and keeping qualities of bread are not caused by enzymes in the bran but are due to bacterial action, the growth of the bacteria being promoted by defective drying of the grain. The enzymes and bacteria are not affected by small quantities of acids or alkalis, and the use of lime-water in making the dough is useless, if not even injurious, when a poor yeast is employed. Bread made with the use of lime-water is less digestible than ordinary bread owing to the conversion of the acid phosphates into insoluble phosphates.—W. P. S.

*Phytin phosphorus in plant products; Determination of —.* J. B. Rathier. J. Amer. Chem. Soc., 1917, 39, 2506–2515.

THE accuracy of the method proposed by Heubner and Stadler for the titration of phytin with ferric chloride solution (this J., 1914, 713) was confirmed by titration of sodium and strychnine salts of phytin from various plant products. The ratio of iron to phosphorus, 1:1.19, accords with the formation of a hepta-ferric salt of inositol-pentaphosphoric acid and insofar confirms the author's previous conclusions as to the composition of phytin (see this J., 1917, 562). The method may be applied to the determination of phytin phosphorus in plant products, such as seeds, as follows:—8 grms. of the finely-ground material is digested with 200 c.c. of 1.2% hydrochloric acid for 3 hours at the ordinary temperature, with frequent shaking. Fifty c.c. of the filtered extract, in a slender conical beaker of about 150 c.c. capacity, is treated with 10 c.c. of 0.3% ammonium thiocyanate solution and 40 c.c. of water, and titrated with a 0.6% hydrochloric acid solution of ferric chloride containing 0.7344 gm. of iron per litre. In a similar beaker 100 c.c. of 0.6% hydrochloric acid containing the same amount of thiocyanate as the liquid to be titrated, is treated with 0.1–0.2 c.c. of the ferric chloride solution, to serve as colour standard. The titration is complete when the supernatant liquid, above the whitish iron precipitate, viewed by reflected light, shows a reddish coloration equal to that of the standard, and persisting for 5 mins. The volume of ferric chloride solution required is reduced by that added to the standard. One c.c. of the ferric solution corresponds to 2 mgrms. P<sub>2</sub>O<sub>5</sub> or 0.8739 mgrm. of phosphorus present as phytin. The end-point is most distinct when the liquid contains not more than 10 mgrms. of phytin phosphorus. Substances other than phytin, present in extracts prepared as above, do not affect the results. The following percentages of phytin phosphorus were found in the products named:—maize, 0.19–0.20; cottonseed meal, 0.76–0.86; wheat shorts, 0.61; kafir, 0.23; wheat bran, 1.24; oats, 0.26; rice bran, 1.18; rice polish, 1.26; soya bean, 0.36; clover seed, 0.29. These amounts ranged from 52 to 89% of the total phosphorus present in the products, and from 74 to 97% of the phosphorus extracted by 1.2% hydrochloric acid. The method does not



appear to give satisfactory results with dried forage plants (soya bean, cow pea, velvet bean, and alfalfa) on account of the dark colour of the extracts.—J. H. L.

*Humin formed by the acid hydrolysis of proteins : Origin of the —. Hydrolysis in presence of formaldehyde.* R. A. Gortner and G. E. Holm. Paper No. 81. Journal Series of Minnesota Agric. Expt. Station. J. Amer. Chem. Soc., 1917, 39, 2177-2501.

THE authors first investigated the influence of formaldehyde on the hydrolysis of fibrin and gelatin by prolonged boiling with 20% hydrochloric acid (see Gortner, this J., 1916, 1030). The products were analysed by Van Slyke's methods (this J., 1911, 771; 1915, 1110). With increasing amounts of added formaldehyde (trioxymethylene), the curve representing the amounts of acid-insoluble humin nitrogen formed in the experiments with fibrin, rose sharply to a maximum and then fell again, the acid-soluble humin nitrogen rose somewhat less rapidly to a much higher maximum and then fell, whilst the ammonia fraction first decreased and then increased steadily and rapidly with larger additions of formaldehyde. In the experiments with gelatin, which unlike fibrin contains no cystine, tyrosine, nor tryptophane and only traces of histidine, formaldehyde had no influence on humin formation; the amount of insoluble humin was in all cases extremely small, and that of soluble humin was much smaller than with fibrin. With increasing amounts of formaldehyde the ammonia fraction increased rapidly, as with fibrin, but without any preliminary decrease. Addition of cystine, histidine, and tyrosine, with or without formaldehyde, had no influence on the formation of insoluble humin in gelatin hydrolysis; tyrosine increased the soluble humin, which however was not black but reddish-yellow and therefore not the true humin of protein hydrolysis. Addition of tryptophane, on the other hand, increased the production of insoluble humin even without formaldehyde; and in presence of increasing amounts of the latter the production of insoluble humin increased to a maximum and then decreased as in the case of fibrin. Indole behaved like tryptophane in this respect but pyridine had no influence on humin formation. When the quantity of added formaldehyde was such as to produce the maximum amount of insoluble humin, the nitrogen of the latter corresponded approximately with that of the tryptophane originally present, and half of it was found to be amino-nitrogen. The authors conclude (see this J., 1916, 1030) that the formation of black insoluble humin in protein hydrolysis by acids, is due to interaction between tryptophane and some as yet unidentified aldehyde or ketone, and the only part which any of the other known amino-acids has in humin-formation is perhaps to furnish some of their nitrogen to the humin fraction either through adsorption or occlusion (cp. Roxas, this J., 1916, 1171). The amino-group of tryptophane is not involved in the primary reaction by which insoluble humin is formed; probably it is the  $\alpha$ -position of the indole nucleus which is reactive. The initial fall in the so-called ammonia fraction observed in the experiments with fibrin is probably due to the removal in the insoluble humin, of some compound (tryptophane), which, when no aldehyde is present, contributes nitrogen to the ammonia fraction. The rapid rise in the ammonia curve with larger additions of formaldehyde, is due to the de-amination of amino-acids and the formation of volatile alkaline compounds other than ammonia, the nature of which is still under investigation.—J. H. L.

## PATENTS.

*Cereals, pulses or other grain : Process for treating — for edible purposes.* T. W. Rees, and The Eastern Flour Co., Ltd., Staines, Middlesex. Eng. Pat. 110,945, Nov. 9, 1916. (Appl. No. 16,070 of 1916.)

CEREALS, pulses, or other grain are steeped, in their natural state with their skins or husks on, in an alkaline solution, e.g., of sodium bicarbonate in the proportion of about 1 lb. per gallon of water. When a sufficient quantity of liquid has been absorbed, the material is sprayed with or immersed in a suitable acid solution other than sulphurous acid, e.g., sulphuric or tartaric acid at a strength of about 1 oz. per gallon of water. The material is then immediately dried by heat in a revolving drying machine until the moisture has been evaporated and the natural flavour modified. After cooling, the husks will readily leave the kernel, which may be ground to flour or otherwise utilised.—J. F. B.

*Food products consisting of globules of starchy material and methods of preparing same.* A. E. White, London. From Kellogg Toasted Corn Flake Co., Battle Creek, Mich., U.S.A. Eng. Pat. 111,119, Aug. 10, 1916. (Appl. No. 11,309 of 1916.) (See Eng. Pat. 18,916 of 1913; this J., 1913, 920.)

STARCHY material is mixed with water and sweetening and seasoning substances, with or without the addition of flavouring or colouring matters, to form a moist friable cake. This is granulated into small flaky particles, and these are rolled and shaken in a rotating drum, which is also subjected to a jarring action, until round globules are formed, and these are cooked until sufficiently gelatinised, and then rapidly heated by dry heat until puffed up into a light porous product, which, if desired, may be toasted by the further application of heat.—C. A. M.

*Fruit and vegetables : Preservation of —.* D. Chambers, Chideock, Dorset. Eng. Pat. 111,112, Nov. 13, 1916. (Appl. No. 16,223 of 1916.)

A room for the preservation of fruit and vegetables is constructed with a raised perforated floor of sheet iron, steel, or tin above a surface of earth which has been dug and dressed with a mixture of lime, salt, sand, and crude pitchblende, "which distributes to the contents of the fruit room mild currents of emanations of magnetism, electricity or radium." The walls of the room are coated with a mixture of lime, salt, cloves, allspice, and size, and wooden or metal rods are provided on which the fruit or vegetables may rest or be suspended. Apples and the like are wired, and enclosed in germ-proof air-tight paper bags, which are enclosed in inverted sterilised jars or boxes. A steam box with a glass near the top is provided for sterilising fruit or jars of fruit, and this contains a kettle, lamp, and trays for holding fruit skewers, etc.—C. A. M.

*Cereal pastes : Drying room for —.* N. D'Agostino, Assignor to A. Bizzarri and R. Rauli, Los Angeles, Cal. U.S. Pat. 1,244,429, Oct. 23, 1917. Date of appl., Sept. 5, 1916.

THE apparatus consists of a casing divided into compartments communicating with each other, one of them being adapted to contain the material to be dried and having an opening to the outside, capable of being regulated, and the other being closed and containing a blower with a deflector for directing the air currents from the one compartment into the other through the communicating opening.—J. F. B.

*Vegetable drying oil [and cattle food].* U.S. Pat. 1,244,521. See XII.

## XIXB.—WATER PURIFICATION; SANITATION.

*Hardness in water: Note on the Blacher method for the determination of —.* A. S. Behrman. Philippine J. Sci., 1916, 11A, 291—293.

VARIOUS points in the Blacher method (this J., 1913, 158) were examined. The presence of as much carbon dioxide as 28 parts per million was not found to interfere with obtaining correct results, and higher amounts were easily reduced by the aspiration of air for 5 mins., which was as efficient as boiling. Amounts of sodium chloride corresponding to 2000 parts of chlorine per million did not affect the end-point. It was found desirable to dilute a water with a total hardness of over 250 parts per million about five times. There are two end-points to the titration in presence of phenolphthalein, the first being a faint pink, the second an intense pink; the latter point gives correct results. The following method of carrying out the test is recommended:—The free carbon dioxide in 100 c.c. of the sample is determined by titration with sodium carbonate in presence of phenolphthalein. If this result exceeds 25 parts per million, the temporary hardness is determined on another quantity of 100 c.c. by aspirating for 10 mins. and titrating with N/10 sulphuric acid, using a drop of dimethylaminoazobenzene as indicator. A few drops of N/10 sulphuric acid are then added in excess, air aspirated through the solution for 5 mins., and 1 c.c. of phenolphthalein (1%) added, followed by N/10 alcoholic caustic potash until a slight pink colour is noticed. Titration is then carried out with N/10 potassium palmitate (made by neutralising an alcoholic glycerin solution of palmitic acid, and then standardising with saturated lime water) until an intense pink is obtained. This result gives the total hardness.—J. H. J.

*Water; Identification and determination of zinc in —.* R. Meldrum. Chem. News, 1917, 116, 271—272.

Two hundred c.c. of the water is treated with 1 c.c. of concentrated hydrochloric acid, evaporated to 80 c.c., and 1 c.c. of concentrated ammonia is added; the precipitated iron and aluminium hydroxides, if present, are filtered off, dissolved in a small quantity of hydrochloric acid, reprecipitated with ammonia, and again filtered off. The combined filtrates are diluted to 100 c.c. in a graduated cylinder, and 2 c.c. of concentrated ammonium sulphide is added. If, after the lapse of 15 mins., a distinct opalescence or turbidity develops, zinc is present in the water and its quantity may be determined by comparison with the turbidity produced by a known amount of zinc under the same conditions. The method will detect about 1 part of zinc in 100,000 parts of water.—W. P. S.

*Dakin or Carrel-Dakin (hypochlorite antiseptic) solution; The —.* I. Griffith. Amer. J. Pharm., 1917, 89, 497—500.

The original Dakin solution consisted of hypochlorite in conjunction with boric acid. This preparation, however, was found to have irritating properties when applied to wounds and has been modified by Daufresne by the substitution of sodium bicarbonate for the boric acid. The solution is made from bleaching powder, according to the following recipe, which is calculated on the basis of 30% of available chlorine in the bleaching powder:—151 grms. of bleaching powder is placed in a 12-litre bottle with 5 litres of water, the mixture is shaken vigorously for a few minutes and allowed to stand, with intermittent shaking, for 12 hours. 77 grms. of dry sodium carbonate

and 64 grms. of sodium bicarbonate are dissolved in 5 litres of water and added to the mixture in the bottle. After the calcium carbonate has settled, the solution is siphoned off through a filter and is ready for use. The liquid must contain between 0.15 and 0.50% of sodium hypochlorite and must give no pink coloration when 0.2 gm. of phenolphthalein is sprinkled over its surface. If the available chlorine in the bleaching powder be different from 30%, the above quantities of ingredients must be corrected accordingly.

—J. F. B.

### PATENT.

*Insecticidal composition [from smelter fume dust].* W. R. Macklind and E. C. Holton, Assignors to The Sherwin Williams Co., Cleveland, Ohio. U.S. Pat. 1,211,153, Oct. 23, 1917. Date of appl., Oct. 17, 1916.

RECOVERED smelter fume dust containing arsenic is mixed with calcium oxide in amount less than one-half (e.g., 30%) of the arsenic contained in the dust.—J. F. B.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Hydrogen cyanide: New plant, Isopyrum fumarioides, yielding —.* M. Mirande. Comptes rend., 1917, 165, 717—718.

THE Siberian plant, *Isopyrum fumarioides*, L. (*Ranunculaceae*), is one of the most strongly cyanogenetic known; a 100-grm. sample, representing all parts of the plant, yielded 0.249 gm. of hydrogen cyanide on digestion with water for several hours at 25°—30° C. A much smaller amount, 0.042 gm., had previously been obtained from *I. thalicteroides*, the only European plant of the same genus.—J. H. L.

*Biochemical synthesis of diglucosides of polyhydric alcohols; Attempted —.*  $\beta$ -Diglucoside of glycol. E. Bourquelot and M. Bridel. Comptes rend., 1917, 165, 728—730.

EMULSIN was allowed to act for 2 months on an aqueous solution of ethylene glycol (1 mol.) and dextrose (2 mols.). Synthesis of hexobioses occurred (cp. this J., 1915, 161), but the change in rotatory power indicated other reactions as well, the products of which, however, could not be isolated.—J. H. L.

*Cyanuric acid; Identity of — with the so-called "tetracarbonimide."* E. H. Walters and L. E. Wise. J. Amer. Chem. Soc., 1917, 39, 2172—2177.

SHOREY and Walters (this J., 1915, 239) found in soils a compound having the properties of the "tetracarbonimide" obtained by Scholtz by the oxidation of uric acid by hydrogen peroxide in alkaline solution (Ber., 1901, 34, 1130). The authors have repeated Scholtz's experiments, and find that the "tetracarbonimide" formed is in reality cyanuric acid (cp. this J., 1917, 1057). By modifying the method of oxidation employed by Scholtz, they have isolated another product, probably carbonylurea,  $\text{CO}(\text{NH}\cdot\text{CO}\cdot\text{NH})_2$ , which no doubt is an intermediate product in the oxidation of uric acid to cyanuric acid, for on heating with alkalis, or alone above 227° C., it decomposes into the latter and ammonia.—J. H. L.

*Contact catalysis. Studies in decomposition of ethyl alcohol.* C. J. Engelder. J. Phys. Chem., 1917, 21, 676—704.

BANCROFT (this J., 1917, 1169) has recently proposed the view that adsorption of the reagents or of the reaction products represents the mechanism



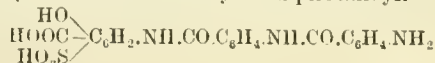
of so-called "contact catalysis." Ethyl alcohol can undergo catalytic decomposition in two different ways, giving rise either to ethylene and water or to aldehyde and hydrogen. Experiments are now described in which alcohol vapour was passed over various catalysts between 380° and 500° C., and the results are compared with those obtained under similar conditions with a mixture of alcohol and water vapours. The catalysts used were aluminium oxide, silica, zirconium oxide, and titanium dioxide; under normal conditions these cause the simultaneous but unequal occurrence of the dehydration and dehydrogenation processes, aluminium oxide, indeed, assisting the former change almost exclusively. In every case, however, the admixture of water vapour with the alcohol causes a relative retardation of the dehydration process and therefore a decrease in the proportion of ethylene and an increase in the proportion of hydrogen in the gaseous products. This result is explained by the partial adsorption of the additional water vapour by the catalyst and a consequent smaller adsorption of water from the alcohol molecules. A similar but reverse effect was observed on submitting a mixture of alcohol vapour and hydrogen to the catalytic influence of titanium oxide, the dehydrogenation process in this case being partially suppressed. Titanium oxide catalyses very slightly the formation of ethane from a mixture of ethylene and hydrogen near 400° C. and does not induce appreciably the decomposition of ethylene into methane nor of aldehyde into carbon monoxide and methane. At 210° C. aluminium oxide converts alcohol into ether, and the formation of ethylene at higher temperatures is due to the further dehydrating action of the alumina on the ether; the production of acetaldehyde in the presence of alumina occurs of course by direct dehydrogenation of the alcohol molecule. Finely-divided nickel is well known to be a very active catalyst for the dehydrogenation of alcohol, but it also tends to induce the further decomposition of acetaldehyde into methane and carbon monoxide. So powerful is nickel as a dehydrogenating catalyst that alcohol when submitted to the influence of a mixture of alumina and nickel at 380° C. undergoes decomposition almost entirely by dehydrogenation into acetaldehyde. The most active form of alumina was obtained by treating aluminium nitrate solution with ammonia and dehydrating the washed and carefully-dried precipitate at 240° C.; the silica was best produced by pouring an aqueous solution of sodium silicate into dilute hydrochloric acid and heating, the resulting jelly being subsequently broken up, washed, dried, and finely powdered; the zirconia was prepared by calcining the nitrate at a low heat, and the titania by dehydration of the precipitated tetrahydroxide at 150° C.—D.F.T.

*The Dakin or Carrel-Dakin [hypochlorite anti-septic] solution.* Griffith. See XIXB.

#### PATENTS.

*Ureas or thioureas of the aromatic [benzene or carbazole] series; Process for producing* —. Farbenfabr. vorm. F. Bayer und Co., Leverkusen, Germany. Eng. Pat. 8591, June 10, 1915. Under Int. Conv., June 11, 1914. Addition to Eng. Pat. 9472 of 1914 (this J., 1914, 825).

AMINO-ACIDYL amino sulphonics of the benzene or carbazole series are treated with phosgene or thiophosgene or other substance capable of introducing the CO or CS group. The ureas from *m*-aminobenzoyl - *HO*-aminobenzoyl - sulphosalicylic acid



and from *bis* [*m*-aminobenzoyl-] aminocarbazoledisulphonic acid are specially claimed. The products possess valuable therapeutic properties.—F. W. A.

*Acetaldehyde; Method of producing* —. E. F. Scheller, Lorsbach in the Taunus, Germany. U.S. Pats. (A) 1,244,901 and (B) 1,244,902, Oct. 30, 1917. Date of appl., June 26, 1917.

(A) PURIFIED acetylene, freed from phosphorus compounds, is mixed with steam, and the mixture is passed at an elevated temperature over a catalyst consisting of oxides or combinations of oxides. (B) The exhausted catalyst, *e.g.*, molybdic acid, is regenerated and freed from catalytic poisons by treatment at a high temperature with a current of gas containing oxygen, *e.g.*, air.

—F. W. A.

*Halogenation of gaseous hydrocarbons of the paraffin series or of aliphatic hydrocarbons and their halogen derivatives.* C. W. Bedford, Tallmadge, Assignor to The Goodyear Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,245,553, Nov. 6, 1917. Date of appl., June 29, 1914.

IN chlorinating gaseous saturated aliphatic hydrocarbons, *e.g.*, methane, the reaction is induced in direct and intimate contact with a cooling agent capable of removing halogen acids, *e.g.*, ice, in order to limit the reaction and remove the lower chlorinated products by condensation.—F. W. A.

*Ethers; Process of manufacturing paraffin* —. B. S. Lacy, Sewaren, N.J., Assignor to The Roessler and Hasslacher Chemical Co., New York, N.Y. U.S. Pat. 1,245,742, Nov. 6, 1917. Date of appl., July 21, 1914.

A MIXTURE of the vapours of a hydrocarbon chloride (chloro-hydrocarbon) and an inert gas is treated with a basic oxide, *e.g.*, calcium oxide, at a temperature of from 250° to 450° C. In a cyclic process for the manufacture of ethyl ether, a mixture of 1 vol. of chlorine with not less than approximately 3 vols. of ethane is passed at 300°—550° C. through a suitable reaction vessel, the reaction products are cooled, and the hydrochloric acid is separated by means of water; the moisture is removed from the remaining mixture of ethyl chloride and excess of ethane, and the dry gas mixture is passed over calcium oxide at a temperature of approximately 250°—450° C.; the reaction product is cooled, compressed, and cooled whilst compressed, and the liquefied ether separated from the excess of ethane, which may be used again in the process.—F. W. A.

*Production of aminoalkyl esters and alkylaminoalkyl esters of *p*-aminobenzoic acid.* Eng. Pat. 111,328. See III.

*Process of producing toluene [and propane from turpentine].* U.S. Pat. 1,244,444. See III.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photographic plate; Physical characteristics of the elementary grain of a* —. M. B. Hodgson. Communication No. 56 from the Eastman Kodak Research Laboratory. Brit. J. Phot., Oct. 19, 1917, 532—534.

THE author has examined the grain of photographic plates before, during, and after development. Silver bromide occurs as more or less regular semi-transparent crystals, the most common form being the tetrahedron; the size varies between 0.2μ and 8.5μ in the plates examined, and the average size ranges from 1μ to 4μ. In some cases the developed grain retains practically the size and shape of the halide grain, but more

generally there is expansion and distortion during development, which does not occur when the film is merely swollen with water. It is suggested that this is due to a strain of the halide grain produced by the drying of the surrounding gelatin, the strain being released when the coherence of the grain is removed by its reduction to metal, or that gelatin is actually present in the body of the halide grain itself and produces disintegration by swelling. Photomicrographs are shown of the various steps of a Hurter and Driffield step-negative developed for a uniform time, and of strips with uniform exposure and varying development; only in the shorter developments of the latter series is a greater concentration of silver grains apparent in the upper layers of the film. Scheffer's phenomenon of the throwing out of a feeder from a grain during development was observed in only one case of a very thin film on a microscope slide; it is explained as due to stream lines in the spreading of the developer through the film. There is evidence of the development of a grain at several nuclei.—B. V. S.

*Development of roll-film, film-packs, plates, and papers; High temperature*——. J. I. Crabtree. Communication No. 62 from Eastman Kodak Research Laboratory. Brit. J. Phot., Nov. 2, 1917, 555—559.

IN the development, etc., of plates or films under tropical conditions, i.e., where the solutions and washing water may be at a temperature as high as 95° F. (35° C.), the chief difficulty met with, apart from the actual melting of the swollen gelatin film, is reticulation. This is very easily produced with swollen gelatin by a change of temperature or by an osmotic change such as might be produced by transfer from a solution of one concentration to one of another, or by transfer to an acid solution. It is necessary therefore to keep all solutions at about the same temperature and also to avoid abnormal swelling of the gelatin during any part of the process. The latter may be achieved by (1) hardening before development, (2) hardening during development, or (3) temporary hardening during development and a permanent hardening afterwards. (1) The author obtained better results by the use of formalin than with alum or chrome alum, but with old films weak, foggy negatives were produced. (2) The method of Saal and others of using formalin in the developer, either with or without alcohol or acetone, was found to produce fog, and the use of alum or chrome alum in alkaline developers was of course impossible. (3) The author found this method most generally applicable. Addition of alcohol or acetone to the developer in sufficient quantity to reduce the water absorption appreciably was not possible with a celluloid base. The addition of large quantities of certain salts such as sodium sulphate or sulphite, with addition of bromide to reduce fog, is fairly efficient with most developers, but the best developer for the purpose was found to be:—*p*-aminophenol hydrochloride, 7 grms., sodium sulphite, 50 grms., sodium carbonate, 50 grms., and water up to 1000 c.c., which prevents swelling of the gelatin and develops quickly and cleanly. If the temperature does not exceed 75° F. (24° C.) no further hardening is necessary, the usual acid fixing bath being used; up to 85° F. (29° C.) a chrome alum hardening and fixing bath should be used and at higher temperatures a formalin hardening and fixing bath. This method is applicable to both plates and films; much fewer precautions are necessary with papers, the film of which is generally fairly hard, but it is preferable to use extra bromide in development, an acetic acid stop-bath, and a potash alum hardening and fixing bath.—B. V. S.

*Sepia tones by direct development*. A. Nietz and K. Huse. Communication No. 53 from the Eastman Kodak Research Laboratory. Brit. J. Phot., Sep. 28, 1917, 497—499.

THE development of very fully exposed printing papers with certain strongly restrained developers tends to give coloured images. The authors have succeeded in producing good sepia tones on "Artura" paper by this process. They find the best developer to be chloroquinol, 5 grms., sodium sulphite, 30 grms., sodium carbonate, 16 grms., potassium bromide, 6 grms., potassium metasulphite, 6 grms., and water 1000 c.c. The colour of the image is due to the size of the silver grains and not to any staining process; the size of the grain increases with development, the colour passing from yellow through red and greenish-black to black. For this reason the exact concentration of the reducer in the developer is not of much consequence, but the concentrations of the alkali, the bromide, and the metasulphite, which control the speed of development, must be carefully observed in order to produce the best tone. Under definite conditions the tone is dependent on the time of development, and exposure must be regulated accordingly; it is from 75 to 100 times that required with the usual developers, a slight printing-out image being visible. The gradation of the paper is somewhat steeper, and softer negatives are required. The tone alters on fixing and drying and can only be properly judged on the dry print.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

### PATENT.

*Smoke-producing compound*. W. Arthur. Philadelphia, Pa. U.S. Pat. 1,244,940, Oct. 30, 1917. Date of appl., Dec. 6, 1916.

CADMIUM or a material containing cadmium is mixed with a solid oxidising agent, such as a nitrate, capable of supporting combustion, or a mixture of oxidising salts, with or without the addition of finely-divided aluminium or other metal burning with a bright light.—C. A. M.

## XXIII.—ANALYSIS.

*Alkali metals; The perchlorate method for the determination of*——. F. A. Gooch and G. R. Blake. Amer. J. Sci., 1917, 44, 381—386.

IF the volume of the washing solution (97% alcohol containing 0.1% of perchloric acid) is kept quite small, the solubility of the precipitated perchlorates is insignificant; it is unnecessary to saturate the solution with the substance to be washed. A single evaporation with a moderate excess of perchloric acid (0.1 c.c. for each 0.1 gm. of salt) is not sufficient to convert considerable quantities (0.3 gm. or more) of alkali chlorides completely into perchlorate; the treatment should be repeated. A double precipitation is required to separate large amounts (0.3 gm.) of insoluble perchlorates from sodium perchlorate. In the case of rubidium, the precipitate should be digested for 15 mins. with the washing solution. It is to be noted that perchloric acid has a distinct solvent action on glass.—W. P. S.

*Textile fibres; Use of*—— in microscopic qualitative chemical analysis. E. M. Chamot and H. I. Cole. J. Ind. Eng. Chem., 1917, 9, 969—971.

FROM a study of the method devised by Emich (this J., 1901, 1142; 1902, 498) the authors find that litmus silk of the right colour and intensity may be prepared by treating raw silk for 2 hours with 10% sodium hydroxide solution, washing it



free from alkali, and then dyeing in a 20% solution of commercial litmus or a 10% solution of purified litmus, acidified with 3 or 4 drops of sulphuric acid (1:4). The solution containing the silk is evaporated to a thick syrup and the silk then removed, washed in running water, neutralised with very dilute sodium hydroxide solution, and again washed thoroughly. By treating the dyed fibres with dilute acetic acid or sodium hydroxide solution and washing them in running water they are tinted red or blue. Applied to a drop of liquid under the microscope a dyed fibre is capable of detecting hydrochloric acid in  $N/4500$  solution, acetic acid in  $N/100$  solution, and sodium or potassium hydroxide or ammonia in  $N/200$  solutions. In like manner silk or viscose fibres may be dyed in a 0.5% or 2% solution of Congo red made alkaline with sodium hydroxide, and then pressed between filter papers. The fibres change colour with many organic acids in concentrations of 0.1 to 1 grm. per litre, and with mineral acids in concentrations as low as 0.01 grm. per litre. Hence they cannot be used to differentiate between organic and mineral acids.—C. A. M.

*Aluminium; Separation of*—*from iron by means of ether.* S. Palkin. J. Ind. Eng. Chem., 1917, 9, 951–953.

THE solution of the chlorides of iron and aluminium is evaporated to dryness on the water bath and the residue dried at about 120° C. for 30 mins., with occasional stirring. It is then moistened with 0.5 to 1 c.c. of alcoholic hydrochloric acid solution (25 to 35% HCl) and heated on the water-bath, eventually after the addition of 3 to 4 c.c. of absolute alcohol, until all salts have dissolved and have begun to crystallise out again. About 0.5 c.c. of alcoholic hydrochloric acid is added to the viscous solution, the flask warmed, and the mass treated with 30 c.c. of ether (sp. gr. 0.713 to 0.716 at 25° C.), which should precipitate the hydrated aluminium chloride in a granular form. Forty c.c. of ether is then added, and the solution filtered through a Gooch crucible, the contents of the flask being meanwhile washed with a mixture of 100 parts of ether with 2 parts of alcoholic hydrochloric acid to prevent ferric chloride drying on the white precipitate. The aluminium chloride precipitate is dissolved in water, the solution diluted to about 100 c.c., and, after the addition of about 5 grms. of ammonium nitrate, rendered just alkaline with ammonia (methyl red as indicator), and the precipitate separated, washed, and ignited in a covered crucible. The ethereal solution of iron is distilled, and the residue transferred by means of as little water as possible, together with some hydrochloric acid, to a weighed platinum dish, in which the liquid is evaporated to dryness. The residue of ferric chloride is converted into sulphate and finally to oxide.—C. A. M.

*Laboratory methods for benzol-recovery plant operation.* Sperr, jun. See IIA.

*Method of staining to distinguish between bleached and unbleached sulphite pulps.* Bright. See V.

*McLean-Van Slyke iodometric method for the titration of halides, in its application to chlorides.* McCracken and Walsh. See VII.

*Some compounds of lead.* [Detection of hydrogen peroxide and of lead.] Zotier. See VII.

*Direct determination of the hygroscopic coefficient [of soil].* Alway and others. See XVI.

*Preparation of ammonium citrate solution, and determination of insoluble phosphoric acid [in fertilisers].* Shuey. See XVI.

*Determination of phytin phosphorus in plant products.* Rather. See XIXA.

*Note on the Blacher method for the determination of hardness in water.* Behrman. See XIXB.

*Identification and determination of zinc in water.* Meldrum. See XIXB.

#### PATENTS.

*Anemometers.* South Metropolitan Gas Co., E. V. Evans, and J. S. G. Thomas, London. Eng. Pat. 111,015, Feb. 17, 1917. (Appl. No. 2333 of 1917.)

IN anemometers where the rate of flow of a current of gas is estimated by the cooling effect on an electrically heated wire, interference by decomposition of the gas by the hot wire or by the action of the gases on the wire, or by a deposit of soot or carbon on the wire, is obviated by coating or encasing the wire in an electrical non-conductor such as glass. The wire may be maintained below a red heat. By using two such wires, one immediately behind the other and, by suitable indicators, observing the difference of the temperature changes, the direction of the current of gas can be ascertained.—B. V. S.

*Viscometer.* C. Browning, jun., Sacramento, Cal. U.S. Pat. 1,244,025, Oct. 23, 1917. Date of appl., Aug. 16, 1913.

THE apparatus consists of a transparent tube pivoted on a standard and adapted to receive and discharge the liquid under examination. The tube may be jacketed. A sphere of greater specific gravity than the liquid and of smaller diameter than the bore of the tube, is placed in the tube, and means are provided for fixing the tube at any desired angle.—J. H. P.

*Apparatus for and process of estimating the volume of acid required to coagulate a given volume of india-rubber latex.* Eng. Pat. 100,591. See XIV.

#### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Arter, Grinding, etc., machines. 19,332, 19,333, 19,334. Dec. 31.

Baker and Prescott. Apparatus for subjecting articles to varying temperatures. 19,041. Dec. 22.

Bullock, and Fraser and Chalmers. Separating machines. 19,235. Dec. 29.

Cracknell, and Ransomes and Rapier. Refrigerating-apparatus. 18,875. Dec. 19.

Davis and Twigg. Gas-fired furnaces. 19,189 and 19,190. Dec. 28.

Laurie. Drying solid substances precipitated from solution in water, etc. 19,209. Dec. 29.

Menzies. Apparatus for treating liquids with gases. 18,870. Dec. 19.

Ross. Grinding-machines. 18,999. Dec. 22.

Wade (Davis). Refrigerating-machines. 19,348. Dec. 31.

##### COMPLETE SPECIFICATIONS ACCEPTED.

14,531 (1916). Barbet et Fils et Cie. See under XX.

15,477 (1916). Bostaph, and Bostaph Engineering Co. Retort. (111,871.) Dec. 28.

3689 (1917). Richardson and Hammond.

Combined condenser and heat interchanger. (111,961.) Dec. 28.  
12,735 (1917). Fairweather (Pacific Evaporator Co.). Drying apparatus. (111,991.) Dec. 28.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### APPLICATIONS.

Boorne. Wet-carbonising. 19,019. Dec. 22.  
Comp. pour la Fabr. des Compteurs et Matériel d'Usines à Gaz. Water-gas producers. 18,757. Dec. 17. (Fr., Dec. 19, 1916.)  
Drake, and Drakes, Ltd. Vertical retorts for manufacture of gas. 18,996. Dec. 22.  
Grocott. Furnaces for generating producer-gas. 18,815. Dec. 19.  
Harger. 19,052. *See under XXIII.*  
Nelson. Low-temperature distillation of coal, shale, etc. 18,983. Dec. 21.  
Pleasance. 18,744. *See under X.*  
Schuck, and Superior Oil and Process Co. 10,305. *See under XII.*  
Thaberg. Producing gas from moist fuel in furnaces, gas-producers, etc. 18,911. Dec. 20. (Sweden, June 1, 1915.)  
Thompson. 19,065. *See under XI.*  
Twynam. Production of artificial fuel and utilisation of peat. 19,245. Dec. 31.  
Walster and Walster. Generating coal gas in retorts. 18,770. Dec. 18.  
Williams and Williams. 19,168. *See under XXIII.*

### COMPLETE SPECIFICATIONS ACCEPTED.

12,579 (1916). O'Brien. Production of liquid fuel suitable for use in internal-combustion engines. (111,861.) Dec. 28.  
15,477 (1916). Bostaph, and Bostaph Engineering Co. *See under I.*  
17,698 (1916). Wellington and Etherton. Treatment of refuse liquors from coke ovens and gas works. (111,883.) Dec. 28.  
251 (1917). Heyl. Liquid hydrocarbons. (111,933.) Dec. 28.  
252 (1917). Heyl. Production of carburetted water-gas. (111,931.) Dec. 28.  
3196 (1917). Hammond and Richardson. *See under III.*  
3963 (1917). Wollaston. Gas-producers. (111,964.) Dec. 28.

## III.—TAR AND TAR PRODUCTS.

### COMPLETE SPECIFICATIONS ACCEPTED.

883 (1917). Aylsworth, and Savings Investment and Trust Co. Production of phenol. (103,664.) Dec. 28.  
3196 (1917). Hammond and Richardson. Apparatus for distillation and dehydration of coal tar, mineral or vegetable oils, and the like, and debenzolising of wash and other oils and liquids. (111,956.) Dec. 28.

## IV.—COLOURING MATTERS AND DYES.

### COMPLETE SPECIFICATION ACCEPTED.

17,982 (1916). Sokal (Kalle und Co.) Production of vat dyestuffs. (111,901.) Dec. 28.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### APPLICATIONS.

Brown and Cowan. Apparatus for treating paper pulp, etc. 18,952. Dec. 21.  
Dubourg. 18,662. *See under XVIII.*  
Heys. Beating-machines for making paper-pulp. 18,997. Dec. 22.

Johnston and Sutherland. Treatment of stalk fibres for removal of gum or mucilage. 19,208. Dec. 29.

Russell and Wood. Paper-making machines. 18,938. Dec. 21.

Stevenson. Acetylcellulose. 19,049. Dec. 21.

### COMPLETE SPECIFICATIONS ACCEPTED.

3249 (1917). Korselt. Increasing the strength and elasticity and preventing or decreasing the subsequent weakening of non-loaded raw or boiled fibres in silk. (104,684.) Dec. 28.

10,676 (1917). Gadsden. Method of water-proofing and rendering impervious to dust, bags and sacks. (109,252.) Dec. 28.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### APPLICATIONS.

Jackson. Cloth dyeing, bleaching, etc., machines. 18,933. Dec. 21.

Nevill. Manufacture of printing, etc., surfaces used in calico or wall-paper printing and drying machines. 19,251. Dec. 31.

### COMPLETE SPECIFICATION ACCEPTED.

6302 (1917). Tidswell. Machines for printing piece goods. (111,970.) Dec. 28.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATIONS.

Dreyfus. 19,353 and 19,351. *See under XX.*

Maxted. Synthesis of ammonia. 18,931. Dec. 21.

Nicholson. Utilisation of complex solutions of sodium and ferrous salts. 19,061. Dec. 24.

Toniolo. Manufacture of nitric acid. 19,139. Dec. 27.

### COMPLETE SPECIFICATION ACCEPTED.

16,555 (1916). Grossmann. Utilisation of nitro cake. (111,875.) Dec. 28.

## VIII.—GLASS; CERAMICS.

### APPLICATION.

Dauner. Forming sheet glass. 19,311. Dec. 31.

### COMPLETE SPECIFICATION ACCEPTED.

17,791 (1916). Cox. Method of gathering glass. (111,890.) Dec. 28.

## IX.—BUILDING MATERIALS.

### APPLICATION.

Andrews and Evans. Substitute for wood. 19,058. Dec. 28.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

### APPLICATIONS.

Armstrong, Whitworth, & Co., Rowden, and Trevelyan. Production of steel from scrap. 19,220. Dec. 29.

Armstrong, Whitworth, & Co., Rowden, and Trevelyan. Production of steel. 19,221. Dec. 29.  
Auld and Tennick. Casting metals. 18,986. Dec. 21.

Basset. Treatment of ores in blast furnaces. 18,974. Dec. 21. (Fr., Dec. 22, 1916.)

De Ducezele et Cie. Formation of adherent metal deposits on common metals. 18,756. Dec. 17. (Belgium, July 30.)

Leech and Slater. Electrolytic preparation of metals and alloys in the form of paste, sludge, or sponge. 19,170. Dec. 28.



Lumsden. Casting ingots. 18,743. Dec. 17.  
Marks (U.S. Alloys Corporation). Treatment of iron and steel. 19,313. Dec. 31.

Marks (U.S. Alloys Corporation). Alloy of iron, titanium, and silicon, and process for the production thereof. 19,314. Dec. 31.

Mead and Wood. Metallurgical heating-furnaces. 18,719. Dec. 17.

Parkinson Stove Co., and Thompson. Protection of iron and steel surfaces. 19,057. Dec. 24.

Pleasance. Apparatus for screening and washing gold, sand, coal, etc. 18,744. Dec. 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,248 (1916). Armstrong, Whitworth, & Co., and Rowden. Manufacture of steel. (111,916.) Dec. 28.

18,329 (1916). Iyer. Coating iron with brass. (112,045.) Dec. 31.

6111 (1917). Cusquel and Goupil. Devices for pulverising molten or liquid metals. (112,085.) Dec. 31.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Chance and Hunt, Cunningham, Mott, and Smith. Electrolytic processes. 18,900. Dec. 20.

Chem. Fabr. Weissenstein, and Walter. Diaphragms for electrolytic cells. 19,082. Dec. 24. (Austria, Dec. 19, 1916.)

Coates, and Watford Electric and Manufacturing Co. Electric arc furnaces. 18,876. Dec. 19.

Leech and Slater. 19,170. *See under X.*

Thompson. Manufacture of carbon electrodes. 19,065. Dec. 24.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Bendixen. Methods for keeping fat-globules in emulsions apart. 19,094. Dec. 27.

Buchner. Washing and cleansing agents. 18,966 and 19,039. Dec. 21 and 22.

Davidson. Machines for forming oil cakes. 19,323. Dec. 31.

Schuck, and Superior Oil and Process Co. Process of purifying oily material. 19,305. Dec. 31.

#### COMPLETE SPECIFICATION ACCEPTED.

3196 (1917). Hammond and Richardson. *See under III.*

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATION.

Craven, and Yorkshire Dyeware and Chemical Co. Linoleum manufacture. 18,891. Dec. 20.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

François. Method of impregnating and covering tissues with balata without the use of solvents. 19,224. Dec. 29.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,991 (1916). Ransford (Icken). Manufacture of rubber and other products from latices and treatment of raw, inferior, or scrap rubbers or rubber gums. (111,906.) Dec. 28.

17,992 (1916). Ransford (Icken). Process for purifying raw, inferior, or scrap rubbers. (111,907.) Dec. 28.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATION.

Aktionernoje Obszestwo Kaoutshouk, and Zirg. Manufacture of a substitute for leather, etc. 18,801. Dec. 18.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

Robinson. Manufacture of fertilisers. 18,802. Dec. 18.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATIONS.

Feldenheimer and Plowman. Starch substitutes. 18,749. Dec. 17.

Harris. Starch and farina settling-vats. 18,956. Dec. 21.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATIONS.

Barbet et Fils et Cie. Apparatus for treatment of phlegms or wines or fermented musts. 19,236. Dec. 31.

Dubourg. Manufacture of alcohol from wood or any cellulosic substance. 18,862. Dec. 19. (Fr., Oct. 21, 1916.)

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Fellowes and Hyatt. Insecticides. 19,008. Dec. 22.

Krogh. Production of colourless soluble albumen of blood. 18,790. Dec. 18.

Malcolm. Treatment of fruit and vegetable refuse. 18,831. Dec. 19.

Pearson. Production of protein substances. 18,866. Dec. 19.

#### COMPLETE SPECIFICATION ACCEPTED.

8394 (1917). Alcock. Apparatus for treating by heat coffee, cereals, and like materials. (112,100.) Dec. 31.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Coley and Wilbraham. Manufacture of bismuth tribromphenylate. 19,017. Dec. 22.

Dreyfus. Manufacture of acetic acid from acetaldehyde. 19,353 and 19,354. Dec. 31. (Fr., Feb. 22.)

#### COMPLETE SPECIFICATION ACCEPTED.

14,531 (1916). Barbet et Fils et Cie. Apparatus for recovering ether and alcohol vapours contained in the air of factories. (101,875.) Dec. 28.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,104 (1916). Edwards. Photographic films. (111,913.) Dec. 28.

18,631 (1916). Petrelius. Manufacture of colour prints. (103,120.) Dec. 28.

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATION.

Adde. Explosives. 18,906. Dec. 20. (Ger., Dec. 20, 1916.)

### XXIII.—ANALYSIS.

#### APPLICATIONS.

Harger. Gas calorimeters. 19,052. Dec. 24.  
Williams and Williams. Appliance for giving warning of the presence of combustible gases or vapour in the atmosphere. 19,168. Dec. 28.

**I.—GENERAL; PLANT; MACHINERY.**

*Fire protection in industrial plants.* C. W. Johnson. J. Amer. Inst. Metals, 1917, 11, 139—147.

THE general principles laid down for fire protection in industrial plants are: (1) everything possible must be done to prevent the start of a fire; (2) positive arrangements so that the start of a fire may be known at once and announced; (3) the provision of trained men with proper equipment and means for bringing them on the scene without loss of time, whatever the period when the fire may start. Rules for the prevention of fires are summarised. The following are dealt with: water supply, sprinklers, hydrants, records, hose, hose carts, chemical engines, hand portable fire extinguishers, hose house and boxes, fire alarm systems, fire pumps, fire escapes, aisles, inflammable materials, refuse materials. Weekly inspections should be arranged to ensure that inflammable and refuse materials, such as petroleum spirit, alcohol, soldering flux, varnish, shellac, linseed oil, waste, wiping rags, etc., are kept in minimum quantities only throughout the works and then only in safety cans or receptacles with covers. All corners, storerooms, etc., in out-of-the-way places should be inspected daily for cleanliness and to ensure that where inflammable materials are stored, every precaution has been taken to prevent fire; for example, canvas-duck treated with linseed oil should be stacked so that there is ample ventilation all around and through the piles, air spaces being provided at 1-inch intervals throughout the height of the pile. All heating pipes should be cleaned outside from dust at least monthly. All motors should be blown out at least monthly. Shields of paper or other inflammable material should not be allowed on incandescence lamps. All wiring should be removed as it becomes obsolete.—B. N.

**PATENTS.**

*Filter: Automatic self-cleaning*—. O. Best, San Francisco, Cal. U.S. Pat. 1,246,850, Nov. 20, 1917. Date of appl., Apr. 24, 1916.

LIQUID is forced under pressure into the top of a filtering chamber and passes through a filtering medium at the bottom into a storage chamber below; the two chambers communicate by a vertical pipe having its upper end in the bottom of the filtering chamber below the filtering medium and its lower end near the bottom of the storage chamber. Air is thereby compressed in the storage chamber to a pressure normally less than that in the filtering chamber, and an outlet from the filtering chamber may be controlled so as to reduce the pressure in this chamber and thus cause liquid to flow back from the storage chamber to clean the filter.—W. F. F.

*Refrigeration: Process of*—. A. W. Browne, Ithaca, N.Y., and R. P. Nichols, Philadelphia, Pa. U.S. Pat. 1,246,866, Nov. 20, 1917. Date of appl., Dec. 3, 1912.

ANHYDROUS ammoniated copper sulphate is heated to a temperature and at a pressure at which ammonia is given off, but below the fusion or decomposition point of the sulphate. The ammonia is condensed to a substantially anhydrous liquid, which is passed through refrigerating passages, and the gaseous ammonia then drawn off at reduced pressure and passed over the residual anhydrous copper sulphate. The original compound is thus re formed, and the cycle of operations is repeated.—W. F. F.

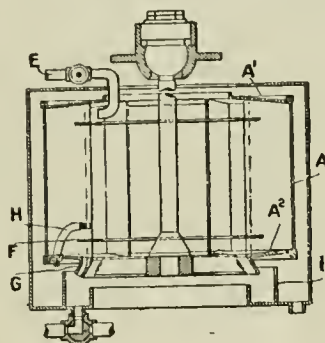
*Evaporator.* P. B. Sadtler, Chicago, Ill. U.S. Pat. 1,246,939, Nov. 20, 1917. Date of appl., Nov. 19, 1914.

THE liquid to be evaporated is forced by a pump into the conical lower end of a steam drum containing vertical heating tubes for the liquid. The liquid passes from the top of the steam drum down through the annular space formed by the outer casing, which has a conical lower end terminating in an outlet pipe leading back to the pump. A valved outlet is provided between the pressure side of the pump and the inlet chamber of the drum, and a valved by-pass from the inlet chamber around the valve in the outlet pipe.

—W. F. F.

*Centrifugal separator.* D. B. Weston, Sharon, Mass. U.S. Pat. 1,247,473, Nov. 20, 1917. Date of appl., Nov. 28, 1913.

THE top and bottom members, A<sup>1</sup>, A<sup>2</sup>, of a rotating container, A, are provided with large central openings, to permit access to the interior and



discharge of the solid material through the bottom. Liquid to be treated is fed by the pipe, E, into the top of the separator, and an annular baffle-plate, F, is provided having its edges overlapping the discharge outlet, G, for the lighter separated liquid. A discharge outlet, H, is provided for the heavier separated liquid, having its inlet end above the baffle-plate, F, and at a greater radial distance from the centre than the outlet, G. The container is surrounded by a casing having a lower central opening of the same diameter as the opening in the container, A, and is provided with an annular partition, I<sup>1</sup>, forming separate annular collecting troughs for the separated liquids.—W. F. F.

*Binding and cohering composition.* R. F. Tompkins, New York. U.S. Pat. 1,247,595, Nov. 20, 1917. Date of appl., Feb. 11, 1916.

A BINDING composition is composed of a mixture of a carbonaceous extract of tan bark liquor containing a hygroscopic glucoside, sodium silicate, and water.—W. F. F.

*Separating from a stream of liquid, solids held in suspension; Apparatus for*—. A. Ross, London. Eng. Pat. 111,509, Nov. 24, 1916. (Appl. No. 16,884 of 1916.)

*Drying machines.* E. C. R. Marks, London. From The Universal Drying Machine Co., Chicago, U.S.A. Eng. Pat. 111,515, Nov. 27, 1916. (Appl. No. 16,993 of 1916.)

SEE U.S. Pat. 1,213,962 of 1917; this J., 1917, 329.



*Separating solids of different specific gravities; Process for —.* W. J. Mellersh-Jackson, London. From Virginia Haloid Co., New York. Eng. Pat. 111,638, May 29, 1917. (Appl. No. 7703 of 1917.)

SEE U.S. Pats. 1,244,884 and 1,244,885 of 1917; this J., 1918, A 1.

*Refrigeration; Process of —.* J. C. Bertsch, Edgewood Park, Pa. U.S. Pat. 1,246,849, Nov. 20, 1917. Date of appl., Sept. 17, 1915.

SEE Eng. Pat. 101,544 of 1916; this J., 1917, 955.

*Centrifugal separator.* D. B. Weston, Sharon, Mass. U.S. Pat. 1,247,472, Nov. 20, 1917. Date of appl., Nov. 15, 1913.

SEE Eng. Pat. 4942 of 1915; this J., 1916, 294.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Carbonising coal.* J. West. Gas J., 1917, 140, 563 and 611. (See this J., 1917, 1265.)

EXPERIMENTS on the heating and lighting power of coal gas have shown that probably a gas ranging from about 450 to 500 B.Th.U. would be suitable for all purposes, and the author gives results of tests aiming at the manufacture of gas of this quality. Comparative tests were made in Glover-West continuous vertical retorts under three conditions:—(1) High temperature, without steaming; (2) high temperature, together with steam at 25 lb. pressure; (3) high temperature, with steam at 40 lb. pressure. With Orrell nuts the results were respectively:—Gas made per ton (corrected), 14,989, 17,759, and 21,684 cub. ft.; dehydrated tar per ton, 17.4, 18.2, and 18.6 galls.; ammoniacal liquor (10 oz.), 39.7, 42.4, and 40.3 galls.; gross calorific value, 503, 460, and 442 B.Th.U. The corresponding figures with Wigan Arley Mine coals were:—Gas per ton, 14,507, 15,987, and 23,016 cub. ft.; tar per ton, 16.5, 17.7, and 18.8 galls.; ammoniacal liquor per ton, 29.2, 33.0, and 44.3 galls.; calorific value, 526, 511, and 453 B.Th.U. Summarising these results the author gives the following figures as representing the respective increase in volume of gas per ton of coal as due to:—(1) higher temperature only, (2) higher temperature and 25 lb. steam pressure, (3) higher temperature and 40 lb. steam pressure. Orrell nut coal, (1) 2599, (2) 5369, (3) 9294 cub. ft. Wigan Arley Mine coal, (1) 1673, (2) 3173, (3) 10,182 cub. ft.—J. E. C.

*Petrol and other volatile substances; Fractionating apparatus for —.* E. Hildt. Comptes rend., 1917, 165, 790—793.

THE apparatus consists of six flasks having wide necks and each provided with a small reflux condenser connected with a side tube at the top of the neck. Each neck is closed by a stopper through which passes a tube closed at the bottom and extending into the neck; suitable tubes connect these tubes in series. The flasks contain petroleum fractions having the following b. pts. respectively:—above 150°, 130°—150°, 110°—130°, 90°—110°, 70°—90°, 50°—70°, and below 50° C. The flasks are heated by separate burners so that the tubes in the necks are surrounded by vapours at the temperatures mentioned. The petrol under examination is admitted, drop by drop, into the first tube and the more volatile portion is at once driven over into the second and succeeding tubes, so that fractions of decreasing b. pts. collect in the series of tubes. Means are provided for drawing off these fractions and for condensing the very volatile vapours which escape from the last tube of the series.—W. P. S.

*Laboratory methods for benzol recovery plant operation.* Sperr, jun. See III.

*Manufacture of concentrated and pure liquor ammonia.* Hebden. See VII.

*Manufacture of concentrated ammonia liquor.* Parrish. See VII.

*A new blast-furnace gas cleaning machine.* Ruddiman. See X.

### PATENTS.

*Distillation of coal for the manufacture of gas, and apparatus therefor.* A. Pinet and A. Debout, Paris. Eng. Pat. 10,207, July 13, 1915. Under Int. Conv., July 27, 1914.

SEE Fr. Pat. 475,716 of 1914; this J., 1916, 33.

*Gas-producer.* P. A. J. Cousin, Loos-lez-Lille, France. U.S. Pat. 1,246,888, Nov. 20, 1917. Date of appl., Oct. 16, 1912.

SEE Eng. Pat. 23,256 of 1912; this J., 1913, 413.

*Apparatus for manufacturing hydrogen.* U.S. Pat. 1,246,867. See VII.

*Method and apparatus for testing [calorific value of] gases.* U.S. Pat. 1,245,888. See XXIII.

## III.—TAR AND TAR PRODUCTS.

*Benzol recovery plant operation; Laboratory methods for —.* II. Method for boiling point test of benzols. F. W. Sperr, jun. Met. and Chem. Eng., 1917, 17, 586—588. (See this J., 1918, 2 A.)

A STANDARD Engler flask is used, of globular shape, 6.5 cm. in diameter, with a cylindrical neck 1.6 cm. in internal diameter and 15 cm. in length. Half-way up the neck a tube of not less than 5 mm. diameter and 10—12 cm. long extends downwards at an angle of 75°. The condenser tube is a straight piece of glass tubing 24 in. long and of  $\frac{1}{2}$  in. internal diameter. The outer jacket is 15 in. in length. A standard Bunsen burner, with an opening  $\frac{5}{8}$  in. at the top, is used for heating, the flame burning blue at the top. The flask rests on an asbestos slab,  $\frac{1}{2}$  in. thick, 6 in. square and having a circular hole 1 in. in diameter in the centre. The furnace, or enclosure for the burner, is cylindrical in shape, 5 in. in diameter and 8 in. high, with four holes 1 in. in diameter located  $\frac{1}{2}$  in. below the top. The receiver is a 100 c.c. cylinder graduated in 1 c.c. The thermometer for crude oils and solvent naphthas is graduated in degrees from 0° to 400° C.; for pure benzene and toluene it is graduated in  $\frac{1}{10}^{\circ}$  from 70° to 120° C. For the test, 100 c.c. (at 15.5° C.) is measured into the flask and distilled at the rate of 4—5 c.c. per min. For refined products, the following temperature readings are taken: First drop, 5%, 10%, and each 10% to the dry point. For light oils, the temperature of the first drop is read, and the % distillate at the following temperatures:—80°, 95°, 100°, 120°, 150°, 180°, and 200° C. For boiling point tests of refined products, corrections should be made for emergent stem of thermometer and barometric pressure. The above details of the method adopted by the author are based on the European method (Lunge's "Coal Tar and Ammonia," 4th Amer. Edn., p. 755), the American method (Church, J. Ind. Eng. Chem., 1911, p. 227), and the method given in the Gas Chemists Handbook, p. 180, published by the American Gas Institute.—L. A. C.

## IV.—COLOURING MATTERS AND DYES.

*Logwood; Examination of*—Colour Trade Journal, Oct., 1917. J. Amer. Leather Chem. Assoc., 1917, 12, 586–598.

EVIDENCE of the probable value of logwood is obtained by examination of a freshly cut surface, which should be reddish and not grey. The value of the extract depends upon the content of hamatoxylin, formed by fermentation of the glucoside, and hamatein, formed by further fermentation, a process which would eventually destroy the tinctorial power. Colorimetric processes are described depending on the development of colour with alum or with copper salts. The normal ash of logwood contains alkali chlorides and phosphates; the wood should contain less than 2%, the extract less than 3% of ash. The sugar content is determined after removal of hamatoxylin and hamatein by means of hide powder. Pure dry logwood extract contains 87% of ether-soluble and 13% of alcohol-soluble material, whereas the common adulterant, chestnut extract, is almost insoluble in ether but readily soluble in alcohol. A method for determining gelatin, molasses, and chestnut extract is detailed, and the methods to be used for valuing logwood extracts by dye-trials, more particularly in reference to the variable contents of hamatoxylin and hamatein, are discussed.—F. W. A.

## PATENT.

*Indophenolic compounds and dyestuffs therefrom; Manufacture of*—E. Wray, and Claus and Co., Ltd., Manchester. U.S. Pat. 1,247,475, Nov. 23, 1917. Date of appl. Aug. 12, 1916.

SEE Eng. Pat. 101,433 of 1916; this J., 1916, 1105.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Textile plants; The Carbone method for retting*—by microbiological action. D. Carbone and A. Tombolato. Annali d'Igiene sperim., 1916, 26, [1]; Staz. sperim. Agrar. Ital., 1917, 4, 261–299, and Progressi Nelle Ind. Tintorie e Tessili, 1917. Bull. Agric. Intell., 1917, 8, 1417–1418.

REFERENCE is made to Rossi's method (this J., 1917, 79) for the retting of hemp, etc., by cultures of a specific pectic ferment, *B. Cornesi*, which in the author's view is identical with or very similar to *B. asterosporus*. This method has given satisfactory results in Italy and France, since it shortens the time of retting, gives no bad smell, and allows inferior hemp to be retted more rapidly than in field retting pools. On the other hand, the product differs from that obtained by field retting; the green parts of the bark remain adherent to the fibres and special machines must be used for separating them and washing the fibre. The resulting material is of a different character from that of the standard product and some temporary difficulty may be experienced in disposing of the fibre to advantage. These differences are attributed to the use in Rossi's process of an aerobic bacillus, which is not a natural agent of the field retting process, having first been isolated from a decomposing potato. In Carbone's process, on the other hand, an obligatory anaerobic bacillus is employed, which was isolated from the mud of retting pits at Bologna and which has been named *B. felsineus*. This bacillus has been found fairly generally distributed in the hemp pits of Italy and is very probably the specific natural retting agent. Together with *Saccharomyces*, this bacillus actively rets hemp, in stalks or green "harl," in less than 2½ days at 37° C. It gives the same type of retting as the field retting, i.e.,

complete detachment of the woody parts and spontaneous exfoliation. This renders the use of special machines and washing unnecessary and the manipulation is the same as in field retting. The Carbone method is being tested extensively on the 1917 crop; in applying it, pools are built, the water is heated to 37° C. by steam or otherwise, and the selected culture is used. Besides hemp, *B. felsineus* is capable of retting other bast fibres and various species of *Malvaceæ* and agave.—J. F. B.

## PATENTS.

*Non-inflammable cellulose compound and process of making same*. W. G. Lindsay, Newark, N.J., Assignor to The Celluloid Co. U.S. Pat. 1,245,476, Nov. 6, 1917. Date of appl., Jan. 27, 1915.

A NON-INFLAMMABLE cellulose compound is obtained from acetylcellulose and triphenyl phosphate by dissolving them in chloroform and dichlorohydrin, with or without acetone.—F. Sp.

*Vegetable proteid product [celluloid substitute] and process of making same. Celluloid-like substance and process of making same. Process of making celluloid-like substances*. S. Satow, Sendai, Japan. U.S. Pats. (A) 1,245,975, (B) 1,245,976, and (C) 1,245,983. Nov. 6, 1917. Dates of appl., Nov. 25 and Dec. 11, 1916, and Apr. 12, 1917.

(A) A TRANSPARENT substance resembling celluloid is made by "glutinating" proteins with a compound which reacts alkaline in solution, and combining the product with a cellulose derivative. (B) A celluloid-like substance is produced by "glutinating" vegetable proteins with an acid, and treating the mass with an active methylene compound. The physical properties of the product vary with the amount of acid used. (C) The soluble contents of materials containing vegetable proteins are extracted with sodium sulphite, and the separated proteins are glutinised to form celluloid-like substances; or the vegetable proteins may be treated with an oxymethane sulphonic compound.—F. Sp.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid and oleum; Thermal properties of*—A. W. Porter. Faraday Soc., Dec. 12, 1917. [Advance proof.] 26 pages.

THE thermal properties of sulphuric acid and oleum of different concentrations have been calculated from published data. (1) *Sulphuric acid*. (a) *Heat of dilution at ordinary temperature*. From the data of Pfaundler, Brönsted, and Knietzsch (Ber., Oct. 19, 1901; this J., 1903, 172, 343) for the heat of dilution of sulphuric acid, and of Thomsen (*Thermochem. Untersuch.*, 3, 34) for the heat of formation of sulphuric acid from sulphur trioxide and water, the following equation is derived:—

$$D_{15} = \frac{101.5}{[M + 0.2013]^2}$$

in which  $D_{15}$  is the true or differential heat of dilution, i.e., the heat set free at 15° C. when a large amount of solution containing 1 gm. of sulphur trioxide and M grms. of water is diluted with 1 gm. of water. (b) *Variation of heat of dilution with temperature*. Using Henning's values for the latent heat of water,

$$D_t = \frac{101.5}{[M + 0.2013]^2} + \frac{0.0443(t - 15^\circ)}{[M + 0.062]^2}$$

in which  $D_t$  is the heat of dilution at temperature  $t$ . (c) *Heat of concentration as in Gaillard towers*.



The heat required to evaporate dilute acid containing 1 grm. of sulphur trioxide and  $M_1$  grms. of water until it only contains  $M_2$  grms. of water is

$$L_w(M_1 - M_2) + [h] \frac{M_1}{M_2}$$

in which  $L_w$  is the latent heat of evaporation of water and  $h$  the heat of dilution of 1 grm. of  $\text{SO}_3$  with  $M$  grms. of water. The value of this expression when  $M_2=0$  is called the *heat of total evaporation*. The utility of this quantity is not impaired by the fact that total evaporation could not take place. If this quantity is plotted for different strengths, it is only necessary to take the difference of the numbers corresponding to the two strengths at the beginning and end of the process, each at the temperature of the evaporation, to obtain the heat of concentration desired. When change of temperature occurs at the same time, a correction is made for the thermal capacity of the solution, and the external work done during evaporation also influences the quantity of heat required. (2) *Oleum*. (a) *Latent heat of evaporation of liquid sulphur trioxide*. This is derived from Knietsch's values for the vapour pressure of 100% oleum, and is given by

$$L_s = \frac{116.6}{P^{.50}}, \text{ where } P \text{ is the vapour pressure. (b)}$$

*Latent heat of fusion of sulphur trioxide* is calculated from Knietsch's heat of solution for solid and liquid sulphur trioxide. The author disagrees with Knietsch's smoothed values, and substitutes his own. There is no evidence of the existence of  $\text{H}_2\text{S}_2\text{O}_7$ . (c) *Heat of solution*. Some corrected values of  $k_t$  are tabulated ( $k_t$ =heat set free when 1 grm. of water is mixed with  $m$  grms. of sulphur trioxide at temperature  $t$ ). (d) *The heat of evaporation (or condensation) of sulphur trioxide*

*vapour* is given by  $L_s(m_2 - m_1) + [k] \frac{m_2}{m_1}$ , where

$L_s$ =latent heat of evaporation of liquid sulphur trioxide, and  $m_1, m_2$  are the initial and final amounts of sulphur trioxide per 1 grm. of water. The *heat of total evaporation of sulphur trioxide vapour* (analogous to the expression found above for sulphuric acid concentration) is tabulated for selected values of  $m_1$  ( $m_2=0$ ). (e) The heat  $k$  evolved when 1 grm. of water is mixed with  $m$  grms. of liquid  $\text{SO}_3$  is given by the equation

$$k = \frac{50.43 m}{1 + 0.2013 m} + \frac{0.714 m (t - 1.5)}{1 + 0.062 m}$$

A series of tables is given containing values calculated from the equations derived in the paper, and instructions are given for the construction and reading of an alignment chart, giving results graphically. Several examples are given of the application of the formulae to problems arising in the concentration of sulphuric acid and the manufacture of oleum.—F. SP.

*Soda Liquors: Causticising*—M. L. Griffin, W. H. Howell, jun., and G. K. Spence. Report of Soda Pulp Committee, Joint Convention of Techn. Assoc. of Pulp and Paper Ind., and Tech. Sect. of Canadian Pulp and Paper Assoc., Sept. 27, 1917. Met. and Chem. Eng., 1917, 17, 599—603.

The report deals with four processes for causticising sodium carbonate liquors and separating the sludge. (1) Intermittent precipitation, sedimentation, and decantation. The alkali, after causticising with lime, is run into a series of tanks in which the sludge is allowed to settle and the clear liquor is decanted. The sludge is washed by dilution and further decantation, the weaker wash liquors being used for washing strong sludges or for leaching the black ash. Such a system requires extensive plant and much labour. (2) Continuous

precipitation, sedimentation, washing, and separation. In the Dorr system, the causticised liquor is passed through a series of thickeners working on the counter-current principle, the sludge being removed by a special device and passed on to the next of the series. Weak wash liquors are disposed of in this system, but it involves much plant, labour, and loss. (3) Intermittent or continuous precipitation, filter-pressing, and washing. After sedimentation and one decantation, the sludge is passed to a filter-press, the liquor being mixed with the decanted liquor. By washing the cake in the press, a weak liquor is obtained, which is disposed of in the usual way. (4) Intermittent or continuous precipitation with continuous filtering and washing. The "Boston tank" for separating liquors from sludge is cylindrical in shape with a conical bottom having a trumpet feed in the centre. The clear liquor flows away through an annular collecting gutter and the sludge, drawn off continuously from a suitable level, is passed on to a rotary continuous filter. By using filter-presses, either intermittent or continuous, there is a great saving of space and labour.—L. A. C.

*Ammonia liquor: Manufacture of concentrated*—P. Parrish. Gas. J., 1917, 140, 554—556.

IN pre-war times the process in vogue for the manufacture of liquor ammonia involved an initial admixture of milk of lime with the gas liquor to fix the whole of the carbonic acid and hydrogen sulphide. The gases from the still passed through a dephlegmator to obtain an approximately dry ammonia gas which was absorbed in distilled water in three vessels arranged in cascade form. The fixation of the whole of the hydrogen sulphide in this manner would have resulted in serious loss of sulphur compounds and an attendant reduction in the output of sulphuric acid, and consequently the present tendency is to endeavour to desulphurise and decarbonate the liquor by preliminary heat treatment. This is not altogether an easy matter, and results obtained by J. W. Lee showed that 42.1% and 52.6% respectively of the hydrogen sulphide content was removed by the decarbonator, 35.33% and 34.2% respectively being removed by subsequent treatment in a lime purifier. The provision of a subsequent lime purifier is, however, not entirely satisfactory, and a plant capable of removing hydrogen sulphide and carbon dioxide by heat alone would be a great advantage in the by-product industry. The return of the reflux from a lime washer to the fixed ammonia section of the still would (in the case of a plant dealing with 100 tons of crude liquor per day) entail a removal of an extra 2.61 tons of ammonia in addition to the normal amount of 0.25 ton. This necessitates additional steam, and is accompanied by a liability to blockages by calcium sulphide and calcium carbonate. The effective absorption of effluent gases from a decomposer presents difficulties and the author quotes the following calculated analysis of such gases based on a consumption of 100 tons of 1.21% gas liquor per day:—Air, 5.0%;  $\text{NH}_3$ , 15.0%;  $\text{H}_2\text{S}$ , 6.1%; and  $\text{CO}_2$ , 73.9% by volume. Owing to the absence of any "carrying" gas, the carbon dioxide and ammonia are afforded an opportunity to react, giving rise to possibilities of blockage by deposition of ammonium bicarbonate and other complex compounds. The use of a small quantity of steam as a "carrying" medium has been found advantageous in preventing deposition of sublimate from the gases before they reach the absorbing tower. Pressure in the decomposer should be controlled as it tends to reduce the efficiency of the dissociation. In designing the trays of a decomposer the surface area for the

ascent of the gas and the space for the descent of the liquor should be in proportion to the computed volume of gas at each tray. Cast-iron circular coils are recommended for cooling purposes, steel coils being attacked by the gas liquor, and lead coils being liable to burst. It is advisable to use pure water in the scrubbers after the decomposer. Care should be exercised in filling tanks to minimise loss of ammonia due to displacement of ammoniacal air, and the author recommends that all vents from storage boilers and railway tanks, and the effluent gases from the decomposer be led to one common scrubber of adequate size.—J. E. C.

*Ammonia; Manufacture of concentrated and pure liquor*—. G. A. Hebden. *Coke Oven Managers' Assoc.*, Dec. 15, 1917. *Gas J.*, 1917. 140, 609—610.

"CONCENTRATED ammonia" is a highly concentrated liquor, containing 25%  $\text{NH}_3$  and not more than 0.5%  $\text{H}_2\text{S}$ , used in the ammonia-soda process. "Pure liquor ammonia" contains up to 30%  $\text{NH}_3$  and no sulphide, phenols, pyridine, or tarry matter. In the manufacture of these products carbon dioxide and hydrogen sulphide are selectively expelled by heating crude gas liquor to about 95° to 100° C. The decomposers are worked on the principle of a continuous ammonia still, direct steam being admitted into the lowest chamber whilst occasionally indirect steam is utilised in the other trays. The crude liquor distilled contains 1.60% free  $\text{NH}_3$ , 0.15% fixed  $\text{NH}_3$ , 1.7%  $\text{CO}_2$ , and 0.24%  $\text{H}_2\text{S}$ . A portion enters the decomposer at the top tray and the other portion, after passing through the first and second condensers, enters about half-way down, a definite economy in steam consumption resulting from this interchange of heat. The temperature at the bottom tray is kept at 100° to 105° C., with a pressure of 10 to 12 lb. per sq. in. The temperature of the gas from the top of the decomposer is kept at about 45° C., any ammonia passing over being retained in water catchers along with a little carbon dioxide and hydrogen sulphide. The ammonia-free gas is then passed through oxide beds. Under the above conditions the  $\text{CO}_2$  content is kept below 0.10% and  $\text{H}_2\text{S}$  below 0.03%. The lime is slaked with hot spent liquor, agitated, and after being mixed with the condensed liquid from some of the condensers is pumped into the main still. The top tray of this still is worked under a pressure of 6 lb. per sq. in., and the waste liquor has a content of 0.005%  $\text{NH}_3$ . The ammonia vapours pass through three condensers fitted with cast iron cooling coils, cold liquor passing through the first and second coils, cold water through the third. The condensed liquids from the first and second vessel contain 2.5% and 7.4%  $\text{NH}_3$  respectively. In making concentrated liquor the condensed liquid and gases from the third condenser pass through absorbers of cast iron, arranged in series, and fitted with cooling coils, any residual gases being absorbed in coke towers fitted with water sprays. The final concentrated liquor contains 25%  $\text{NH}_3$  and 0.15 to 0.25%  $\text{H}_2\text{S}$ . In making pure liquor ammonia the gases leaving the last condenser pass through three air-cooled condensers, as it is essential that the gases be dry and at a lower temperature than 15° C. The gases after this additional cooling pass through caustic soda washers to absorb traces of carbonate, then through slaked-lime purifiers to remove hydrogen sulphide. Pyridine, phenols, etc., are extracted in oil washers, and the gases, after passing coke towers to trap any oil in suspension, are led to absorption tanks; these consist of closed iron tanks in series fitted with cooling coils, and arranged in counter-current fashion. The plant described deals with 400 tons

of crude liquor per day, 25 cwt. of lime and 1.8 cwt. of coal being used per ton of liquor.

—J. E. C.

*Water, sodium sulphate, and ammonium sulphate; Equilibria in the ternary system*—. C. Matignon and F. Meyer. *Comptes rend.*, 1917, 165, 787—789.

THE possible solid phases in this system are ice, anhydrous sodium sulphate, crystallised sodium sulphate, ammonium sulphate, and the double salt  $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ , and the following solubilities have been determined:—

I. Solid phase:  $\text{Na}_2\text{SO}_4, (\text{NH}_4)_2\text{SO}_4$ :—

Original mixture.	Temp. °C.	Centigram-mols. in 100 grms. of solution.	
		$(\text{NH}_4)_2\text{SO}_4$ .	$\text{Na}_2\text{SO}_4$ .
Double salt + $2(\text{NH}_4)_2\text{SO}_4$	62.5	27.58	11.50
$\text{Na}_2\text{SO}_4$ + excess $(\text{NH}_4)_2\text{SO}_4$	79	29.16	11.15
Double salt + $2\text{Na}_2\text{SO}_4$ ....	79	28.90	—
Double salt + $3(\text{NH}_4)_2\text{SO}_4$	79	28.91	11.56
Double salt .....	98	30.65	12.23
$\text{Na}_2\text{SO}_4$ + excess $(\text{NH}_4)_2\text{SO}_4$	100	30.62	11.27
$\text{Na}_2\text{SO}_4$ + excess $(\text{NH}_4)_2\text{SO}_4$	109	31.63	11.35

II. Solid phase:  $(\text{NH}_4)_2\text{SO}_4$ , double salt:—

Double salt + $(\text{NH}_4)_2\text{SO}_4$ ..	—19	28.05	2.10
" " " "	—1	28.57	3.34
" " " "	+33	28.58	6.38
" " " "	44	28.83	8.08
" " " "	58	27.70	10.87
$\text{Na}_2\text{SO}_4$ + excess $(\text{NH}_4)_2\text{SO}_4$	44.5	28.20	8.06

III. Solid phase: double salt,  $\text{Na}_2\text{SO}_4$ :—

Double salt + $\text{Na}_2\text{SO}_4$ ....	29	10.55	18.46
" " " "	46	18.13	15.08
$(\text{NH}_4)_2\text{SO}_4$ + excess " $\text{Na}_2\text{SO}_4$	50	21.48	13.73
Double salt + $\text{Na}_2\text{SO}_4$ ....	58	25.31	12.34

IV. Solid phase: double salt,  $\text{Na}_2\text{SO}_4, 10\text{H}_2\text{O}$ :—

Double salt + $\text{Na}_2\text{SO}_4, 10\text{H}_2\text{O}$	—13	—	2.98
" " " "	+1	22.02	5.21
$(\text{NH}_4)_2\text{SO}_4$ + excess " $\text{Na}_2\text{SO}_4, 10\text{H}_2\text{O}$	+10	18.47	8.22
" " " "	14	16.36	10.46
Double salt + " $\text{Na}_2\text{SO}_4, 10\text{H}_2\text{O}$	14	16.26	10.80
" " " "	25.5	10.06	18.82

V. Saturated solutions of ammonium sulphate, sodium sulphate, and a mixture of the same boil at 108.9°, 102°, and 111° C., respectively, at ordinary pressure, and at these points the solutions have the following concentrations:—

Solid phase.	Temp. °C.	Centigram-mols. in 100 grms. of solution.	
		$(\text{NH}_4)_2\text{SO}_4$ .	$\text{Na}_2\text{SO}_4$ .
$(\text{NH}_4)_2\text{SO}_4$ .....	108.9	39.22	—
$\text{Na}_2\text{SO}_4$ .....	102	—	21.0
$\text{Na}_2\text{SO}_4, (\text{NH}_4)_2\text{SO}_4$ .....	111	31.75	11.25

—W. P. S.

*Orychloride of tin; A new*—. H. F. Keller. *J. Amer. Chem. Soc.*, 1917, 39, 2354—2356.

AN anhydrous stannous oxychloride, not apparently previously described, has been found in the centre of a small lenticular mass of tin occurring in the Indian Mounds of Florida. Minute brilliant crystals were observed in cavities in the centre of this mass, about 0.3 gm. of which was collected. The pure material was picked out under a lens and analysed, the result being tin 70.92%, chlorine 21.68%, impurities 0.26%. The formula  $\text{SnCl}_2, \text{SnO}$  is suggested. It appears probable that water containing eldride has gained access to the



cavities through fine holes or fissures and the oxychloride has been very slowly produced by the action of chloride and air.—B. V. S.

*Sodium paratungstate; Use of*—*in the determination of metallic oxides from cyanides.* S. B. Kuzirian. J. Amer. Chem. Soc., 1917, 39, 2356—2358.

THE use of sodium paratungstate as a flux in analytical processes (this J., 1911, 800; 1913, 908, 909) has been extended to the determination of cyanides which, in the presence of an oxidising agent, preferably ammonium nitrate, which leaves no residue on fusion, are converted into oxides. The method was checked on a sample of cyanide of which the nitrogen content, chloride equivalent, and potassium content had been previously determined. The cyanide is heated in a crucible with sodium paratungstate, previously ignited to constant weight, a sufficient amount of ammonium nitrate being added to secure complete oxidation. The increase in weight of the paratungstate is then taken. Allowance must be made for carbonates, chlorides, etc., which may be present and which are also converted into oxides in the process.

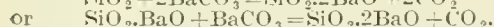
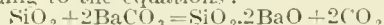
—B. V. S.

*Use of cupferron (phenylnitrosodihydroxylamine-ammonium) in:*—*I. Quantitative separation of zirconium, titanium, iron, manganese, and aluminium. II. Analysis of zircon and baddeleyite.* Brown. See XXIII.

#### PATENTS.

*Barium hydrate [hydroxide]; Production of*—C. Deguide, Pas de Calais, France. Eng. Pat. 110,537, July 10, 1917. (Appl. No. 9937 of 1917.) Under Int. Conv., Sep. 29, 1916.

AN intimate mixture of finely powdered barium carbonate (394 parts) with sand or silica (60 parts) or with monobarium silicate (106½ parts) is heated to 1400°—1500° C., dibarium silicate being formed according to the equations:—



The dibarium silicate does not fuse at the temperature of the reaction: on heating with water it forms barium hydroxide and insoluble monobarium silicate, the latter being used again with a fresh supply of barium carbonate.—B. V. S.

*Sulphate of ammonia; Manufacture of*—E. V. Evans, and South Metropolitan Gas Co., London. Eng. Pat. 111,309, Sep. 14, 1916. (Appl. No. 13,063 of 1916.)

AMMONIA is passed into hot sulphuric acid, of such dilution that ammonium sulphate does not separate, until the liquor is weakly alkaline. The solution is then filtered and cooled: the ammonium sulphate which crystallises out is practically both white and neutral.—B. V. S.

*Calcium chloride; Obtaining of anhydrous*—E. A. Ashcroft, London. Eng. Pat. 111,313, Oct. 19, 1916. (Appl. No. 14,851 of 1916.) Addition to Eng. Pat. 108,920, Sept. 19, 1916 (this J., 1917, 1048).

THE method described in the chief patent for the manufacture of anhydrous magnesium chloride is applied to the calcium salt.—B. V. S.

*Sodium sulphite; Manufacture of*—G. L. Crump, and Ekaertin, Ltd., London. Eng. Pat. 111,314, Apr. 19, 1917. (Appl. No. 14,935 of 1916.)

A MIXTURE of equal weights of sodium sulphate and iron filings, preferably with the addition of about 10% of carbonaceous matter, is heated in an atmosphere of sulphur dioxide.—B. V. S.

*Aluminous ores; Process of treating*—[to obtain crystalline alumina]. H. A. Richmond, Providence, R.I., Assignor to General Abrasive Co., Inc., Niagara Falls, N.Y. U.S. Pat. 1,245,383, Nov. 6, 1917. Date of appl., Nov. 27, 1916.

ALUMINOUS ores are heated in an electric furnace with a sulphide of a basic metal and carbon. Impurities are driven off as gaseous sulphides and the reduced metal is drawn off in the molten state, leaving a purified crystalline alumina.

—B. V. S.

*Hydrogen; Apparatus for manufacturing*—R. E. Brunner, Pittsburgh, Pa. U.S. Pat. 1,246,867, Nov. 20, 1917. Date of appl., May 23, 1917.

IN apparatus for producing hydrogen, the heated gas to be decomposed is passed in series through vertical pipes connected alternately at top and bottom, and water is sprayed downwards into each pipe to remove carbon and other impurities. The water is finally discharged through a liquid seal at the bottom of each pipe.—W. F. F.

*Barium hydrate [hydroxide]; Process for obtaining*—C. Deguide, Ixelles, Assignor to D. Marbais, Chaderoi, Belgium. U.S. Pat. 1,247,510, Nov. 20, 1917. Date of appl., Mar. 6, 1917.

SEE Eng. Pat. 110,537; preceding.

#### VIII.—GLASS; CERAMICS.

*Silica bricks; Manufacture of*—Philippon. Comptes rend., 1917, 165, 1002—1005.

SILICA bricks were made by grinding silica from different sources with milk of lime for ten minutes and then moulding the product by hand. The bricks were dried on cast iron plates at about 100° C. and were then burned at 1300° C. in a down-draught kiln. Crushing tests were made by placing two half bricks one above the other in the testing machine. Refractoriness tests were made on pieces cut to the same shape and size as standard cones and then heated steadily in a corundum cylinder in a furnace using acetylene and compressed air or oxygen. It was found that the highest crushing strengths were possessed by bricks in which the quartz was most finely ground: bricks made of impalpably fine quartz with 2% lime had a crushing strength of 18 kilos, when dried, and 320 kilos, when burned at 1300° C., but similar bricks made of quartz particles 0.13 mm. diameter had crushing strengths of only 2 kilos, and 16 kilos, respectively. Notwithstanding the differences in the geological origin and physical characters of the various forms of silica used, they all were capable of producing bricks of great strength if the raw material was sufficiently finely ground. The minimum crushing strength of bricks made from impalpably fine quartz was found to be 200 kilos, per sq. cm. The expansion produced on burning the bricks is greatest with those containing large grains. The expansion of bricks made of impalpable quartz is almost nil. The crushing strength of the bricks is increased by the use of relatively large quantities of water in the paste of which they are made. The strength of the dried bricks varies with the proportion of lime present; that of the burned bricks increases with the proportion of lime up to 1%, then remains constant up to 2%, and diminishes with still larger proportions. Each 1% of lime lowers the refractoriness by about 20° C. Bricks made of impalpable quartz begin to soften at 800° C.; at 1200° C. they have a crushing strength of 190 kilos, per sq. cm. and 270 kilos, per sq. cm. at 1300° C. Bricks containing larger grains of quartz do not

begin to soften below  $1100^{\circ}\text{C}$ ., but at  $1300^{\circ}\text{C}$ . they have a crushing strength of only 70 kilos. per sq. cm. Tests on bricks made of impalpable quartz mixed with larger grains showed that the most refractory bricks contained the least quantity of impalpable powder and that grains of 8 mm. diameter are the largest permissible in good bricks. On applying the foregoing results to commercial manufacture, it was found best to use material with at least 96% silica, the precise form being unimportant so long as 30% of it is in impalpable powder and 70% in grains of 1–8 mm. diameter. The proportion of lime added was only 0.6% of the bricks, but 2% of the "flour." The bricks fired in a tunnel kiln at  $1300^{\circ}\text{C}$ . had a crushing strength of 200–250 kilos. per sq. cm., an average expansion of 1.6%, a true sp. gr. of 2.4, an apparent sp. gr. of 1.9, and a fusion point of about  $1780^{\circ}\text{C}$ . The bricks have been used for lining a Martin furnace; they have already withstood 200 heats and it is expected that they will withstand more than 300 heats. They are also employed satisfactorily in a number of forges, replacing aluminous bricks.—A. B. S.

#### PATENTS.

*Glass; Opaque semi-translucent* — J. J. Miller, Assignor to Macbeth-Evans Glass Co., Pittsburgh, Pa. U.S. Pat. 1,245,487, Nov. 6, 1917. Date of appl., Nov. 10, 1913.

A "SEMI-OPAQUE, semi-translucent" glass is made by fusing a clear-glass batch with compounds of aluminium, fluorine, chlorine, and sulphur. For the last two, a chloride and strontium sulphate are specified.—A. B. S.

*Refractory bricks for furnaces; Manufacture of* — A. Reynolds, Ilove, Sussex. Eng. Pat. 111,355, Dec. 4, 1916. (Appl. No. 17,397 of 1916.)

THE faces or working surfaces of firebricks are heated to such a temperature that the changes which impart coherence or increased refractoriness to the bricks when in use in a furnace may be effected previous to the bricks being required. Each brick is embedded separately in a highly refractory inert material, such as chromite, plumbago, or ground coke and tar, so that only the face to be superheated is exposed, and is then heated until the exposed face has reached the requisite temperature or until the desired changes have been effected to a suitable depth. Bricks, etc., made of materials with a silica: alumina ratio corresponding to the formula  $\text{SiAl}_2\text{O}_5$  (sillimanite), or with rather more alumina, are heated until incipient fusion occurs at the surface, usually at  $1700^{\circ}\text{C}$ ., the product of the reaction melting at  $1850^{\circ}\text{C}$ . or higher. For foundry-ladle nozzles or stoppers, special devices are needed to prevent the incipiently fused surfaces falling away. For example, a nozzle may be mounted horizontally in a revolving furnace and rotated so rapidly during the superheating that no falling away can occur. Stoppers for steel-casting ladles are conveniently mounted on a rotating rod or screw, similar to the plug-rod of the ladle, and during rotation, the stopper is subjected alternately to heating and cooling so that incipient fusion occurs at its momentarily upper surface, and cooling when the fused portions might tend to fall off.—A. B. S.

*Glazed ceramic wares; Process for making* — C. Stegmayr, Newark, N.J., Assignor to H. A. Pike, New York. U.S. Pat. 1,245,285, Nov. 6, 1917. Date of appl., Nov. 10, 1914.

CERAMIC articles are made of a body composed of a mixture of common clay and a flux, an engobe of vitrifiable porcelain powder, and a leadless glaze. These materials in the form of powders may be

formed into three layers which are compressed to form the article and are then united by a single firing.—A. B. S.

*Kiln*. J. P. Williams, St. Louis, Mo. U.S. Pat. 1,245,846, Nov. 6, 1917. Date of appl., Oct. 6, 1913.

A CONTINUOUS kiln (of the Hoffmann type) is heated by means of gas admitted through transverse flues below the floor to air-containing ignition-chambers, combustion being completed among the goods to be burned and above the floor of the kiln. Means are provided for creating a draught horizontally across the ignition-chambers, whereby the ignited mixture of gas and air may be maintained in a state of incandescence and the temperature of the kiln regulated. The lateral gas flues in each chamber are connected to the main annular gas flue by means of portable hoods containing gas-regulating dampers.—A. B. S.

*Abrasive; Aluminous* — L. E. Saunders and R. H. White, Niagara Falls, N.Y., Assignors to Norton Co., Worcester, Mass. U.S. Pat. 1,247,337, Nov. 20, 1917. Date of appl., Feb. 12, 1917.

AN abrasive material produced in the electric furnace consists of crystalline alumina and 1–15% of iron oxide, but substantially free from silicon and titanium oxides.—W. F. F.

*Refractory materials; Manufacture of* — N. Leccesne, Paris. Eng. Pat. 102,507, Dec. 2, 1916. (Appl. No. 17,289 of 1916.) Under Int. Conv., Apr. 29, 1914.

SEE Fr. Pat. 471,513 of 1914; this J., 1915, 356.

*Refractory materials; Process of making basic* — C. B. Stowe, Lakewood, Ohio, U.S.A. Eng. Pat. 110,147, Dec. 12, 1916. (Appl. No. 17,865, of 1916.) Under Int. Conv., Oct. 6, 1916.

SEE U.S. Pat. 1,205,956 of 1916; this J., 1916, 1261.

*Lacquered product and process of making same*. U.S. Pat. 1,245,982. See XIII.

### IX.—BUILDING MATERIALS.

*Manufacture of silica bricks*. Philippon. See VIII.

#### PATENTS.

*Road-treatment*. H. H. Hurt, Assignor to Robeson Process Co., New York. U.S. Pat. 1,245,349, Nov. 6, 1917. Date of appl., Jan. 9, 1917.

ROADS are surfaced with a granular material impregnated with a mixture of "wax tailings" and a thinning oil.—A. B. S.

*Cement; Manufacture of acid and water-proof* — C. C. Meigs, Assignor to Electro-Chemical Supply and Engineering Co., Philadelphia, Pa. U.S. Pat. 1,245,608, Nov. 6, 1917. Date of appl., July 12, 1915.

AN acid salt, such as calcium phosphate, is added to a cement composed of silicious material and sodium silicate, and the mixture is subjected subsequently to the action of an acid which will react with the silica-sodium silicate complex.—A. B. S.

*Diatomaceous material and process of making the same*. T. M. Caven, Baltimore, Md., Assignor to Maryland Products Co. U.S. Pat. 1,247,503, Nov. 20, 1917. Date of appl., Nov. 4, 1916.

NATURAL diatomaceous silica is transformed into a plastic condition in which it acts as a binder, mixed with a further quantity of the natural diatomaceous silica, and with divided combustible material. The mixture is calcined at such a



temperature that the plastic silica alone fuses, and the combustible material is burned out, leaving the resulting product porous.—W. F. F.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

[Steel] shoes and dies; *Electric furnace manufacture of*—on the Witwatersrand. *Electric steel-melting plant.* G. H. Stanley and W. Buchanan. J. Chem. Met. Soc., S. Africa. 1917, 18, 72—85.

EXPERIMENTAL and working details are given of a process and plant devised to meet the immediate requirements of the Witwatersrand by melting scrap steel shoes and dies in a non-tilting electric induction furnace of the Kjellin type and re-casting the metal into the required forms. The furnace is constructed of the materials available locally and has a capacity of 1 ton. The available electrical power—2000 volts, 3-phase, 50 cycles—is transformed to give a single-phase current up to 600 amp. at about 12 cycles; the efficiency of the conversion being 60—70%. The power consumption of the furnace is between 800 and 900 kilowatt-hours per ton of steel melted. The monthly output (i.e., net weight of serviceable shoes and dies) increased from 37.2 tons in January to 68.6 tons in August, 1917, the corresponding total costs being £20.0 and £13.8 per ton.

—W. E. F. P.

*Copper; The furnace refining of*—L. Addicks. Met. and Chem. Eng., 1917, 17, 579—584.

A NORMAL twenty-four hours cycle for a large modern reverberatory furnace would be about as follows:—Charging and melting, 14 hrs.; blowing, 2 hrs.; skimming, 1 hr.; poling, 2 hrs.; pouring, 5 hrs. Mechanical charging has replaced charging by hand, a novel form of peel consisting of a fork with a special pushing device having proved more successful than the upsetting shovel of the old type. By this means it is possible to charge at the rate of at least 200,000 lb. per hr. "Flapping" or "blowing" is accomplished by blowing compressed air through iron pipes beneath the surface of the molten metal. A small portion of the copper is oxidised to cuprous oxide, which, carried throughout the mass in solution, reacts with any impurities present. The oxidised impurities rise to the top as slag, which is skimmed off through the skimming door. Excess cuprous oxide is generally reduced by poling. Fuel oil, blown beneath the surface, has been tried as a reducing agent, but the method has several objections. Gas might in future be found serviceable for this purpose. During the processes of refining sample buttons are withdrawn from the mass by a ladle, and the shape and structure of these enable the operations to be followed with satisfactory accuracy. For pouring the refined copper into moulds, there are many mechanical devices, among them being the Walker wheel, the Clark machine, the multiple-lip ladle, and the twin ladle. Careful attention must be paid to painting the moulds, bone-ash wash and lampblack giving very satisfactory results. The operation of a reverberatory furnace is very wasteful as regards heat consumption, the efficiency being only about 19%; much of the waste heat, however, can be utilised in waste heat boilers. Great progress has been made in the development of the refining furnace along purely structural and mechanical lines, but there is room for improvement in the system of utilising labour.—L. A. C.

*Non-ferrous metals; The electric furnace as a medium for heating*—D. D. Miller. J. Amer. Inst. Metals, 1917, 11, 257—290.

THE electric furnace has been in use for steel making in America for 11 years, and the number of furnaces has increased from 19 in 1913 to 136 in 1917, out of a total of 471 of 17 different makes in use throughout the world, but until the last 3 or 4 years only intermittent attempts have been made to develop similar furnaces for the production of copper and its alloys, such as brass and bronze. At the present time many different types of furnaces are under construction and experiment with a view to commercial use. These include:—The Gillett furnace of the indirect type, General Electric furnace of the combined arc and resistance type, the Conley furnace of the moulded resistor type, the Thomson-Fitzgerald furnace of the resistance type, the Northrup furnace of the induction type, the Hering "pinch effect" furnace, and the Bennett furnace. The furnaces now in actual commercial use for copper-zinc alloys include the Ajax-Wyatt, Foley, Baily, Rennerfelt, Snyder, and Hoskins furnaces, and some of these are described and working results given. It is considered that the use of the electric furnace for heating and melting of non-ferrous metals will soon be established on a firm commercial basis.—B. N.

*Resistance alloys; Electrical properties of some high*—M. A. Hunter and F. M. Sebast. J. Amer. Inst. Metals, 1917, 11, 115—138.

It has been shown that high-resistance alloys must be sought for amongst those combinations in which the metals are present in the form of solid solutions, and that ternary alloys have in general a higher resistivity than binary alloys of similar composition. Amongst binary alloys the highest resistivities are found with nickel-manganese and nickel-chromium alloys, and the highest amongst ternary alloys are shown by ferro-nickel-manganese, ferro-nickel-chromium, and copper-nickel-chromium. Chromium alloys withstand oxidation at high temperatures better than the corresponding manganese alloys. Low temperature-coefficients were found chiefly amongst the copper-nickel-manganese alloys; an increase in the resistivity of an alloy is usually associated with a decrease in the temperature-coefficient, but the two are not strictly parallel, as shown by ferro-nickel and chromium alloys. A series of alloys of copper-chromium are indicated which have relatively high conductivities and high tensile strengths; e.g., alloys containing from 5 up to 17.3 parts of chromium per 100 parts of copper show in the hard drawn state a resistivity of 3.7 to 4.3 microhms per cm. cube and a tensile strength of 84,100—95,400 lb. per sq. in., the corresponding figures for the annealed alloys being 2—2.5 microhms and 55,300—66,500 lb. per sq. in.

—B. N.

*Blast-furnace gas cleaning machine; A new*—J. Ruddiman. Met. and Chem. Eng., 1917, 17, 608—611.

THE author describes a device for cleaning, drying, and preheating blast-furnace gas in order to obtain higher temperatures in the blast. In the preheater, hot unscrubbed gas from the furnace passes downwards through flues, imparting its heat to the scrubbed gas, which is conducted through a series of tubes. One type of preheater described acts both as preheater and dust catcher for the hot gas; in another type, it is necessary for the hot gas to have passed through a primary dust catcher, the preheater, however, serving to remove further quantities of dust. From the preheater, the unscrubbed gas passes on to the primary scrubber, which consists of a series of perforated

rotating baffle plates half immersed in water. The nozzles in each baffle are opposite the blank interspaces of the following baffle; thus the gas, impinging on a moist surface, is cooled and scrubbed. Spray pipes between the baffles furnish cooling water in finely divided form. In passing through the water in the lower half of the tank, the plates are cleaned. The gas then passes on to a tower where it is further scrubbed and cooled. In the tower is a central rotating vertical shaft on which are arranged a series of baffles similar to those in the primary scrubber. Sprays of water are supplied between the baffles. The rotation of the baffles imparts a rotary motion to the gas passing up the tower, ensuring thorough cooling, and the water and dirt thrown outwards by centrifugal force, pass downwards between the baffles and the tower, being removed at the bottom. The cool, scrubbed gas passes back into the series of pipes in the preheater and thence through a brick-lined stove main to the burners.—L. A. C.

*Determination of iron in Ferrum reductum.* Eberhard. See XX.

*Some substitutes for platinum ware.* Health. See XXIII.

*Colorimetric determination of manganese by oxidation with periodate.* Willard and Greathouse. See XXIII.

#### PATENTS.

*Alloy containing iron, cobalt, and silicon.* F. M. Becket, Assignor to Electro Metallurgical Co., Niagara Falls, N.Y. U.S. Pat. 1,247,296, Nov. 20, 1917. Date of appl., Apr. 19, 1915.

The alloy contains iron 55—65%, cobalt 30—40%, and silicon 3—6%.—W. F. F.

*Iron alloy.* J. M. Flannery, Assignor to Standard Chemical Co., Pittsburgh, Pa. U.S. Pat. 1,247,252, Nov. 20, 1917. Date of appl., Feb. 19, 1916.

An iron or semi-steel alloy consists of cast iron or semi-steel and 0.05—1% of uranium.—W. F. F.

*Open-hearth [steel] furnaces; Method of and apparatus for tapping.* M. A. Smith, Vandergrift, Pa. U.S. Pat. 1,247,606, Nov. 20, 1917. Date of appl., July 17, 1917.

MOLTEN steel is discharged from a tapping spout in front of the furnace into a receptacle having an opening in the bottom controlled by a stopper operated manually. An overflow outlet is provided in the upper edge of the receptacle, and a quantity of steel is always maintained in the receptacle so that slag is continuously discharged at the top and thereby separated from the steel. The molten steel is discharged into a ladle below.—W. F. F.

*Metallic articles; Method of coating — to prevent oxidation.* M. L. Crowe, Holliston, Mass. U.S. Pat. 1,247,086, Nov. 20, 1917. Date of appl., Apr. 3, 1915.

To produce a black or substantially black, rust-resisting finish on articles of steel or iron, the articles are immersed in a bath of molten caustic soda and sodium nitrate at a temperature above 900° F. (480° C.), and subsequently washed by immersion in boiling water.—W. F. F.

*Blast-furnaces and the like; Hoists for charging —.* Blast-furnaces and the like. T. G. Wrightson, J. M. Ringquist, and Head, Wrightson, and Co., Ltd., Thornaby-on-Tees, Eng. Pats. 111,592 and 111,593, Feb. 15, 1917. (Appl. Nos. 2273 and 2274 of 1917.)

*Ore concentrating machines.* W. B. Vanderlip, London, Eng. Pat. 111,685, Oct. 5, 1916. (Appl. No. 14,156 of 1916.)

*Zinc; Electrodeposition and extraction of —.* U. C. Tainton, London, and J. N. Pring, Chester. U.S. Pat. 1,247,179, Nov. 20, 1917. Date of appl., Nov. 7, 1916.

SEE Eng. Pat. 16,336 of 1914; this J., 1917, 37.

*Ores of certain metals; Treatment of —.* R. Lance, Paris. U.S. Pat. 1,247,413, Nov. 20, 1917. Date of appl., June 24, 1911.

SEE Fr. Pat. 476,304 of 1914; this J., 1916, 54.

*Process of treating aluminous ores.* U.S. Pat. 1,245,383. See VII.

## XI.—ELECTRO-CHEMISTRY.

### PATENTS.

*Electric batteries.* H. D. Henry, London, Eng. Pat. 111,383, Jan. 17, 1917. (Appl. No. 860 of 1917.)

IN electric batteries containing a zinc negative element and an acid electrolyte, a preliminary treatment is given to the zinc by placing it in the electrolyte in presence of a fully charged lead dioxide positive plate, discharging the battery through a suitable resistance so as to cause violent action on the surface of the zinc, washing the zinc free from acid, and drying it. It is then amalgamated by applying mercury to the surface, or by dipping it in a solution of a mercury salt. The action of the electrolyte on zinc so treated, when the circuit is open, is reduced to a minimum. The exposed area of the zinc element is reduced so that only so much is sulphated as is necessary for the required amperage. This is found to be about 1-40th of the area of the lead positive plate, and the zinc may be made in the form of a block, cube, or disc, partly covered by an acid-proof impermeable coating so as to reduce the surface to the required extent.—F. Sp.

*Insulating compound and process of making same.* S. Satow, Sendai, Japan. U.S. Pat. 1,245,980, Nov. 6, 1917. Date of appl., Dec. 30, 1916.

AN insulating compound is produced by mixing formaldehyde and a fibrous body with a liquid, adhesive, glue-like mass of glutinised vegetable proteins.—F. Sp.

*Electrolytic cells.* F. G. Wheeler, Appleton, Wis., U.S.A. Eng. Pat. 102,049, Oct. 24, 1916. (Appl. No. 15,137 of 1916.) Under Int. Conv., Oct. 30, 1915.

SEE U.S. Pat. 1,236,025 of 1917; this J., 1917, 1053.

## XII.—FATS; OILS; WAXES.

*Copra; Experiments on the preparation of — in Java.* M. B. Smits, Teysmannia, 1916, 27, 495—501. Bull. Agric. Intell., 1917, 8, 1253.

IT was found that 500 very ripe coconuts weighing 844.35 kilos, yielded 127.15 kilos. of copra, whilst 500 nearly ripe nuts weighing 952.86 kilos, gave 128.75 kilos. (i.e., 150 grms. and 135 grms. per kilo, respectively). There was also no material difference between the yields from nuts picked near the coast and those collected inland. The variety of nuts known as "kerambil sirah" gave a much better yield, both of copra and oil, than the variety "kerambil idjau," which is also common in Java.—C. A. M.



*Catalytic hydrogenation [of oils]; Influence of carbon monoxide on the velocity of —.* E. B. Masted. Faraday Soc., Dec., 1917, 7 pages.

ESPECIAL interest attaches to the effect of carbon monoxide on the catalytic hydrogenation of oils, owing to the fact that hydrogen prepared commercially from water-gas usually contains a small amount of carbon monoxide. In the experiments described, pure neutral olive oil was hydrogenated in a vessel which was mechanically agitated in an oil-bath maintained at 180° C. The hydrogenation vessel was charged with 10 grms. of the oil and a catalyst containing 0.1 gm. of nickel, and, after being exhausted by means of a Geryk pump, in successive experiments pure hydrogen and hydrogen containing from 0.25 to 2.0% of pure carbon monoxide respectively were admitted and the amounts of absorption were measured by reading the volumes of gas left in burettes connected with the hydrogenation vessel. It was found that when the hydrogen contained as little as 0.25% of carbon monoxide, hydrogenation for several hours was necessary to introduce into the oil the same volume of gas as was absorbed in one hour in the case of pure hydrogen. The first traces of carbon monoxide are relatively the most poisonous, and the inhibitive effect of successive increases in the proportion of carbon monoxide decreases with the rise in the proportion. The toxic action of carbon monoxide may be distinguished from the diluting action of any foreign gas which merely obstructs the hydrogenation. This diluting effect may be calculated theoretically, and the result deducted from the total effect experimentally determined.—C. A. M.

*Fir seed oil.* J. Prescher. Pharm. Zentralh., 1917, 58, 533—534.

FIR SEEDS yield a clear yellow drying oil having the following physical and chemical characters:—sp. gr., 0.925; refractometer reading at 25° C., 78.2; iodine value, 155.8; saponif. value 182.6; acid value, 0.8; Reichert-Meißl value, 1.2. The seeds contain about 33% of oil on the dry substance.—W. P. S.

*Iodine value of oils and fats; Comparison between the bromide-bromate method and the methods of Hübl and of Wijs for the estimation of the —.* C. Kelber and H. Rheinheimer. Arch. Pharm., 1917, 255, 417—424.

COMPARATIVE experiments on the estimation of the iodine value of certain fats and oils by Gäbel's method (Arch. Pharm., 1912, 250, 72) and by the methods of Hübl and of Wijs led to the following conclusions. Oils and fats with small iodine values, including hydrogenised oils which before hardening had large iodine values, give concordant results by all three methods provided that a sufficiently long time is allowed for the action of the halogen solution. Oils with large iodine values yield trustworthy results by the methods of Hübl and Wijs; the bromide-bromate method, however, always gives too low results in these cases. The authors prefer Wijs' method to that of Hübl on account of the stability of the solution and the shorter time required for the estimation.—C. S.

*Comparative keeping qualities of palm-kernel, coconut, ground nut [arachis], and other oil-cakes.* Godden. See XIXA.

*Comparative digestibility of palm-kernel cake, extracted palm-kernel meal, and undecorticated cottonseed cake.* Crowther and Woodman. See XIXA.

*Influence of palm-kernel cake upon the composition of milk-fat.* Crowther and Woodhouse. See XIXA.

## PATENTS.

*[Hydrogenising] oils; Process and apparatus for treating —.* C. Ellis, Montclair, N.J. U.S. Pat. 1,247,095, Nov. 20, 1917. Date of appl., May 2, 1917.

THE apparatus consists of a reaction vessel connected with a number of gas-holders by means of pipes and valves. Hydrogen is passed from one of the gas-holders to the reaction vessel containing the oily material and catalyst, the once-used unabsorbed gas being collected in a separate gas-holder. The once-used hydrogen is passed into the reaction vessel a second time and the unabsorbed gas is collected in yet another separate gas-holder. Such partial absorptions and collections of unabsorbed gas are continued until the desired hydrogenation has been effected. A heat-economising device is provided enabling a transference of heat units to take place between gas leaving the reaction vessel and that entering.

—A. DE W.

*Oily materials; Process of treating — preparatory to hydrogenation.* C. Ellis, Montclair, N.J. U.S. Pat. 1,247,516, Nov. 20, 1917. Date of appl., Mar. 15, 1915.

OILY material containing substances inimical to the activity of a nickel catalyst is treated with a finely divided "detoxicating material" containing copper hydroxide. The copper hydroxide and absorbed "catalyst poisons" are then removed from the oil, which is afterwards mixed with nickel catalyst and hydrogenised in the usual way.

—A. DE W.

*Coconut oil; Process for the extraction of —.* G. G. Turner, New York, and C. H. Leighton, Brooklyn, N.Y. U.S. Pat. 1,247,597, Nov. 20, 1917. Date of appl., Apr. 21, 1917.

COCONUT oil is obtained from copra or the like by soaking the finely comminuted "meat" in a solution of sodium chloride of at least 3% strength, with agitation, until the oil is completely separated from the remaining material. The oil, together with a part at least of the other material, is separated by sedimentation and is passed through a centrifugal specific gravity separator and finally clarified.—A. DE W.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

### PATENTS.

*Coloured calcimines [distempers]; Preparation of ingredients for the production of —.* A. B. Merry, Springfield, Mass., Assignor to The Cott-A-Lap Co., Somerville, N.J. U.S. Pat. 1,247,128, Nov. 20, 1917. Date of appl., Mar. 27, 1914. Renewed Oct. 1, 1917.

A COLOURED calcimine is made and preserved ready for use by preparing units of calcimine body material and separate units of colour material differing if necessary as to colour, the calcimine units and colour units being kept separate until shortly before use. The units may be in the form of solid tablets.—A. DE W.

*Lacquer and process of making same. Lacquered product and process of making same.* S. Satow, Sendai, Japan. U.S. Pats. (A) 1,245,981 and (B) 1,245,982, Nov. 6, 1917. Dates of appl., Dec. 30, 1916, and Feb. 1, 1917.

(A) A LACQUER is produced by "glutinsing" vegetable proteins with a phenol to form a liquid,

adhesive, glue-like mass, and treating it with an active methylene compound, e.g., formaldehyde, controlling the consistency if necessary with benzene. (B) Brick, cement, tile, or similar substances are shaped without heat, and the surfaces, preferably after the removal of air, are coated with a glaze consisting of a liquid, adhesive, glue-like mass produced by condensing vegetable proteins with an active methylene compound.

—F. Sp.

*Linoleum-like substance and process of making same.* S. Satow, Sendai, Japan. U.S. Pat. 1,245,978, Nov. 6, 1917. Date of appl., Dec. 11, 1916.

A LINOLEUM-LIKE substance is produced by glutinising vegetable proteins, incorporating with the mass a filler containing cork dust, and rolling the product on to a fabric.—F. Sp.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### PATENT.

*Rubber substitute and process of making same.* S. Satow, Sendai, Japan. U.S. Pat. 1,245,979, Nov. 6, 1917. Date of appl., Dec. 11, 1916.

A RUBBER substitute is produced by mixing glutinised vegetable proteins and vulcanised oil, or by mixing glutinised vegetable proteins with a phenol, an oxidising oil, an active methylene compound, and an alkali, and vulcanising the product.

—F. Sp.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### PATENTS.

*Artificial leather and process of making same.* S. Satow, Sendai, Japan. U.S. Pat. 1,245,977, Nov. 6, 1917. Date of appl., Dec. 11, 1916.

ARTIFICIAL leather is produced by mixing together "glutinised" vegetable proteins, cellulose derivatives, a sticky viscid substance, fibrous material, a condensing agent, and an alkali.—F. Sp.

*Proteidal [plastic] composition and process of making same.* S. Satow, Sendai, Japan. U.S. Pat. 1,245,984, Nov. 6, 1917. Date of appl., May 17, 1917.

VEGETABLE proteins are separated from material containing them, then "glutinised," and mixed with the residue of the material from which they were separated to form a plastic composition.

—F. Sp.

*Binding and cohering composition.* U.S. Pat. 1,247,595. See I.

#### XVI.—SOILS; FERTILISERS.

*Ammonification [in soils]; Influence of the chemical structure of the compounds to be ammonified upon the rate of —.* K. Miyake. J. Amer. Chem. Soc., 1917, 39, 2378—2382.

THE rates of ammonification of leucine, tyrosine, acetanilide, benzanilide, acetamide, and benzamide in five different soils were observed. The soils were air-dried and sifted and then mixed with the compound and water in the proportion of 100 grms. of soil, a quantity of the compound equivalent to 100 mgrms. of nitrogen, and 10 c.c. of water. Samples were withdrawn at intervals up to 16 days and the ammonia produced determined by distillation with magnesia. The average figures of the highest amounts of ammonia produced (in mgrms. of ammonia-nitrogen from 100 mgrms. equivalent nitrogen per 100 grms.

of soil) were as follows:—acetamide, 88.36; leucine, 76.46; tyrosine, 59.28; benzamide, 8.07; acetanilide, 5.28; benzanilide, 5.30. The variation among the soils was considerable, the extreme figures, e.g., for acetamide being 71 and 95.5 and for benzanilide a trace and 14. The author concludes that fatty compounds are more easily ammonified than aromatic compounds, and compounds containing the amino group more easily than those containing the amino group.—B. V. S.

*Nitrogen transformations in soils; Influence of sodium nitrate upon — with special reference to its availability and that of other nitrogenous manures.* D. A. Coleman. Soil Sci., 1917, 4, 345—432.

A VERY full historical review of the literature is given and the results referred to are discussed to some extent. The influence of sodium nitrate upon nitrogen transformations in soils, as shown by its effect upon the ammonifying, nitrifying, and nitrogen-fixing powers of seven types of soil, has been studied. Applications of sodium nitrate markedly increased the simplification of protein substances applied to the soil, the increase varying with the organic substance used, cottonseed meal being ammonified to a greater extent than dried blood. Acid phosphate (superphosphate) increases the ammonification of dried blood nitrogen but potash in the form of potassium chloride has very little effect. In alkaline soils sodium nitrate loses to a large extent its stimulating effect owing to an increase in the numbers of bacteria which assimilate the simplified material and also owing to a rearrangement of the soil flora. Of the soil flora studied, the fungi respond most to the application of sodium nitrate, and the bacteria next, the stimulating effect being due to the anion. Sodium nitrate at first stimulates the nitrification of dried blood, cottonseed meal, and, to a lesser extent, of ammonium sulphate, but when present in large quantities it depresses nitrification. The same is true of its effect on nitrogen fixation by azotobacter. For dressings up to 5000 lb. per acre, the effect of sodium nitrate on the activity of nitrobacter is the same as on that of nitrococcus, but with larger dressings the activity of nitrobacter is inhibited. The action of this salt is thus most adverse towards the nitrogen-fixing bacteria, less detrimental to the nitrifying group, and still less to the ammonifying group. As applied in agricultural practice it generally enhances the activity of the two latter groups, whilst depressing the activity of the first group of organisms.—W. G.

*Farmyard manure; Changes taking place during the storage of —.* E. J. Russell and E. H. Richards. J. Agric. Sci., 1917, 8, 495—563.

THE variation of the dried matter and combined nitrogen occurring during storage of farmyard manure under various conditions was investigated. It is shown that, owing to an oscillation between aerobic and anaerobic conditions in the heap, considerable loss of nitrogen in the free state occurs. The authors explain this loss of free nitrogen on the general hypothesis that, under anaerobic conditions, molecular groupings tend to arise which become unstable as soon as aerobic conditions set in, and *vice versa*. It is known that, on the entry of air, certain carbon compounds formed under anaerobic conditions may be converted into compounds with shorter chains, by the elimination of  $\text{CH}_4$ , and it is suggested that some complex nitrogen compound may be converted into a simpler compound by elimination of nitrogen. Alternate nitrification and denitrification in the heap is but a special case of this. The conditions to be aimed at in storing farmyard



manure are complete anaërobic conditions, preferably at 26° C., and further experiments on the practical side are in progress in connection with this point.—W. G.

*Fumigation with hydrocyanic acid: Studies in greenhouse*—*Physiological effects on the plant*. W. Moore and J. J. Willaman. *J. Agric. Res.*, 1917, 11, 319—338.

THE presence of hydrocyanic acid in the plant after fumigation was proved by grinding up the tomato plants used, adding 5% tartaric acid solution, and slowly distilling off any hydrocyanic acid into caustic soda. As much as 9.01% of hydrocyanic acid in the dry plant was present in some cases, and this caused severe injury to the plant. The acid gradually disappeared within a few hours in all cases. The immediate effect of the presence of the acid in the plant was a sudden reduction in the oxidase and catalase activity after fumigation, followed by a fairly rapid and permanent recovery. Resulting from these respiratory changes, there was an inhibition of photosynthesis and of the translocation of starch, which required several days for complete restoration. There was also an increased permeability in the leaf septa and a more rapid cuticular respiration, which led to wilting of the plant; if the fumigation had been severe, the wilting was permanent and the plant died. There was a stimulation of growth in those plants which had been fumigated without injury and an increase in the yield of fruit. No change was found in the chemical composition of the plants as a result of the fumigation.—J. H. J.

*Hydrocyanic acid gas as a soil fumigant*. E. R. de Ong. *J. Agric. Res.*, 1917, 11, 421—436.

EXPERIMENTS were made to determine the relation between the least dose of hydrocyanic acid which was toxic to insects and the largest dose which was non-toxic to plants. The physical and chemical action of the gas in soil was also investigated. A solution of 1 gm. of sodium cyanide in 1 litre of water acidified with dilute sulphuric acid, when placed in an enclosed space, caused the air of the space to contain 0.3% of hydrocyanic acid. Other strengths of hydrocyanic acid in air were prepared similarly, and various insects placed in the air space. 0.0046% of the gas in air was the least toxic amount for the house fly, whilst beetles required 0.0365%, 0.0046% of the gas also retarded the germination of lettuce seed, although the seeds were not killed by much higher concentrations. Seedlings were not affected by treatment twice as strong as the toxic dose for flies, and cuttings were very much more resistant. For experiments with soil, it was found necessary to generate the gas in the soil from sodium cyanide and sulphuric acid. Water and soil absorbed the gas very strongly, and the variability of the absorption by soils of varying characters prevented any determination of the amount necessary to destroy insects and at the same time be safe for plants. A sandy soil took up the gas in proportion to the water present, but in a clay soil the gas entered into combination with the soil constituents or else was adsorbed by the soil particles. The gas diffused very slowly in wet soils, but fairly rapidly in moist soils. It proved satisfactory as a fumigant for porous soil, seed beds, and potting soil.—J. H. J.

## XVIII.—FERMENTATION INDUSTRIES.

*Wine: Separation and determination of lactic, succinic, and malic acids in*—*J. Laborde*. *Comptes rend.*, 1917, 165, 793—795.

*Dry wines*. Fifty c.c. of the wine is neutralised with potassium hydroxide solution, 5% mercuric acetate solution is added in quantity just sufficient

to precipitate the tannins, the mixture is heated at 50° C., filtered, and the precipitate washed twice with warm water. To the filtrate is added a quantity of N/1 sulphuric acid equal to the amount of alkali used for the neutralisation plus a further amount equivalent to the difference between the total alkalinity of the ash expressed as potassium bitartrate and the alkalinity of the potassium bitartrate corresponding with the total tartaric acid present. The solution is then evaporated to a syrup, water is added, and the evaporation repeated. The residue is treated with animal charcoal (2 grms.), sand, and 25 c.c. of 95% alcohol, and extracted with ether (50 c.c.), the extraction being twice repeated with 15 c.c. of a mixture of equal volumes of alcohol and ether. The extracts are filtered, the filtrate is evaporated to a few c.c., and this residual solution is treated with 0.2 gm. of animal charcoal and 10 c.c. of water, and filtered. The filtrate is neutralised, while boiling, with alkali solution (calcium hydroxide solution), evaporated to 8 c.c., 1 drop of 50% acetic acid is added, followed by 90 c.c. of warm 95% alcohol. The mixture is heated to boiling and the precipitated calcium malate and succinate are filtered off and washed with hot 85% alcohol; a determination of calcium in the filtrate gives the quantity of lactic acid present. The precipitate is then dissolved in boiling water (a small amount of calcium phosphate may remain insoluble), the solution is evaporated to 8 c.c. and treated with 1 c.c. of glacial acetic acid and 90 c.c. of 95% alcohol. The mixture is boiled and the precipitated calcium malate is collected and washed with 85% alcohol acidified with acetic acid. Determinations of calcium in the precipitate and filtrate, respectively, give the quantities of malic and succinic acids present. If citric acid is contained in the wine it is precipitated with the malic acid. *Sweet wines*. After removal of tannins and treatment with sulphuric acid, the solution is evaporated to a syrup, 10 to 20 c.c. of alcohol is added and the mixture is extracted with 50 c.c. of ether. After the ethereal layer has been decanted, the extraction with alcohol and ether is repeated once or twice. The extracts are then evaporated and the residue used for the determinations as described. Normal wines contain about 0.70 gm. of succinic acid per litre; the quantities of lactic and malic acids vary considerably according to the origin and quality of the wine.—W. P. S.

*Carbone method for retting textile plants by microbiological action*. Carbone and Tombolato. See V.

*Enzymes of milk and butter*. Thatcher and Dahlberg. See XIXA.

*Digestibility of dried yeast*. Crowther and Woodman. See XIXA.

### PATENT.

*Process of obtaining proteid matter from maize*. U.S. Pat. 1,245,818. See XIXA.

## XIXA.—FOODS.

*Milk and butter: Enzymes of*—*R. W. Thatcher and A. C. Dahlberg*. *J. Agric. Res.*, 1917, 11, 437—450.

IN the case of milk it was found that the use of 1% of chloroform to prevent bacterial action prevented proteolysis also, and the same result was brought about by 15% of sodium chloride. In following the protease in fresh milk through the process of butter making, it was found that protease was slightly increased in the separated cream and was highly concentrated in the separator slime, and, in general, the protease increased along with the increase in the proportion of

casein nitrogen in the total nitrogen. In butter the protease cannot act because of the high salt content. The activity of any lipase in butter was too small to be accurately measured. No oxidase could be found in milk and butter, and the amount of peroxidase was very small. Catalase was present in milk and butter. The enzyme content of butter was very small on account of the high dilution with fat, and it is concluded that enzymic action is not a factor in the deterioration of butter in cold storage.—J. H. J.

*Palm-kernel cake; Influence of — upon the composition of milk-fat.* C. Crowther and H. Woodhouse. J. Agric. Sci., 1917, 8, 451—454.

PALM-KERNEL cake at the rate of 2 lb. per head per day was fed to cows at grass. Samples of the milk-fat from each cow were taken twice a week and the following determinations made thereon:—Koettstorfer (saponification) value; Reichert-Wollny value; Polenske value; iodine absorption, and refractive index. The results with each cow show a rise in the first three values and a fall in the iodine absorption and refractive index, as an apparent consequence of feeding with palm-kernel cake. The differences in the cases of the Reichert-Wollny and Polenske values were, however, small in comparison with the probable errors.—W. G.

*Palm-kernel, coconut, ground-nut [arachis], and other oil-cakes; Comparative keeping qualities of —.* W. Godden. J. Agric. Sci., 1917, 8, 419—428.

PALM-KERNEL, coconut, and ground-nut cakes were compared with linseed, undecorticated cottonseed, "soycot," and soya cakes. So far as keeping properties are concerned, palm-kernel cake compares favourably with most of the oil-cakes commonly used on farms. During storage under ordinary farm conditions the only change which occurs is one common to all the oil-cakes examined, namely, an increase in the free fatty acid content of the oil. During incubation in a moist state at 37°C., on only four of the cakes examined, namely cottonseed, ground-nut, "soycot," and soya cakes, was there any marked development of moulds, this being accompanied by a loss of organic matter, the loss being distributed between the oil and the soluble carbohydrates of the cake. Dry storage is essential to prevent moulding and the consequent loss of organic matter.—W. G.

*Palm-kernel cake, extracted palm-kernel meal, and undecorticated cottonseed cake; Comparative digestibility of —.* C. Crowther and H. E. Woodman. J. Agric. Sci., 1917, 8, 429—447.

Two kinds of palm-kernel cake, one of cottonseed cake, and one of palm-kernel meal, were fed in turn to two sheep on a basal ration of "seeds" hay, and from the experimental results the following digestion coefficients were calculated, making due allowance for the presence of metabolic products in the faeces:

	Undecort. cotton- seed cake.	Palm kernel cake.	Extracted palm kernel meal.
Organic matter .....	% 55.0	% 70.8	% 76.7
Crude protein .....	67.4	75.4	79.2
Crude protein (corrected) ..	74.7	88.5	90.0
Ether extract .....	(100 ?)	98.2	96.3
Nitrogen-free extractives ..	62.0	78.6	86.0
Crude fibre .....	34.6	20.6	44.8

—W. G.

*Yeast; Digestibility of dried —.* C. Crowther and H. E. Woodman. J. Agric. Sci., 1917, 8, 448—459.

THE digestion coefficients obtained with a "low-grade" sample of dried yeast fed to sheep along with "seeds" hay were:—organic matter, 93.5%; crude protein, 96.6%; true protein, 96.6%; nitrogen-free extractives, 96.9%. Owing to the very small proportions of ether extract and crude fibre in the dried yeast, it was not possible to measure the digestibility of these ingredients.—W. G.

*Cocoa powders; Examination of — for the content of husks.* O. Keller. Arch. Pharm., 1917, 255, 405—416.

ALL cocoas examined by the author during the last six months have been adulterated with husks, and a simple, trustworthy method of estimating the latter is very desirable. The author criticises various chemical and mechanical methods which have been proposed and describes in some detail a method based on his observations that the ethereal extract of pure cacao kernels is colourless or faintly yellow whilst the extract of the husks is distinctly brown (Apoth.-Zeit., 1915, 560). Two grams of the cocoa powder and 15 c.c. of ether, are frequently shaken for 24 hours in a well-stoppered vessel at the ordinary temperature, the shaking being discontinued during the last 2 or 3 hours. Ten c.c. of the supernatant liquid is pipetted into a test-tube and filtered through kieselguhr, repeatedly if necessary, until a clear filtrate is obtained, into one comparison tube of a colorimeter. The test-tube and filter are washed with so much ether that after the washings have been added to the solution in the comparison tube the liquid has a depth of 5 cm. The second comparison tube contains water having a depth of 5 cm. and to it is added ferric chloride (the liquid must be freshly prepared from officinal *liq. ferri sesquichlor.* and contain 0.1 grm. of iron in 100 c.c.) until the colours of the liquids viewed from above have the same strength. The comparison is repeated, with the difference that the quantity of water is diminished by a volume equal to that of the ferric chloride solution added. The volume of ferric chloride solution required increases with the husk content of the powder. Pure cocoa powder containing 54% of fat requires 2.4 c.c. of the ferric chloride solution; pure husk-free powder requires 1.4 c.c.; pure husks require at least 3.5 c.c., the average value being 4.4 to 4.5 c.c. The author is of opinion that powders containing 20% or more of fat and requiring 2.5 c.c. or more of the ferric chloride solution have been adulterated with husks.—C. S.

#### PATENTS.

*Condensed buttermilk and buttermilk powder; Process and apparatus for the production of —.* E. C. R. Marks. London. From Merrell-Soule Co., Syracuse, N.Y., U.S.A. Eng. Pat. 111,340, Nov. 24, 1916. (Appl. No. 16,857 of 1916.)

BUTTERMILK is concentrated by causing it to flow, by centrifugal action, upwards in the form of a thin layer over the interior walls of a heated vertical cylinder from which the air is exhausted. The concentrated buttermilk escapes from the top of the cylinder and, after passing through a heating coil, is atomised into a chamber, a current of dry air also being introduced into the chamber.—W. P. S.

*Milk; Modified — [for infants].* E. C. R. Marks. London. From Merrell-Soule Co., Syracuse, N.Y., U.S.A. Eng. Pat. 111,341, Nov. 24, 1916. (Appl. No. 16,859 of 1916.)

THE modified milk is obtained by mixing milk,



cream, and whey in suitable proportions, *e.g.*, 270 lb. of milk containing 3.5% of fat, 45 lb. of cream containing 26% of fat, and 1000 lb. of whey are mixed. The mixture is sterilised at 180° F. (82° C.), and converted into a dry powder by atomising it into a current of dry air. The resulting powder contains approximately: fat 18, lactose 57.8, casein 8.6, lactalbumin 7.5, ash 7.3, and water 1.2%, and is composed of spherical particles practically devoid of bacteria but containing active enzymes.—W. P. S.

*Canning; Process of*—K. Miyauchi, Kago-shima-Ken, Japan. U.S. Pat. 1,245,489, Nov. 6, 1917. Date of appl., Apr. 23, 1917.

A LAYER of "degutinated" and deodorised wood shavings is placed over the interior surface of the tin receptacle so as to prevent the contained substance from coming into contact with the metal.—W. P. S.

*Protein matter from maize; Process of obtaining*—J. Takamine, New York, and S. Satow, Sendai, Japan. U.S. Pat. 1,245,818, Nov. 6, 1917. Date of appl., Nov. 25, 1916.

GROUND maize is cooked and the gelatinised starch is saccharified; the mass is then fermented and distilled. The proteins are separated from the residual liquid, dissolved in an alkali solution, and then precipitated by the addition of an acid.

—W. P. S.

## XIXB.—WATER PURIFICATION; SANITATION.

*Water; Identification and determination of zinc in*—R. Meldrum. Chem. News, 1917, 116, 295—296, 308—310.

THE ferrocyanide reaction may be used for the detection and determination of zinc in water; it is capable of detecting 1 part of zinc in 1 million parts of water. One hundred c.c. of the water is acidified with 1 c.c. of 20% hydrochloric acid in a glass cylinder and, after 1 min., 1 c.c. of 1% potassium ferrocyanide solution is added. An opalescence appears within 20 mins. if zinc is present and the quantity of the metal is found by comparison with standards containing known amounts of zinc. In preparing the standard zinc solutions, the same kind of water as the sample, but free from zinc, must be employed, since the sensitiveness of the test is influenced by the salts, *etc.*, present. If a quantity of the water cannot be obtained before it is contaminated with zinc, an artificial water may be prepared of corresponding composition as regards saline substances. When distilled water is used the results may be from 50 to 100% too high. (See also this J., 1918, 19 A.)

*Sewage and its precipitation.* R. Brown. Soc. of Engineers, Nov. 19, 1917. [Reprint.] 12 pp.

EXPERIMENTS on the large scale, extending over about a fortnight in each case, were made with three precipitants: (1) lime, (2) ferric sulphate solution and lime, (3) aluminium sulphate and lime. The sewage treated was acid from acetic acid and passed through a detritus tank before the precipitant was added; afterwards it passed through a continuous-flow precipitation tank. The amounts of the precipitants used per gallon were: 7.6 grains of lime, 2 grains of ferric sulphate plus 4 grains of lime, and 1.88 grains of aluminium sulphate plus 2.8 grains of lime. The percentage purification obtained varied from 35 to 49 in oxygen absorption, 33 to 43 in albuminoid ammonia, 70 to 78 in suspended solids, and 21—27 in colloids; in each case the highest figures were given by aluminium sulphate and lime. The

results obtained with ferric sulphate solution and lime were about the same as those previously obtained by the author with solid ferric sulphate and lime.—J. H. J.

*Studies in greenhouse fumigation with hydrocyanic acid. Physiological effects on the plant.* Moore and Willaman. See XVI.

*Hydrocyanic acid gas as a soil fumigant.* De Ong. See XVI.

## PATENTS.

*Sewage; Treatment of—*for the purification thereof and the production of combustible gas therefrom. H. Flicker, Parramatta, N.S.W. Eng. Pat. 111,401, Feb. 27, 1917. (Appl. No. 2893 of 1917.)

THE sewage, together with a regulated quantity of air, is admitted into the upper part of a closed tank, sprinklers distributing the incoming liquid over the surface of sewage already contained in the tank. The tank is provided with a sludge pit and with a partition extending to the surface of the sludge, the liquid passing below this partition on its way to the exit from the tank. The upper portion of the sludge is thus disturbed and accumulated gases are liberated. The gases resulting from the decomposition of the sludge are collected at the top of the tank and may be utilised for heating water to raise the temperature of the contents of the tank when necessary in order to accelerate bacterial action.—W. P. S.

*House waste and sewage; Method of and apparatus for treating*—E. L. McGill, New York. Eng. Pat. 111,422, Apr. 18, 1917. (Appl. No. 5469 of 1917.)

THE sewage is delivered into the first of a series of covered tanks situated on an incline so that the liquid may pass from one to another. Longitudinal partitions near the sides of the tanks form channels which are filled with sand up to the level of the liquid in the central part. The liquid passes under the lower edges of the partitions, rises upwards through the sand, and is discharged on to a sand filter and thence into a stream or over land. Raking devices are provided for agitating the sand in the channels, and air is circulated through the tanks. The tanks may be arranged in parallel series so that one set may be in use while the other set is being cleaned.—W. P. S.

*Sewage and analogous liquids; Purification of*—W. Jones, Stourbridge. U.S. Pats. 1,247,540—1,247,543, Nov. 20, 1917. Dates of appl., Oct. 9, 1914, Sept. 13, Oct. 18, and Oct. 18, 1915.

SEE Eng. Pats. 22,952 of 1913, 1141 of 1915, and 22,737 and 22,736 of 1914; this J., 1915, 244; 1916, 326, 195.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Tobacco; Effect of some alkaline salts upon the fire-holding capacity of*—H. R. Kraybill. Bot. Gazette, 1917, 64, 42—56. Bull. Agric. Intell., 1917, 8, 1256—1258.

TOBACCO leaves were sprayed with 28.9% solutions of the respective salts, and, after being left under a bell jar, pieces were cut from the tip, middle, and base of each leaf, and the duration of their combustion determined. The results showed that the carbonates of caesium, rubidium, and potassium have a pronounced effect on the fire-holding capacity of tobacco, whereas the carbonates of lithium and sodium have not this effect. Of the oxalates tried only potassium oxalate had any effect. Potassium citrate also promotes the

burning properties, but the citrates of sodium and lithium have practically no action. Organic salts of potassium, tripotassium phosphate, dipotassium phosphate, and potassium sulphate promote the combustion, but the chloride, acid sulphate, and monopotassium phosphate have a bad effect. The favourable action of potassium salts is not due to their reduction, nor is the injurious effect of chlorides due to their melting, but it seems probable that the favourable salts may have a specific catalytic action upon the combustion, whilst chlorides have a negative catalytic action. Possibly the effect of salts in raising the temperature may also play a part in the process.—C. A. M.

*Uzara root; Chemical constituents of* —. W. Hennig. Arch. Pharm., 1917, 255, 382—405.

UZARA root is of medicinal value on account of its anti-diarrhetic properties. The drug is procurable under the name "uzaron," which is the dried alcoholic extract of the root. From uzaron the author has isolated two glucosides having different physiological effects, one of which is amorphous and is present in very small amount, whilst the other, *uzarin*,  $C_{15}H_{108}O_{30} \cdot 911_2O$ , forms colourless needles, m.pt. about  $210^{\circ}C$ . decomp. about  $200^{\circ}C$ . On hydrolysis uzarin yields propyl alcohol, dextrose, and uzaridin,  $C_{15}H_{124}O_4 \cdot 11_2O$ , colourless leaflets, decomposing about  $246^{\circ}C$ . (anhydrous), the last substance being partly changed to anhydrouzaridin,  $C_{15}H_{122}O_4 \cdot 11_2O$ , colourless needles, decomp. about  $208^{\circ}$ — $214^{\circ}C$ . (anhydrous). Uzaridin forms a triacetyl derivative, needles, m.pt.  $225^{\circ}$ — $227^{\circ}C$ . —C. S.

*Frail of Asparagus officinalis*. L. N. E. Helmer. Chem. News, 1917, 116, 296—297.

THE red berries of this plant contain:—Reducing sugars, 36.12; oil, 1.08; proteins, 9.75; ash, 3.5%. Malic acid is present in large amount, together with a trace of citric acid, but tartaric and oxalic acids are absent.—W. P. S.

*Iron; Determination of metallic* — in *Ferrum reductum*. A. Eberhard. Arch. Pharm., 1917, 255, 357—381.

THE author finds that the simple method described by Winkler (Z. angew. Chem., 1917, 64) is not trustworthy, since the increase in weight of the sample does not attain a constant value even after the ignition in air for 15—20 minutes has been repeated five or ten times; moreover, by the prolonged ignition the triferic tetroxide originally present in the *ferrum reductum* is partially converted into ferric oxide, with the result that the percentage of metallic iron found is too high. The author has examined the official methods adopted in different countries. He recommends that, in addition to the total iron, the metallic iron be estimated by Merck's mercuric chloride method; 25 c.c. of the filtrate should be used, the solution of potassium permanganate should be dilute (2—2.5 grms. per litre), and the confirmatory iodometric estimation may be omitted. The author has examined a method suggested by Schmidt, in which bromine instead of iodine is used for the solution and estimation of the iron. The method fails on account of the unavoidable loss of bromine vapour during the estimation.—C. S.

#### PATENTS.

*Blood-coagulating substance; Manufacture of a* —. O. Inray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 111,533, Nov. 30, 1916. (Appl. No. 17,218 of 1916.) Addition to Eng. Pat. 11,215 of 1915 (this J., 1916, 72).

[SEE Ger. Pat. 296,678 of 1916; this J., 1917, 615.]

An alternative process consists in mixing an alcoholic extract of the blood with water and then extracting with an organic solvent, other than a chlorinated hydrocarbon, e.g., ether, benzene, or toluene. The original process or any of the modified processes may be applied directly to freshly drawn blood.

*Opium; Product derived from* — and process of making same. A. Gams, Assignor to Soc. of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,243,729, Oct. 23, 1917. Date of appl., Mar. 24, 1917.

OPIUM extracted with water is treated with alcohol containing formic acid, which forms stable salts with narcotine, papaverine, and thebaine; the alcohol and excess of formic acid are removed from the solution and the separated inactive substances added to the aqueous extract; after diluting the mixture with water to precipitate mucilaginous substances, with or without subsequent addition of a lead salt to remove dissolved albuminous substances and subsequent removal of the excess lead, the filtered liquid is evaporated *in vacuo*. The product is suitable for injections, containing all the alkaloids of opium in their natural forms of combination and proportions.

—F. W. A.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*X-ray materials; Sensitometry of* —. M. B. Hodgson. Communication No. 63 from The Kodak Research Laboratory. Brit. J. Phot., 1917, 64, 654—657.

THE author used a modified form of the exposing apparatus designed by Jones, in which a series of exposures was given by moving the plate across a lead-protected diaphragm; this avoided the difficulty of synchronisation which occurs by reason of the intermittent nature of X-radiation, if the usual revolving sector wheel is employed. A Coolidge tube was used to obtain a steady output of X-rays in a continued exposure, the usual type of tube not being satisfactory in this respect. No unit of radiation is suggested, an arbitrary unit being employed in the curves given. These do not quite resemble those obtained by light exposures, being more hyperbolic in shape and showing no over-exposure period. Curves are given showing the effects of variation in development, exposure, and hardness of tube, and also the effect of using an intensifying screen. The two former are quite in line with light-exposures. With increasing softness of tube, as measured by the equivalent spark-gap, the curve gets steeper and the useful exposure range smaller; an intensifying screen gives greater speed and greater steepness.—B. V. S.

*Fixation [photographic]; Laws of* —. A. W. Warwick. American Photography, 1917. Brit. J. Phot., Dec. 7, 1917, 617—620.

THE amount of silver salt removed from a film by a bath of sodium thiosulphate was determined by the colour reaction of the solution with sodium sulphide, compared with a solution of known content. Under normal conditions the reaction follows the usual rule, a certain fraction of the remaining silver salt being removed in unit time; with 20% solution at  $65^{\circ}F$ . ( $18^{\circ}C$ .) the rate is roughly half the silver in 2 minutes. The total time necessary for efficient fixation is dependent on this factor and on the actual silver content of the film; a convenient guide is the time taken to "clear" the film (remove the opalescence), which is about half that required for complete fixation. The factor depends on the temperature, the character of the silver salt in the film, and the



strength of the fixing bath; the rate rises gradually with increase of strength up to about 40% and then falls away somewhat more quickly, being zero again at saturation, about 63% at 65° F. (18° C.).

—B. V. S.

*Iodine and iodine-thiourea as subtractive reducers for photographic negatives and positives.* S. Becher and M. Winterstein. Z. wiss. Phot., 1917, 17, 1—16.

IODINE can be used as a photographic reducer in solution in potassium cyanide, in potassium iodide, or in alcohol and also in combination with thiourea. The solution in cyanide is not satisfactory in use because of the continued action after removal of the image from the reducing bath; treatment with "hypo" stops the action of the iodine but not that of the cyanide, which is still considerable as cyanide itself has a definite reducing action. The other methods of using iodine are free from this defect, "hypo" having an immediate arresting effect on their action; on this account very thorough washing is necessary before treatment with an iodine reducer. Very dilute solutions are recommended:—For plates (a) 1 to 4 c.c. of iodine-potassium iodide solution (1:2:200) in 100 c.c. of water; (b) 2—8 c.c. of the iodine-potassium iodide to 100 c.c. of 4% thiourea solution; for paper, solutions about half the strength of those for plates or a solution of from 4 to 16 c.c. of iodine tincture (1:100 c.c. of 95% alcohol) to 100 c.c. of 50% alcohol. The time necessary for reduction varies from 1 min. to about 6 mins. with the first two baths and up to as long as 10 mins. with the alcohol solution. The potassium iodide and alcohol solutions give a yellowing of the image by the formation of silver iodide but it is quite easy to judge the amount of reduction. A final bath of "hypo" is necessary to dissolve the iodide and to arrest the action of the reducer, for the latter reason also after the thiourea bath. The strength of the thiourea solution must not be appreciably higher than 4%, as although the authors were unable to confirm previous statements that thiourea has itself a reducing action, in stronger solution its destructive action on the gelatin film is quite marked. The reducing action of iodine was compared with that of other reducers: it resembles cyanide and Farmer's ferricyanide-hypo reducer in acting evenly over the whole image, in contrast with permanganate which acts proportionally to the depth of the image and copper chloride and ammonium persulphate which act more strongly on the shadows. The solution in potassium iodide but not the alcohol or thiourea solution gives the usual blue colour with papers containing starch; it disappears, however, immediately in the subsequent "hypo" bath.—B. V. S.

#### PATENTS.

*Photographic negatives; Process for improving* —. A. Boularan, dit Devel, Paris, U.S. Pat. 1,245,152, Nov. 6, 1917. Date of appl., Sept. 15, 1916.

A TRANSPARENCY from an intensified negative is coated with coloured bichromated gelatin, dried, and exposed through the back. After development of the resulting negative the silver image (of the positive) is removed by treatment with a bath of potassium ferri cyanide and sodium thio-sulphate.—B. V. S.

*Colour photography.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,247,116, Nov. 20, 1917. Date of appl., Mar. 12, 1915.

In making a coloured print from a three-colour

mosaic transparency, three monochrome images in silver are first made, corresponding with the three colours of the transparency. These are then transformed to images in the complementary colours to those of the transparency and the three resulting prints superposed in register. For the blue print, complementary to red, the silver is replaced by Prussian blue in the usual way. The other colours are obtained by bleaching the print in bichromate and bromide, developing in hot water to form colloid relief prints, and then dyeing to the required colour.—B. V. S.

#### XXIII.—ANALYSIS.

*Platinum ware; Some substitutes for* —. R. F. Heath. Met. and Chem. Eng., 1917, 17, 666—667.

COMPARATIVE tests were made on crucibles made of platinum, gold, silver, nickel, fused quartz, and an alloy of canadium (see this J., 1912, 29), 1 part, palladium, 1 part, platinum, 2 parts, and nickel, 6 parts. In the first test, heating the weighed crucibles 32 hrs., cooling, and reweighing, platinum gave distinctly the poorest result, losing 0.14% as against the next highest, silver 0.017%. In the tests by immersion in strong acids (hydrochloric, sulphuric, and nitric) platinum gave each time the lowest figure, but gold, canadium alloy, and quartz all gave good results, the highest figures being 0.04% loss for gold in hydrochloric acid, and 0.02% for the canadium alloy in nitric acid; silver gave a better test than gold in hydrochloric acid but a high figure in sulphuric acid, and nickel was not good in either of them. The particular merits and demerits of the various materials as regards their resistance to halogens, alkalis, high temperatures, etc., are also considered. The canadium alloy is a good general substitute for platinum, having similar resisting powers and a melting point of about 1570° C.; its cost is about half that of platinum.—B. V. S.

*Fractional distillation under diminished pressure; An efficient apparatus for* —. W. A. Noyes and G. S. Skinner. J. Amer. Chem. Soc., 1917, 39, 2718—2720.

A SEPARATING funnel is fused into the side of a Claisen flask, thus permitting the introduction of successive fractions without losing the vacuum, and a simple fractionating column is sealed on to the side tube of the flask.—W. G.

*Micro-analysis; Quantitative* —. C. Reichard. Pharm. Zentralh., 1917, 58, 493—498, 534—540.

INSTANCES are given of the applicability of micro-analysis, i.e., the determination of very small quantities of constituents when using minimum amounts of material. Provided that accurate measuring vessels and a sensitive balance are employed, it is quite possible to determine accurately the extractives and ash in 5 c.c. of wine, etc.; small capsules formed out of platinum foil are used for the purpose. Micro-analysis is particularly suitable for the determination of phosphorus, since this yields a precipitate of ammonium phosphomolybdate having a weight fifty-five times that of its phosphorus content. In the determination of small quantities of carbon, it is preferable to use a gas-volumetric method, the volume of carbon dioxide produced from the carbon being readily determined; the same applies

to nitrogen, ammonia, and hydrogen. Very small quantities of metal in oxides (e.g., zinc in zinc oxide) may be determined by measuring the volume of hydrogen evolved when the substance is treated with an acid.—W. P. S.

*Manganese; Colorimetric determination of — by oxidation with periodate.* H. H. Willard and L. H. Greathouse. J. Amer. Chem. Soc., 1917, 39, 2366—2377.

MANGANESE salts are oxidised by periodates according to the equation:  $2\text{Mn}(\text{NO}_3)_2 + 5\text{KIO}_4 + 3\text{H}_2\text{O} = 2\text{HMnO}_4 + 5\text{KIO}_3 + 4\text{HNO}_3$ , and the amount of permanganate produced may be determined by comparison with a solution of known strength. A slight excess of periodate is necessary to ensure complete oxidation of the manganese salt, but the only effect of increasing the excess is the acceleration of the reaction. A minimum excess of acid is also necessary to complete the reaction and to avoid precipitation; the minimum increases with increase of the amount of manganese and varies with different acids, being lowest with phosphoric acid. Heating is necessary, boiling for 10—15 mins. being the usual method. If chlorides are present they are decomposed, and the chlorine driven off by boiling. Ammonium salts do not interfere with the reaction. The amount of manganese present has no effect except that with large amounts the colour is too strong for accurate matching. Reducing substances such as ferrous salts must first be oxidised with nitric acid to prevent the formation of iodine. If ferric salts are present their disturbing effect on the colour may be counteracted by the addition of an equal amount to the standard solution. The colour is very permanent in presence of an excess of the periodate. The method is applicable to the analysis of iron and steel, of which several examples are given. A comparison is also given with the persulphate oxidation of manganese and a bibliography of the subject.—B. V. S.

*Cupferron (phenyl-nitrosohydroxylamine-ammonium); Use of — in: I. Quantitative separation of zirconium, titanium, iron, manganese, and aluminium. II. Analysis of zircon and baddeleyite.* J. Brown. J. Amer. Chem. Soc., 1917, 39, 2358—2366.

IN the analysis of mixtures of salts of iron, zirconium, titanium, aluminium, and manganese, cupferron in acid solution was used for the separation of the first three elements. The metals were brought into solution in sulphuric acid, an excess of about 12½% of acid being present. The cupferron precipitate, after thorough washing, was ignited to remove the excess of cupferron, the resulting oxides fused with bisulphate and dissolved in dilute sulphuric acid. The iron was separated as sulphide in the presence of tartrate. Three methods were used for the determination of the zirconium in the residual filtrate, after oxidation of the tartaric acid. When large amounts of titanium were present, the zirconium and titanium were precipitated together as hydroxides and weighed as oxides, and then either the titanium determined by reduction with sulphur dioxide and titration with permanganate and the zirconium determined by difference, or the zirconium separated as phosphate, the operation being repeated to remove the last traces of titanium. When smaller amounts of titanium were present, Weller's colorimetric method with hydrogen peroxide was used for the determination of titanium, and the zirconium was determined as

phosphate. The separation of the aluminium and magnesium was made by standard methods. In the analysis of zircon, which contains no manganese, a preliminary treatment for the removal and estimation of silica was necessary; in the analysis of baddeleyite, which contains manganese, water as well as silica had to be determined.—B. V. S.

*Fractionating apparatus for petrol and similar volatile substances.* Hildt. See II A.

*Laboratory methods for benzol recovery plant operation.* Sperr, jun. See III.

*Examination of logwood.* See IV.

*Use of sodium paratungstate in the determination of metallic oxides from cyanides.* Kuzirian. See VII.

*Comparison between the bromide-bromate method and the methods of Häbl and Wijs for the estimation of the iodine value of oils and fats.* Kelber and Rheinheimer. See XII.

*Separation and determination of lactic, succinic, and malic acids in wine.* Laborde. See XVIII.

*Examination of cocoa powder for the content of husks.* Keller. See XIX A.

*Identification and determination of zinc in water.* Meldrum. See XIX B.

*Determination of metallic iron in Ferrum reductum.* Elberhard. See XX.

## PATENT.

*Gases; Method of and apparatus for testing [calorific value of] —.* G. G. Crewson, Tottenville, N.Y., Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,245,688, Nov. 6, 1917. Date of appl., July 3, 1916.

To determine the calorific value of gas the latter is pumped from a container to a burner placed below a metal coil through which a current of water is passed. The supply of water is regulated and actuated by the gas pump, and the temperature of the water is read before and after it has passed through the coil.—W. P. S.

## Patent List.

The dates given in this list are in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Avery. Drying-apparatus. 22. Jan. 2.

Brand. Channel furnaces. 146. Jan. 2.

Breakell. Means for covering in and discharging grinding, crushing, and mixing mills. 227. Jan. 4.



British Carbonizing Co., and Garbutt. Rotary furnaces. 246. Jan. 4.

Bynoe. Drying and/or heating air. 462. Jan. 8.

Chemical Engineering and Wilton's Patent Furnace Co., Hinds, and Stinchcombe. Furnaces. 530. Jan. 9.

Faïencerie de Gien. 443. *See under VII.*

Forge. Continuous emulsifying and mixing machine. 18. Jan. 2.

Geurten. Heating or drying plant. 446. Jan. 8. (Ger., Aug. 21, 1917.)

Halkes. 702. *See under IX.*

Hilton and Willis. Apparatus for bringing gases and liquids into intimate contact. 218. Jan. 4.

Loewenstein. Drying-apparatus. 51. Jan. 2.

Shearman. Grinding-mill. 21. Jan. 2.

Workman. Sublimating-apparatus for purifying chemical substances. 149, 150, 151. Jan. 2.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,303 (1916). Mond (Bradley and Schmidt). Method and apparatus for separating gases. (112,153.) Jan. 9.

18,394 (1916). Boby. Cloth filters. (112,159.) Jan. 9.

2160 (1917). Chammon. Syphons for corrosive and other liquids. (112,188.) Jan. 9.

5972 (1917). Andrews. Distilling-apparatus. (112,223.) Jan. 9.

6372 (1917). Lotozky. Apparatus for mechanically separating materials. (112,227.) Jan. 9.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Colona. Generation of gases for use in internal-combustion engines. 675. Jan. 11.

Kilburn (Norsk Aktieselskab for Elektrokemisk Industri). 551. *See under XI.*

Lundén and Thorsell. 549. *See under VII.*

McLellan, Merz, and Michie. Use of solid fuel for large-scale power production. 717. Jan. 11.

Sivyer. Manufacture of coke. 531. Jan. 9. (Australia, Feb. 2, 1917.)

Tully. Carbonisation of coal, etc., for production of gas, tar oils, etc. 694. Jan. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

836 (1917). Baker. *See under VII.*

2233 and 10,517 (1917). Manufacture of composition fuel. (112,341.) Jan. 16.

6008 (1917). Yates and others. *See under VIII.*

## III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Tully. 694. *See under II.*

Westwood. Distillation of tar, etc. 47. Jan. 2.

#### COMPLETE SPECIFICATION ACCEPTED.

6314 (1917). Major. Cooling, solidification, and delivery of pitch. (112,370.) Jan. 16.

## IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Marsh. Production of aniline dyes. 400. Jan. 8.

Newton (Bayer und Co.). Manufacture of azo dyestuffs. 84. Jan. 2.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Bailey and Robertshaw. Machine for preparing and treating wool, etc. 334. Jan. 7.

Blyth. Paper-making machines. 298. Jan. 5.

Heys and Nuttall. Beating-machines for making pulp for paper manufacture. 653. Jan. 11.

Miln. Coated paper. 250. Jan. 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1081 (1915). Coppier. Treating safety or fiduciary papers for the purpose of preventing fraud. Jan. 9.

181 (1917). Oman. Preparation of oxylinegne from ligninesulphonates. (103,649.) Jan. 16.

182 (1917). Oman. Preparation of oxylinegne from lignonsulphonates. (103,650.) Jan. 16.

202 (1917). Oman. Preparation of nitro compounds from ligninesulphonic acid or its salts. (103,651.) Jan. 16.

203 (1917). Oman. Preparation of nitro compounds from lignonsulphonic acid or its salts. (103,652.) Jan. 16.

206 (1917). Oman. Treatment of ligninesulphonates prepared from sulphite-cellulose waste lye so that they may be dried without decomposition. (103,655.) Jan. 16.

4170 (1917). Bell and Perry. Preparation of sisal fibres for spinning. (112,207.) Jan. 9.

8856 (1917). Schmid. Process for ungumming silk and silk waste. (107,966.) Jan. 9.

11,537 (1917). Bloxam (Zellstoff-fabrik Waldhof). Purifying exhausted sulphite-cellulose liquors. (112,395.) Jan. 16.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Cole and Kershaw. Machines for dyeing, mordanting, bleaching, scouring, etc., wool, yarn, etc. 279. Jan. 5.

Dudley. Dyeing-machine. 681. Jan. 11. (U.S., Jan. 11, 1917.)

Glaister and Hoyle. Dyeing textile fabrics. 566. Jan. 10.

Haworth and Livesey. Machines for printing yarn. 499. Jan. 9.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Adams, Bailey, Denny, and Norris. Production of ammonium sulphate. 310. Jan. 5.

Bailey. 13. *See under X.*

C.I. Syndicate, Jenkins, and Parsons. Manufacture of ammonia, etc. 703. Jan. 11.

Cocking, and Kynoch, Ltd. Processes for concentrating nitric acid. 117. Jan. 2.

Falengerie de Gien, anc. Geoffroy et Cie. Coils etc., for cooling acids, etc. 443. Jan. 8. (Fr., Oct. 6, 1917).

Hulin. Preparation of anhydrous metallic chlorides. 467. Jan. 8. (Fr., Jan. 13, 1917.)

Lundén and Thorsell. Manufacture of hydrogen. 549. Jan. 9.

Maxted. Synthesis of ammonia. 660. Jan. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

836 (1917). Baker. Recovery of ammonium chloride from ammoniacal liquor, with extraction of cyanides and other products. (112,329.) Jan. 16.

1060 (1917). British Cyanides Co., Chance, Rossiter, and North Lincolnshire Iron Co. Production of potassium chloride. (112,338.) Jan. 16.

8550 (1917). Welsbach Co. Recovery of thorium as pyrophosphate. (112,380.) Jan. 16.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Bailey and Hartley. Continuous kilns for firing pottery, etc. 329. Jan. 7.

Falengerie de Gien. 443. *See under VII.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

969 and 11,798 (1917). Method for prolonging the life of acid-proof pots or vessels made of stoneware, enamelled fireclay, earthenware, or the like. (112,330.) Jan. 16.

6008 (1917). Yates, Brayshaw, and Brayshaw. Gas-fired furnaces applicable for pottery and other purposes. (112,365.) Jan. 16.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Ardagh. Fire-resisting plastic material and its combination with and application to other materials for constructional purposes. 216. Jan. 4.

Chance and Hunt, Holley, and Webb. Acid-resisting cement. 749. Jan. 12.

Halkes. Kilns and chambers for drying timber. 702. Jan. 11.

Plant. Damp-proofing walls, etc. 217. Jan. 4.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Aktiebolaget Svenska Kullagerfabriken. Hardening-furnaces. 303. Jan. 5. (Sweden, Dec. 11, 1916.)

Aktiebolaget Svenska Kullagerfabriken. Means for removing hardened annular bodies from hardening-liquid. 301. Jan. 5. (Sweden, Dec. 16, 1916.)

Bagley. Removing molten slag from open-hearth steel furnaces by air or steam blast. 747. Jan. 12.

Bailey. Recovery of tin or its compounds from tin ores or residues. 13. Jan. 2.

Bullen. Concentration of ores. 688. Jan. 11.

Donald. Open-hearth, etc., metallurgical furnaces, etc. 27. Jan. 2.

Jackson, Lancaster, Rogers, and Walter. Treatment of tinned scrap. 577. Jan. 10.

Leech. Rust and corrosion prevention fluid, and process of applying it to metal. 327. Jan. 7.

Merton and Rowley. Roasting-furnaces. 588. Jan. 10.

Meurer. Production of metallic coatings. 430. Jan. 8.

Palmer. Production of basic steel. 162. Jan. 3.

Pitlivil. Welding different metals to form a composite ingot. 526. Jan. 9.

Rand and Vautin. Aluminium alloy. 369. Jan. 7.

Reynard. Method of treating iron and steel to prevent rusting. 572. Jan. 10.

Richmond Gas Stove and Meter Co., and Thompson. Steel furnaces. 276. Jan. 5.

Soc. Franç. d'Exploit. de Fours Spéciaux à Haute Température. Furnaces for melting steel, iron, etc. 388. Jan. 7. (Fr., Aug. 28, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

297 (1917). Bowles and Wilson. Protective coating of steel or iron sheets. (112,319.) Jan. 16.

1249 and 10,665 (1917). MacKinlay. Puddling-furnaces. (112,179.) Jan. 9.

1633 (1917). Gillies and Gillies. Treatment of zinciferous ores. (112,336.) Jan. 16.

3254 (1917). Riddle. Removing solid-metal bottoms of blast furnaces or steel furnaces. (112,199.) Jan. 9.

3486 (1917). Quasi-Arc Co., and Cole. Electric welding or fusion-deposition of metals. (112,353.) Jan. 16.

5550 (1917). Whiteinch Galvanizing Co., and Ordidge. Hot-process galvanising iron and steel plates, sheets, etc. (112,220.) Jan. 9.

5781 (1917). Morin. *See under XXIII.*

7644 (1917). Jones. Metal alloys. (112,377.) Jan. 16.

8550 (1917). Welsbach Co. *See under VII.*

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Kayser and Robinson. Electrode holders for electric furnaces. 729. Jan. 12.

Kilburn (Norsk Aktieselskab for Elektrokemisk Industri). Manufacture of carbon electrodes. 551. Jan. 9.

Western Electric Co. (Western Electric Co.). Electron-emitting cathodes. 411. Jan. 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,483 (1916). White (United States Light and Heat Corporation) Storage battery plates. (112,133.) Jan. 9.

18,611 (1916). Chkliar. Electric accumulators. (102,976.) Jan. 16.

1146 (1917). Filmer and Denton. Electric furnaces. (112,177.) Jan. 9.

3486 (1917). Quasi-Arc Co., and Cole. *See under X.*

10,072 (1917). Electrolytic Zinc Co. Electrolytic anodes. (108,311.) Jan. 16.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Allsebrook, and Wilkins and Co. 505. *See under XIII.*

Blogg. Oxygenation and polymerisation of oils. 44. Jan. 2.



Cannon and Reeves. Treatment of oil nuts. 259. Jan. 4.

Jenny and Zollinger. 483. *See under XX.*

Malone and Malone. Preparation of wax polish. 321. Jan. 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,621 (1916). Woltman. Means for adding hydrogen to non-saturated fatty acids and their glycerides. (112,293.) Jan. 16.

5664 (1917). Smethurst. Oil extracting, cooling, and like apparatus for use with compressed air. (112,362.) Jan. 16.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Allsebrook, and Wilkins and Co. Manufacture of linseed oil substitutes for use in manufacture of paints, linoleum, etc. 505. Jan. 9.

Blogg. 44. *See under XII.*

Rogers, Welch and Co. Dope-resisting solution or paint for aeroplanes, etc. 631. Jan. 10.

Wade (Redmanol Chemical Products Co.). Phenolic condensation product varnishes, etc. 604. Jan. 10.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

Jackson (India-Rubber Co.). Processes for producing pigmented rubbers. 147. Jan. 2.

#### COMPLETE SPECIFICATION ACCEPTED.

192 (1917). Burke. Method and means for vulcanising rubber. (112,312.) Jan. 16.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATION.

Heyl. Process for drying japanned or patent leather. 148. Jan. 2. (Ger., Nov. 14, 1916.)

### XVIII.—FERMENTATION INDUSTRIES.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,559 (1916). Crawford. *See under XX.*

7324 (1917). Hughes. Manufacture of alcohol from molasses. (112,232.) Jan. 9.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Dunlap and Stevens. Process for canning rice and milk. 380. Jan. 7. (U.S., May 2, 1917.)

Hamilton and Quirk. Sterilising cereals, seeds, nuts, etc. 585. Jan. 10.

Lear. Butter substitute. 9. Jan. 2.

MacKenzie. Production of flour or farina from potatoes. 598. Jan. 10.

Noar. Butter substitute. 633. Jan. 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,538 (1916). Rushen (Quarzlampen Ges.). Method of sterilising water. (112,149.) Jan. 9.

18,500 (1916). De Bruyn, Ltd., and de Bruyn. Utilising certain nuts or seeds in the manufacture of artificial butter or the like. (112,164.) Jan. 9.

4997 (1917). Place and Sons, and Place. Preservation of eggs. (112,215.) Jan. 9.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Albertsen. Process for treating tobacco. 138. Jan. 2.

Jenny and Zollinger. Production of polyfatty acid esters. 483. Jan. 8. (Switz., Jan. 8, 1917.)

Jones (Chem. Fabr. J. A. Wülfing). Preparation of a double compound of caffeine with alkali salts of acetylsalicylic acid. 484. Jan. 8.

Myddleton. Manufacture of *p*-oxyphenylarsinic acid. 573. Jan. 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,559 (1916). Crawford. Purification of crude alcohols. (112,166.) Jan. 9.

18,621 (1916). Woltman. *See under XII.*

### XXI.—EXPLOSIVES; MATCHES.

#### APPLICATIONS.

Lucas and Martin. Methods for testing explosives, etc. 195. Jan. 3.

Match Machine Corporation. Match-making machine. 41. Jan. 2. (U.S., Dec. 20, 1916.)

Marshall. Lucifer matches. 607. Jan. 10.

### XXIII.—ANALYSIS.

#### APPLICATION.

Crnyt, and Heenan and Froude. Apparatus for detection of moisture. 1. Jan. 2.

#### COMPLETE SPECIFICATION ACCEPTED.

5781 (1917). Morin. Testing or determining the hardness of metals and other substances. (108,460.) Jan. 9.

**I.—GENERAL; PLANT; MACHINERY.**

*Gas-fired boilers.* T. M. Hunter. Inst. Elect. Engineers. [Advance proof.] 18 pages.

THE author referred to the difficulties that had been experienced in firing boilers economically with gas, due chiefly to unsatisfactory arrangements for burning the gas. The three chief classes of gas used are:—coke-oven gas, blast-furnace gas, and producer gas. Coke-oven gas has a high calorific value and is fairly clean, but blast-furnace gas carries a great deal of dust and should be passed through a gas-cleaning plant before use. From the point of view of efficiency it is more important that a gas should be dry than free from dust, especially if used with water-tube boilers. The presence of water in the gas causes: (1) a lower efficiency, (2) less evaporation, and (3) a considerable loss due to lowering of flame temperature. As with coal-fired boilers, it is important to have the boilers covered and the settings air tight, as the introduction of "false air" causes most serious losses. Painting the settings with several thick coats of tar is advocated, and either induced draught or automatic dampers are advised when using gas. In addition to draught control it is important to have the gas at a constant pressure or to use a type of burner that is not affected by the gas pressure. To obtain the best results the gas and the water should be measured, the waste gases should be analysed, and the temperature in the flues should be recorded. The author gives a number of useful tables in an appendix.—W. H. C.

*Standardisation of chemical stoneware.* (1) Nielsen and Garrow. (2) Griffiths. See VIII.

*Action of caustic soda on steel plates.* Stromeier. See X.

**PATENTS.**

*Ether and alcohol vapour contained in the air of factories; Process and apparatus for recovering the —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 101,723, Oct. 6, 1916. (Appl. No. 14,235 of 1916.) Under Int. Conv., Oct. 7, 1915.

THE air containing the ether and alcohol vapours is passed up a plate column of acid-proof material down which relatively dilute sulphuric acid flows. The vapours are absorbed by the acid which is then, without further dilution, passed down a column still, and the ether, alcohol, and part of the water are distilled off, cooled, and condensed in suitable vessels. The acid is returned to the absorbing column for use over again. By regulating the temperature of distillation, the dilution of the residual acid may be varied so that any desired proportion between the alcohol and ether absorbed may be maintained.—W. H. C.

*Ether and alcohol vapours contained in the air of factories; Apparatus for recovering —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 101,875, Oct. 12, 1916. (Appl. No. 14,531 of 1916.) Under Int. Conv., Oct. 16, 1915. Addition to Eng. Pat. 101,723 (see preceding abstract).

TO overcome the difficulties caused by the variation in the amount of ether and alcohol vapours which occurs in the air to be treated, a valve, operated by a densimeter or float placed in an extension of the air main and which rises or falls as the percentage of vapours passing to the recovery apparatus varies, is placed in the inlet main to the recovery apparatus described in the principal patent.—W. H. C.

*Waterproofing and rendering impervious to dust bags and sacks; Method of —.* J. Gadsen, Melbourne, Australia. Eng. Pat. 109,252, July 25, 1917. (Appl. No. 10,676 of 1917.) Under Int. Conv., Aug. 28, 1916.

BEFORE filling, the interior of the bag is sprayed or coated with a suitable adhesive fluid and the bag is at once filled. A thin layer of the material packed in the bag adheres to the fluid and forms an impervious layer which protects the rest of the contents.—W. H. C.

*Gas filtering apparatus.* The New Jersey Zinc Co., New York, Assignees of G. S. Brooks and L. G. Duncan, De Pue, Ill., U.S.A. Eng. Pat. 110,907, Mar. 9, 1917. (Appl. No. 11,048 of 1917.) Under Int. Conv., Nov. 1, 1916. (See also U.S. Pat. 1,240,305 of 1917; this J., 1917, 1171.)

THE dust-laden gases pass through a flue into a bag chamber, the gases being driven forward by a fan placed in the flue. A by-pass is provided to connect the outlet of the bags with the inlet of the fan, and valves controlled by rods are provided on the inlet flue for the gases and on the by-pass flue. The valves are connected with bag-shaking mechanism, which is operated intermittently by electrical means, the arrangement being such that when the bag-shaking device is operated, the bag outlets are put into connection with the fan inlet and the supply of dust-laden gas to the bag is shut off, so that whilst the bag is being shaken a current of air is drawn through and clears the interstices of the filter from solid particles.—W. H. C.

*Cleansing or washing smoke, air and other gases, and extracting soot, dust, and other impurities therefrom; Apparatus for — or for evaporating liquids.* A. Hindmarsh, Newcastle-on-Tyne. Eng. Pat. 111,637, May 21, 1917. (Appl. No. 7339 of 1917.)

THE air or gas is passed through a horizontal cylinder within which a perforated drum is rotated upon a hollow shaft provided with blades. Water or other suitable liquid is fed into the hollow shaft and also independently into the outer cylinder. The perforated drum is provided with external fan blades which dip into the liquid in the bottom of the cylinder as they rotate.—W. H. C.

*Heating and cooling apparatus and the like.* S. S. Guy, Wolverhampton. Eng. Pat. 111,556, Dec. 13, 1916. (Appl. No. 17,879 of 1916.)

IN cooling or heating apparatus consisting of several cylinders enclosed in a common jacket through which the heating or cooling medium is passed, hollow partitions of wedge-shape or V-shape in cross section, made of thin metal, are interposed between the cylinders. In this way the surging of the liquid cooling or heating medium in the jacket is prevented, and the liquid is brought into more intimate contact with the walls of the cylinders.—W. H. C.

*Separating substances of different specific gravities, such as fine coal or ores and the like; Apparatus for —.* J. M. Draper, Bridgend, and The Rhondda Engineering and Mining Co., Ltd., Cardiff. Eng. Pat. 111,826, Oct. 5, 1917. (Appl. No. 14,404 of 1911.)

THE material is fed into a conical perforated funnel placed in the upper part of a vertical vessel up which a current of water flows, and carries the finer particles to the overflow. A slowly-rotating valve is provided at the lower end for the discharge of the heavier particles. A balancing liquid column is provided, contained in a vertical pipe,



the lower part of which is attached to the separating chamber at a point between the overflow and bottom discharge. The upper part of the pipe contains air to form an air cushion and has a valve to allow of the escape of air during filling.—W. H. C.

*Drying apparatus.* H. G. C. Fairweather, London. From Pacific Evaporator Co., Portland, Oreg., U.S.A. Eng. Pat. 111,994, Sept. 5, 1917. (Appl. No. 12,735 of 1917.)

THE materials to be dried are supported on trays having perforated bottoms and placed in tiers on trucks so that the trays are slightly inclined to the horizontal; the trays on alternate rows of trucks are inclined in opposite directions. The trucks are moved on rails along the drying chamber across which heated air or other drying medium is passed. As the drying progresses the trucks with the trays are moved towards the air inlet end of the apparatus. To effect economy, primary and secondary drying chambers are used, the air passing first through the secondary chamber over the nearly dry material and then through the primary chamber over the fresh material.—W. H. C.

*Dryer.* W. D. Palen, Philadelphia, Assignor to The Philadelphia Textile Machinery Co. U.S. Pat. 1,248,389, Nov. 27, 1917. Date of appl., May 3, 1916.

A DRYING chamber is provided with two platforms, one above the other, the lower one extending beyond the upper. A flight conveyor travels on the platforms. A rotary distributor having blades located below and beyond the discharge end of the upper platform and clear of the conveyor is arranged so that material which accumulates in front of the flights of the conveyor on the upper platform will be discharged on to the distributor and will fall from this on to the lower platform.—J. H. P.

*Dryer and evaporator.* C. Offenhauser, Philadelphia, Pa. U.S. Pat. 1,249,177, Dec. 4, 1917. Date of appl., July 9, 1917.

THE material to be dried is passed through the annular space between two co-axial drums. The inner drum is stationary and is heated internally, and the outer drum is rotated. The outer drum is provided with scrapers on the inside which remove the material from the outer surface of the inner drum.—W. H. C.

*Centrifugal separator.* J. H. Blair, Assignor to R. H. Taylor, jun., Philadelphia, Pa. U.S. Pat. 1,246,035, Nov. 13, 1917. Date of appl., Sept. 21, 1915.

A ROTARY dryer comprises a sectional tub having its parts mounted to separate outward and a confining member, movable axially with respect to the tub, to hold the parts in assembled position.—J. H. P.

*Centrifugal machine.* E. R. Curtis, Brighton. U.S. Pat. 1,248,821, Dec. 4, 1917. Date of appl., June 4, 1917.

A CYLINDRICAL vessel is provided with an axial inlet in the cover for liquid to be treated, and an axial outlet in the bottom which has a deep upward indentation. A hood is fitted over the indentation and radial ribs are provided in the enclosed space to provide passages for the liquid. A series of concentric corrugated cylindrical surfaces are provided near the cylindrical wall of the casing, supported alternately from the cover and the bottom, and a number of sector-shaped troughs are arranged in the bottom of the vessel and maintained in position by projections on the hood. The liquid passes first into the central space of the vessel, over the trays, and then in a zig-zag path

over the corrugated surfaces to the space between the hood and the bottom, and thence to the central outlet in the bottom.—W. F. F.

*Vapour-condensing apparatus.* J. E. Phillips, Sherman, Tex. U.S. Pat. 1,246,154, Nov. 13, 1917. Date of appl., June 6, 1916.

A CONDENSER is connected with a preliminary cooling and mixing chamber having a series of annular baffles surrounding a central space and occupying a part of the height of the chamber. The vapour is supplied to the space above the baffles and a pipe supplies liquid which is mixed with the vapour in this space. The mixed liquid and vapour are conveyed from the space below the baffles to the condenser.—J. H. P.

*Filtering machine; Automatic, continuous centrifugal*—H. A. Herr, Philadelphia, Pa. U.S. Pat. 1,246,267, Nov. 13, 1917. Date of appl., Oct. 19, 1914.

THE shaft which carries the basket of the machine is so supported that the basket may be rotated eccentrically at a high speed. When the basket is full, the speed is checked and the eccentricity is arrested, and the basket is emptied while running at a low rate of speed.—W. H. C.

*Separator for pulverising-mills; Pneumatic*—E. J. Steckle, Dixon, Ill. U.S. Pat. 1,246,344, Nov. 13, 1917. Date of appl., Jan. 31, 1916.

THE pulverised material is delivered through a number of nozzles which project upward from the grinding chamber into a separating chamber above. The nozzles pass through the bottom of the separating chamber tangentially and the more finely powdered material is drawn off from the separating chamber by a current of air. The coarser particles fall to the bottom and are returned to the grinding chamber.—W. H. C.

*Pulverising and treating ores, clay, and other mineral substances; Process of and apparatus for*—A. B. Willoughby, Philadelphia, Pa. U.S. Pat. 1,246,514, Nov. 13, 1917. Date of appl., May 6, 1914.

THE material to be pulverised is delivered to a heated platform to be dried, and then passes downward through two shoots to a pair of nozzles directed towards one another. Superheated steam at high temperature and pressure is also supplied to the nozzles so that the two streams of material impinge against one another at high velocity. The pulverised material and steam are withdrawn through an exhaust flue into which fresh superheated steam at high pressure and temperature is also supplied to reheat the cooled steam and to separate the lighter particles of the pulverised material. The steam is passed into a settling chamber, where the pulverised material separates, and the steam is discharged while still dry. The heavier unpulverised material is returned to the top of the shoot. The drying platform is heated by waste gas from the boiler which supplies the steam.—W. F. F.

*Spraying liquids and treating gases; Process and apparatus for*—C. C. Thomas, Baltimore, Md. U.S. Pat. 1,246,355, Nov. 13, 1917. Date of appl., May 3, 1915.

THE apparatus comprises a spray head in the form of a vertical tube of resilient material having in its periphery a helical slot with abrupt discharge edges. This slot varies in inclination from one end to the other, whereby the liquid can be discharged in the form of a helical film varying in direction from one end to the other of the spray head. For treating gases the spray head is fitted inside a chamber provided with a gas inlet and outlet. The liquid is ejected into the current of gas in the

form of a thin helical film at an angle to the direction of the flow of gas, and the pressure of the liquid and width of the slot can be regulated to cause the film to be broken up and atomised in contact with the gas.—J. H. P.

*Solidified plastic articles; Apparatus for rapidly ageing* —. De N. W. B. Young, Narberth, Pa., Assignor to Soap Works Equipment Co. U.S. Pat. 1,246,515, Nov. 13, 1917. Date of appl., Jan. 10, 1916.

AN endless conveyor carrying a series of free swinging holders passes in a zig-zag path through a chamber containing hot air at constant temperature, and then through a cooling chamber. Solidified plastic articles are fed into the hot compartment in the path of the holders, and means are provided in the cooling chamber to tilt the holders and discharge the articles. The air in the hot compartment is heated by pipe coils between the adjacent runs of the conveyor.—W. F. F.

*Fire extinguisher compositions.* H. S. Mork, Brookline, Mass., Assignor to American La France Fire Engine Co., Elmira, N.Y. U.S. Pats. (A) 1,246,640, (B) 1,246,641, and (C) 1,246,612, Nov. 13, 1917. Dates of appl., Feb. 26, Mar. 27, and Mar. 27, 1917.

(A) THE composition consists of carbon tetrachloride, a component to lower the freezing point, e.g., chloroform, and an aromatic ester. A typical composition is 95 c.c. of carbon tetrachloride, 5 c.c. of chloroform, and "2.5 grms. of triphenyl phosphate and phenyl salicylate in equal parts." (B) A mixture of 92½% of carbon tetrachloride, 2½% of chloroform, and 5% of "safrol." (C) A mixture of 90% of carbon tetrachloride, 5% of chloroform, and 5% of pine oil.—J. H. P.

*Evaporator.* W. H. C. Smith, Philadelphia, Pa. U.S. Pat. 1,246,824, Nov. 13, 1917. Date of appl., Apr. 23, 1917.

A VERTICAL cylindrical casing has its base dished inwards, and a tubular heating drum with vertical tubes is provided in its lower portion so that the lower tube-plate is approximately parallel to the base of the casing and forms with it a circular chamber having a number of circumferential openings into the casing. A liquid-supply pipe enters this chamber and the liquid circulates through the tubes. Steam is supplied through a vertical pipe passing axially downward through the casing and terminating in a nozzle of smaller section in the centre of the heating drum and near the bottom of the casing. The connection between the steam pipe and the top of the drum is made flexible to permit relative movement.—W. F. F.

*Evaporating or concentrating process.* F. P. Bergh, Yonkers, N.Y., Assignor to Central Reduction Co. U.S. Pat. 1,249,001, Dec. 4, 1917. Date of appl., Jan. 10, 1912. Renewed Feb. 23, 1917.

THE liquid to be evaporated is introduced as a stratum of spray into the middle portion of the evaporating chamber, and a current of dehydrating air or gas is introduced into the chamber below the stratum of liquid. The gas passes upwards through the liquid and evaporates the latter, and the vapour-laden air or gas and the finely-divided solid pass upwards through a screen which separates the solids.—W. H. C.

*Gases; Apparatus for separating mechanical mixtures of* —. J. O. Carrey, Assignor to Carrey Process Co., St. Louis, Mo. U.S. Pat. 1,248,454, Dec. 4, 1917. Date of appl., Apr. 10, 1915.

THE mixed gases are admitted to a receiving chamber mounted in a vertical frame which supports also a vertical rotating shaft carrying a

separating drum connected with the receiving chamber. A number of outlet passages from the rotating drum are provided at different radii and exhausting devices are provided in the passages to withdraw the separated gases from the drum.

—W. F. F.

*Carboys, kegs, or the like; A two-wheeled, hand-propelled vehicle to carry two* —. British Dyes, Ltd., J. Turner, and W. Hindle, Huddersfield. Eng. Pat. 111,927, Dec. 27, 1916. (Appl. No. 18,443 of 1916.)

*Combined condenser and heat interchanger [for tar distillation].* Eng. Pat. 111,961. See III.

*Acid-collecting apparatus.* U.S. Pat. 1,247,280. See VII.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Spontaneous heating of* —. G. Knox. Gas J., 1918, 141, 110—111.

MICROSCOPICAL and chemical analysis shows that coals are composed chiefly of humus bodies (high oxygen content with much hygroscopic water), resinous bodies (medium oxygen content, melting at 300° C.), and carbon, with a varying percentage of hydrocarbon, moisture, and mineral matter. Coal has the power of absorbing gases, in some cases to the extent of three times its own volume; the absorbed gas is very active chemically, and the chemical action (oxidation) increases the temperature of the coal. Coal being an excellent non-conductor, the heat generated increases steadily and with increasing rapidity until the ignition point of the coal is reached. This point is generally stated as varying between 370° C. for cannel and 477° C. for Welsh steam coal, but it varies according to the state of division of the coal. Spontaneous heating is more common in coals of high volatile content and has been a source of trouble in shipping. The length of the journey has been the determining factor in a number of cases of spontaneous ignition, showing that a certain time limit is necessary to enable the oxidation to generate sufficient heat. Cases of heating in coal stored on the surface are common, and coking coals stored for long periods lose their coking properties. The author gives the following hints as to the prevention of heating. Wherever possible the coal should be sized before stacking, and each size put in a separate heap. "Through" coal or mixed small coals should never be stacked to a height of more than 13 feet. If it is necessary, on account of lack of space, to store to a greater height than this, the coal should be laid down in successive layers of not more than 3 to 4 feet thick. An iron or earthenware ventilation pipe, 3 to 4 ins. in diameter, should be inserted for every 300 sq. ft. of surface. A thermometer should be lowered occasionally to ascertain the temperature at various depths. Coal should never be stacked in contact with wood, moist vegetable soils, or coarse vegetation like bracken. Dirty coals should not be mixed with the better class varieties, and as far as possible all "rashings" or other friable bituminous shale bands should be removed before storing. Coking coals should not be stored in heaps of more than 4 ft. and only for short periods, as very slight heating renders them useless for coking purposes. Wet coal should not be mixed with dry coal, but should be dumped round the edges of the stack with free access to the air.

—J. E. C.



*Naphthalene; Use of crude — as fuel for internal combustion engines.* J. H. Willis. Gas J. 1918, 141, 65—67.

THE author describes a method of utilising crude naphthalene in an internal combustion engine of about 10 B.H.P. installed in a motor launch. In the vaporiser used (designed by the author and G. G. Wilson) the molten naphthalene does not pass through any pipes of small diameter or through a spray. This vaporiser is of the "surface" type and is self-contained, acting also as the fuel storage vessel. It consists of a rectangular exhaust jacketed vessel, built up of thin steel plate, all joints being oxy-acetylene welded. The apparatus is bolted to the top of an exhaust jacketed T-piece, the ordinary petrol carburettor being fitted to the bottom. The back pressure of the silencer is sufficient to force some of the exhaust gases round the vaporiser. The portion of the air which it is proposed to carburet approximately to saturation is drawn by the suction of the engine through the molten naphthalene, the secondary air being drawn through the petrol carburettor, the jet of which is closed. The naphthalene used in the experiments was crude, but of fairly good quality, extracted from coal-gas during the process of benzol and toluol recovery. The engine was run for five or ten minutes on petrol and when the naphthalene had reached a temperature of about 130° C. the petrol was turned off completely. It was found that satisfactory running could be obtained by passing one-thirteenth of the air through the carburettor. Little difficulty was experienced with the carburation even with temperatures varying between 130° and 160° C. The calorific value of naphthalene is 17,395 B.Th.U. per lb. as compared with 20,100 and 18,700 for paraffin and petrol respectively.—J. E. C.

*Gas firing boilers.* Hunter. See I.

*Analysis of crude benzols, etc.* Adam. See III.

*Laboratory methods for benzol recovery plant operation.* Sperr, jun. See III.

*The colloidal state of matter in its relation to the asphalt industry.* Richardson. See IX.

#### PATENTS.

*Briquettes or other moulded articles; Process of making —. Briquetting process.* Weather-resistant briquette. C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co., N.J. U.S. Pat. 1,246,805, 1,246,807, and 1,246,808, Nov. 13, 1917. Dates of appl., (A) Mar. 28, 1913, (B) May 2, 1917, (C) July 5, 1917.

(A) BRIQUETTES, or other moulded articles are made by mixing concentrated acid sulphite-cellulose waste liquor with a bulking material containing coal dust and coal fragments, and quick-lime is incorporated in order to precipitate the solids from the liquor. (B) Acid sulphite-cellulose waste liquor is atomised, and the finely divided solids which are obtained, are dissolved in water to form a binding agent. This is mixed with the bulking material, moulded under pressure, and the material is treated to render the dissolved solids insoluble, and then allowed to set to form a weather-resistant briquette. (C) Sulphite-cellulose waste liquor is incorporated with a bulking agent, and a basic compound of magnesium, such as the oxide, is used as precipitant, the mass being afterwards pressed into the required shape.—B. N.

*Fuel and process of burning the same.* C. F. Schrimpe, Perth Amboy, N.J., Assignor to The Perth Amboy Chemical Works, New York. U.S. Pat. 1,248,557, Dec. 4, 1917. Date of appl., Apr. 28, 1916.

A FUEL contains approximately 100 parts by

weight of hexamethylenetetramine, 20 parts of a substance yielding oxygen slowly to the hexamethylenetetramine, such as potassium permanganate, and 20 parts of solid paraffin.—J. E. C.

*Coke ovens; Regenerative —.* D. Bagley, London. Eng. Pat. 111,801, May 18, 1917. (Appl. No. 7138 of 1917.)

ONE regenerative chamber is placed above and one below a coke oven heated by vertical flues. The regenerators are in the same plane as, and extend the full length of, the coking chamber. They are used alternately according as the direction of the flame in the heating flues is upward or downward. In the first case hot air from the lower regenerator meets the gas at the base of the flues, and the products of combustion ascend through the vertical flues and passing through the upper regenerator give up their residual heat to the chequered brickwork. At certain periods this sequence is reversed, the upper regenerators supplying the hot air and the direction of the heating flames being downward. The upper and lower regions of the oven walls are thus more evenly heated.—J. E. C.

*Furnace and oven apparatus; Operating — with recovery of heat.* H. Koppers, Essen-on-the-Ruhr, Germany. Assignor to H. Koppers Co. U.S. Pat. 1,246,114, Nov. 13, 1917. Date of appl., July 27, 1914.

IN operating a regenerative furnace heated by producer gas, the reversal is effected by interrupting the air and steam supply to the producer to stop the gas production therein, cutting the furnace off from the producer, changing the position of the air and waste heat valves of the furnace, and re-connecting the furnace, the air, and the steam supply to the producer.—J. E. C.

*Coke-producing and by-product-recovery method.* M. Marshall, sen., Port Coquitlam, British Columbia. U.S. Pat. 1,248,883, Dec. 4, 1917. Date of appl., Aug. 14, 1917.

THE heat from the upper strata of a charge of coal is drawn downwards by an induced draught through the remainder of the charge. The volatile constituents are thus extracted, pass downwards in separate streams, and then through finely-divided material to the condensers.—J. E. C.

*Fuel[gas]; Manufacture, utilisation, and combustion of —.* G. Helps, Nuneaton. Eng. Pat. 111,495, Aug. 24, 1916. (Appl. No., 12,011 of 1916.)

A HYDROCARBON gas, e.g., coal gas, is mixed with pure blue water-gas, the mixture having a net calorific value of from 335 to 250 B.Th.U. per cubic foot, or other diluents (Mond gas, producer gas, air, or nitrogen) may be used, in which case the calorific value of the mixed gases may be from 400 to 250 B.Th.U. per cubic foot. These mixtures may be used at low pressures so as to give coneless flames in close contact with the object to be heated. The mixture of coal gas and diluting gas may be made during the process of manufacture as described in Eng. Pat. 14,750 of 1916, or the gases may be mixed subsequently.—J. E. C.

*Carburetted water-gas; Production of —.* G. E. Heyl, London. Eng. Pat. 111,934, Jan. 5, 1917. (Appl. No. 252 of 1917.)

SULPHUR-CONTAINING oils are vaporised and the vapours carried by a stream of water-gas to a superheating chamber in which the vapours are cracked. The mixed gases and vapours then pass through a purifier containing ferric oxide or other sulphur-removing substances.—J. E. C.

*Gas producers.* T. R. Wollaston, Manchester. Eng. Pat. 111,964, Mar. 19, 1917. (Appl. No. 3963 of 1917.)

WATER or tar or both may be admitted in a finely divided or atomised condition into the interior of a fixed hollow core or dome extending upwards from the grate of a gas producer, and the products are thereby vaporised and superheated prior to being introduced into the combustion zone.

—J. E. C.

*Coke-oven and gas works; Treatment of refuse from* —. S. N. Wellington, London, and S. B. Etherton, Barnsley. Eng. Pat. 111,883, Dec. 8, 1916. (Appl. No. 17,698 of 1916.)

REFUSE liquors, such as "devil" or waste liquors from coke-oven or gas works, are admitted, with or without the addition of lime, into gas producers, for total destruction of the noxious constituents.

—J. E. C.

*Liquid fuel suitable for use in internal combustion engines; Production of* —. J. M. O'Brien, Streatham. Eng. Pat. 111,864, Sept. 5, 1916. (Appl. No. 12,579 of 1916.)

ORDINARY commercial paraffin is agitated with strong sulphuric acid, bleaching powder, and petroleum ether. The filtered material may be mixed with petrol in various proportions, and if desired may be treated with silicic acid in an absorbent form.—J. E. C.

*Liquid hydrocarbons [; Production of light] —.* G. E. Heyl, London. Eng. Pat. 111,933, Jan. 5, 1917. (Appl. No. 251 of 1917.)

THE yield of lighter liquid hydrocarbons in fractionally distilling heavier hydrocarbons (mineral oils, coal oils, coal-tar oils, shale oils, and the like) is increased by impregnating the oil, before distillation, with a hydrocarbon gas or with hydrogen, without external heating. By preventing an increase in temperature during absorption, or, preferably, by reducing the temperature of the hydrocarbon, during absorption, below the prevailing temperature, the yield is still further increased.—L. A. C.

*Crude petroleum and the distillates thereof; Apparatus for treating* —. C. B. Forward, Urbana, Ohio. U.S. Pat. 1,217,808, Nov. 27, 1917. Date of appl., Sept. 15, 1915.

A SET of condensers, each with a separate outlet at the bottom, is arranged in series, the top of one condenser being connected with the bottom of the next. Superheated steam is blown into the bottom of "one of the middle of the series of condensers."—L. A. C.

*Heavier hydrocarbons; Method of treating* —. S. Schwartz, Assignor to Jenkins Petroleum Process Co., Milwaukee, Wis. U.S. Pat. 1,247,883, Nov. 27, 1917. Date of appl., May 10, 1917.

HEAVY hydrocarbons are heated under pressure in a horizontal cylindrical still and the vapours withdrawn through an artificially cooled collecting device situated within the still between the ends and at the level above the surface of the liquid most suitable for the complete removal of the condensable vapours.—L. A. C.

*Liquid fuel for internal-combustion engines.* J. P. Foster, Paia, Hawaii. U.S. Pat. 1,248,302, Nov. 27, 1917. Date of appl., Aug. 16, 1917.

A LIQUID fuel comprises a mixture of 2 galls. of methyl alcohol, 100 galls. of ethyl alcohol, 0.5 gall. of pyridine, and substantially 15 galls. of sulphuric ether.—J. E. C.

*Gasoline-renovator; Vacuum* —. *Process of purifying fluids [gasoline, etc.]* J. J. Tokheim, Cedar Rapids, Iowa. U.S. Pats. (A) 1,248,950 and (B) 1,248,951, Dec. 4, 1917. Date of appl., Mar. 30, 1916.

(A) THE liquid is fed from a supply tank to a distillation tank through a valve controlled by a float operated from the latter tank. Conduits lead from the distillation tank, into which a steam pipe enters, to a receiver located below it, and to a separator. The conduit to the latter passes through a condenser and has a cold water supply opening into it between the tank and the condenser. (B) A process for purifying gasoline and similar hydrocarbon cleaning liquids, consists in accumulating two bodies of the impure liquid, introducing steam into one body, condensing the vapour, and "effecting an interchange between the water of condensation and the other body of liquid."—L. A. C.

*Oil and gas; [from oil-bearing strata] Apparatus for and method of recovering* —. F. Squires, Marietta, Ohio, Assignor to W. Squires, Zanesville, Ohio. U.S. Pat. 1,249,232, Dec. 4, 1917. Date of appl., June 19, 1917.

A HEATED, deoxidised gas (products of combustion) is introduced under pressure through a pressure well into an oil-bearing stratum to mix with the hydrocarbon vapour. The mixture is collected from an adjacent venting well, the hydrocarbon (gasoline) recovered by compression and absorption, and the residual products of combustion used again.—L. A. C.

*[Petroleum] oils; Treating* —. C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,249,278, Dec. 4, 1917. Date of appl., Apr. 29, 1913.

THE vapours of an oil consisting essentially of petroleum products heavier than gasoline are subjected first to an increasing temperature at or above that of decomposition; then (in the presence of a catalyst) to a decreasing temperature until equilibrium is established. The products are condensed and the portion heavier than gasoline treated again.—L. A. C.

*Distillation of heavy oils, oil residues, and bitumens [to produce lighter oils].* W. A. Hall, New York. U.S. Pat. 1,247,671, Nov. 27, 1917. Date of appl., May 3, 1916.

SEE Eng. Pat. 8836 of 1915; this J., 1916, 919.

*Gas filtering apparatus.* Eng. Pat. 110,907. See I.

*Apparatus for separating substances of different specific gravities, such as fine coal or ores and the like.* Eng. Pat. 111,826. See I.

*Treatment of bituminous shales and the like for the obtainment of oil therefrom, and apparatus to be used therein.* Eng. Pat. 111,605. See IIB.

*Apparatus for distillation and dehydration of coal tar, mineral or vegetable oils and the like, and debenzolising of wash and other oils and liquids.* Eng. Pat. 111,956. See III.

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Cellulose and starch; Distillation of* — in vacuo. A. Pictet and J. Sarasin. Comptes rend., 1918, 166, 38—39.

PURE cotton cellulose was heated gradually in a distillation apparatus under a pressure of 12—15 mm. After the aqueous fractions there came



over between 200° and 300° C. a thick yellow oil which on cooling solidified to a semi-crystalline mass amounting to 45% of the original cellulose; 10% of carbon remained in the retort. The pasty mass, on recrystallisation from hot acetone or water, gave white tabular anhydrous crystals melting at 179.5° C. Analysis and molecular weight determinations indicated the formula,  $C_6H_{10}O_5$ . The substance was strongly laevo-rotatory and could not be distilled under atmospheric pressure without decomposition; it gave a triacetate, m. pt. 110° C. and a tribenzoate m. pt. 199.5° C. This substance appears to be identical with laevoglucosan obtained by Tanret (Bull. Soc. Chim., 1894, 11, 949) by the splitting of the glucosides picein, salicin, and coniferin. Exactly similar results were obtained by the distillation of starch and apparently also of dextrin. It is suggested, therefore, that laevoglucosan may stand in a direct relationship to the carbohydrates, possibly as a product of simple depolymerisation, and a study of its constitution and the mechanism of its formation may throw some light on the nature of the polymerised carbohydrates.—J. F. B.

#### PATENTS.

*Bituminous shales and the like; Treatment of — for the obtaining of oil therefrom, and apparatus to be used therein.* D. and A. Simpson, London. Eng. Pat. 111,605, Mar. 8, 1917. (Appl. No. 3449 of 1917.)

BITUMINOUS shale is subjected to downward distillation, at such a restricted temperature as to liberate the volatile products only, without breaking up the "combinations of so-called fixed carbon." The volatile products pass directly to a desulphurising chamber, whilst the spent shale may be treated in a separate generator for the production of fuel gas and ammonia.—J. E. C.

*Carbon [charcoal] and method of producing the same.* R. A. Demme, New York, U.S. Pat. 1,249,041, Dec. 4, 1917. Date of appl., Aug. 29, 1917.

HYDROLYSED cellulose material such as wood waste, freed from soluble constituents, is gradually heated to carbonising temperature. Suitable inorganic substances or other carbonisable organic substances may be mixed with the cellulose before heating.—B. V. S.

*Enclosed-arc device.* J. H. Clough, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,246,054, Nov. 13, 1917. Date of appl., Oct. 30, 1915.

A CATHODE of highly refractory material, e.g., tungsten, proportioned to operate at incandescence, and an anode of a chemically active metal, such as calcium, magnesium, titanium or the like, are contained in a sealed envelope filled with argon at a pressure of 5–12 cm. of mercury.—L. A. C.

*Retort [for destructive distillation of coal].* H. P. Bostaph, and Bostaph Engineering Co., Detroit, Mich., U.S.A. Eng. Pat. 111,871, Oct. 30, 1916. (Appl. No. 15,477 of 1916.)

SEE U.S. Pat. 1,204,646 of 1916; this J., 1916, 1253.

### III.—TAR AND TAR PRODUCTS.

*Benzol recovery plant operation; Laboratory methods for —.* F. W. Sperr, jun. Met. and Chem. Eng., 1917, 17, 642–647. (See this J., 1918, 2 A. 26 A.)

THE specific gravity of crude benzol and other light oils is determined with the Westphal balance at 15.5° C., after previously cooling the sample to

12° C. For the sulphuric acid colour test on benzols, toluols, and naphthas, the standard method given in the American Gas Chemists' Handbook is used, the colour being compared with a scale of coloured solutions prepared from cobalt and ferric chlorides and potassium bichromate respectively. The presence of sulphur dioxide and trioxide in washed benzols is detected by exposing methyl orange test paper to the vapours of the benzol issuing from the mouth of the containing bottle. Unsaturated compounds are determined by the method of Kraemer and Spilker (Muspatt's Encyclopædia, Ger. Ed., 1905, Vol. 8, p. 33), viz., titration with a bromide-bromate of potassium solution in presence of sulphuric acid until the red colour of the bromine persists in the oil. The purity of benzene may be controlled by a determination of freezing point. Carbon bisulphide is estimated by the xanthate method. Hydrogen sulphide may be estimated by washing the sample with sodium hydroxide solution and titrating the extract with iodine after acidification. A specification and tests for wash oil—a "straw" petroleum oil—are given. Benzolised and debenzolised oils are tested by a preliminary distillation in steam, followed by a fractionation of the dried distillate up to 200° C. Naphthalene in oils is estimated by a method based on its volatilisation in a current of warm air. Air at the rate of 1½ cub. ft. per hour is blown through the oil contained in an 8 in. × 1 in. test-tube placed in a bath of water which is heated slowly until boiling and kept at that temperature for 1 hour. The heat is withdrawn and the air bubbled through overnight. The test-tube is fitted with a reflux condenser to condense and retain oils, while the vapours pass on to a train of wash-bottles. The first contains 200 c.c. of N/1 sulphuric acid to retain tar and alkali; then follows an empty bottle to serve as trap and afterwards three bottles each containing 150 c.c. of saturated picric acid solution. Naphthalene picrate is formed and estimated by volumetric determination of the total and free picric acid present.—H. J. H.

*Benzols, etc.; Analysis of crude —.* W. G. Adam. Gas J., 1918, 141, 65.

THE author gives details of the method used by the Gas Light and Coke Co., for the evaluation of crude benzol. A preliminary distillation is carried out with 500 c.c. of the sample in a copper vessel, tapering from 6 ins. to 4½ ins. in diameter and 6 ins. high in the body, and fitted with an eight-section Young-Thomas column. The distillate should distil over fairly rapidly, but not in a continuous stream, and is collected up to 165° C., the residue being heavy oil or cresote. In the case of crude naphtha or light oils, the tar acids may be removed by washing with caustic soda. The distillate is then washed as follows:—Carbon bisulphide is removed by agitation with 30 c.c. of 10% alcoholic potash, the potassium xanthate being removed by three water-washes of 100 c.c., 50 c.c., and 30 c.c. Unsaturated compounds, bases, etc., are removed by washing with 20 c.c. of sulphuric acid (C.O.N.) per 250 c.c. of distillate for 5 minutes, the acid being run off after standing for half an hour. Phenolic substances are removed by agitation with 20 c.c. of 10% caustic soda solution per 250 c.c. of distillate. The soda layer is run off after standing for half an hour and excess soda washed out by gentle shaking with water. The oil is dried by standing over calcium chloride overnight. The dried and purified oil is fractionated in the same vessel and column, the following fractions being collected: (a) start to 85° C., (b) 85° to 105°, (c) 105° to 115°, (d) 115° to 165° C. The respective volumes are indicated by:—Benzene =  $a + \frac{1}{2}b$ ; toluene =  $\frac{1}{2}b + c$ ; solvent naphtha =  $d$ . The specific gravities of a, b, and c should be at least 0.883, 0.878, and 0.868 respec-

tively at 60° F. (15.5° C.), low specific gravities denoting the presence of paraffins, which may be approximately determined by the extent of the reduction.—J. E. C.

*Phenol; Determination of*—*in crude carbolie acid and coal-tar oils.* F. W. Skirrow. J. Ind. Eng. Chem., 1917, 9, 1102—1106.

The method is based upon the oxygen absorption values of phenol and the cresols (see this J., 1908, 58). To obtain standard values the solutions of phenol, etc., were diluted so as to contain 0.002 gm. in 50 c.c. Portions of 50 c.c. of potassium permanganate solution (0.395 gm. per litre) were treated with 15 c.c. of dilute (1:3) sulphuric acid (faintly tinged with permanganate), and the bottles closed and placed in a thermostat at 23° C. The solutions of tar acids, etc., were also brought to 23° C., and 50 c.c. run from a pipette heated to 23° C. into the permanganate solution. The time was taken from the fall of the first drop, and the bottles were rapidly shaken, and allowed to stand for 3 mins., whereupon one c.c. of 10% potassium iodide solution was introduced, the liberated iodine titrated with thiosulphate solution (7 grms.

allowed to stand for 5 mins. It was then cooled under the tap, the mixture slightly acidified with sulphuric acid, the excess of iodine removed by means of a slight excess of thiosulphate, and the precipitated iodine compound washed once by decantation, and transferred to a weighing bottle of clear glass for comparison. The method was applied to the distillates obtained by three successive fractionations of crude carbolie acid, and the results are tabulated. Other samples of crude carbolie and refined cresylic acids were freed from hydrogen sulphide, fractionated twice, and the fractions examined as described. It was found that the mean oxygen absorption of the cresols, etc., in these fractions, calculated from the oxygen absorption of the fraction, the oxygen absorption of pure phenol, and the percentage of phenol found in the fraction, was very constant in each fraction, and that the variations in the oxygen absorption were mainly due to the variations in the amount of phenol. For example, the mean oxygen absorptions of the cresols, etc. (constituents other than phenol), in the fractions from the second fractionation of a litre of the various samples were as follows:—

No.	Temp., ° C.	Crude carbolie acid.	Refined cresylic acid.	Carbolie acid, "60's."	Refined cresylic acid.	Crude carbolie acid.	Mean.
1	180—184	1.117	1.120	1.118	(1.110)	1.114	1.117
2	184—187	1.114	1.119	1.113	1.113	1.115	1.115
3	187—190	1.096	1.099	1.102	1.097	1.099	1.099
4	190—193	1.083	1.081	1.077	1.088	1.082	1.082
5	193—196	1.076	1.078	1.076	1.084	1.076	1.078
6	196—199	1.073	1.078	1.070	1.069	1.062	1.070

per litre), and the results calculated to grms. of oxygen absorbed by 1 gm. of the respective substances. The following mean values were thus obtained:—Phenol, 1.434; *o*-cresol, 1.170; *m*-cresol, 1.156; and *p*-cresol, 1.062 gm. Test experiments with solutions containing from 11 to 80% of phenol and mixtures in equal proportions of the three cresols gave slightly increased oxidation values, but not sufficient to invalidate the method. The differences in the rates of oxidation of the three cresols is, however, too great to allow of the application of the method unless the ratio in which they are present is known. It is therefore necessary to fractionate the crude carbolie acid by distillation, and to determine approximately the amounts of the three cresols in the fractions in which the phenol is concentrated. The "pear" still-head, as used by Weiss and Downs (this J., 1917, 863), gave the best results for the fractionation, and there was no advantage in making more than two fractionations. For estimating the relative proportions of phenol and cresols in the several fractions an adaptation of Messinger and Vortmann's method of determining phenol as triiodophenol was used. The final iodine products have the following colorations:—Phenol, rose-pink; *o*-cresol, brown; *m*-cresol, slate-blue; and *p*-cresol, dirty yellow. By determining the oxygen absorption value of a mixture of phenol and cresols, and making trial mixtures of the same oxygen absorption value, it was possible eventually to match the shade given by the iodine compound of the unknown mixture. In the case of the higher fractions, better colour comparisons can be made by using mixtures with slightly higher oxygen absorption than the fractions to be matched, this being probably due to the increasing amounts of substances of higher b. pt. in those fractions. For the preparation of the iodo-compound, 25 c.c. of a solution of 1 gm. of the mixture of phenol and cresols in 500 c.c. of water was added to 3.5 c.c. of *N*/1 sodium hydroxide diluted with 200 c.c. of water, the mixture heated to 70° C. in a stoppered flask, 25 c.c. of 0.02 *N* iodine solution added, and the flask shaken and

Hence the percentage of phenol in each fraction may be calculated from the oxygen absorption of pure phenol and the mean oxygen absorption of the cresol, etc., in that fraction, as given in the table, without the tedious matching operations described above. Small errors are caused by the presence of pyridine, and by water retained by the dissolved sodium phenolate. These errors are corrected by adding the following amounts to the percentages of phenol:—9.62%, 0.38; 23.98%, 1.02; 47.50%, 2.50; and 70.54%, 4.46%. With this correction the results thus obtained agree within 0.2 to 0.3% of the true amounts of phenol. The method may also be used for the determination of phenol in the tar acids from inclined retorts, vertical retorts, blast furnace creosote, and producer gas tar.—C. A. M.

*Use of crude naphthalene as fuel for internal combustion engines.* Willis. See 11A.

#### PATENTS.

*Distillation and dehydration of coal tar, mineral or vegetable oils and the like and debenzolising of wash and other oils and liquids; Apparatus for the —.* T. C. Hammond and C. K. Richardson, Huddersfield, Yorks. Eng. Pat. 111,956. Mar. 5, 1917. (Appl. No. 3196 of 1917.)

In the upper part of a still is a preheater comprising a series of overlapping baffle-plates arranged in step fashion. The crude tar or oil, fed on to the uppermost plate, flows down the series. The lighter portions (light oils, water, etc.) are vaporised and removed through an outlet pipe. From the lowest step, the tar or oil passes into the still proper, whence it is drawn off through a suitable opening. Vapours given off from the still pass between the baffle-plates of the preheater, which thus serves also as a fractionating column, the heavier portions, which may be produced by local overheating, dropping back into the still.—L. A. C.



*Condenser and heat interchanger [for tar distillation]; Combined*——. C. K. Richardson and T. C. Hammond, Huddersfield. Eng. Pat. 111,961, Mar. 14, 1917. (Appl. No. 3689 of 1917.)

THE apparatus consists of a vertical tubular condenser, the tube chamber being surrounded by an annular jacket. The apparatus is specially applicable to dehydrating or distilling tar, and when so used the vapours from the still are passed down the tubes and the crude cold tar is passed upwards around the tubes. The hot dehydrated tar or other hot liquid is passed downwards through the annular jacket and serves to assist in heating the crude tar.—W. H. C.

*Naphthalene; Sublimation of*——. R. C. Taylor, Manchester. Eng. Pat. 111,774. Mar. 2, 1917. (Appl. No. 3104 of 1917.)

CRUDE naphthalene is heated in a stationary tank provided with a mechanical stirrer, above which are a number of condensing chambers. A rotatable pipe with a cut-away portion serves as a valve to connect the tank with each of the condensing chambers in turn, so that sublimate of different qualities can be collected.—F. W. A.

*Chlorine derivatives of toluol [benzyl and benzal chlorides and benzotrichloride]; Manufacturing side-chain*——. H. D. Gibbs, San Francisco, Cal., and G. A. Geiger, Washington, D.C. U.S. Pat. 1,246,739, Nov. 13, 1917. Date of appl., Mar. 20, 1917. (Dedicated to the public.)

GASEOUS toluol and chlorine in theoretical proportions are introduced at a measured velocity into a suitable reaction chamber in which they are subjected to the action of ultra-violet rays. One mol. equiv. of toluol with one, two, and three mol. equivs. of chlorine produce respectively benzyl chloride, benzal chloride, and benzotrichloride.—L. A. C.

*Sulphonation of aromatic hydrocarbons or their derivatives; Process for the*——. H. Bull, Philadelphia, Pa. U.S. Pat. 1,247,499. Nov. 20, 1917. Date of appl., Sept. 2, 1916.

AROMATIC hydrocarbons or their derivatives are sulphonated in the presence of an organic solvent, of lower specific gravity than sulphuric acid, in which the substance and sulphonated product are soluble but the sulphuric acid is not appreciably soluble, and the solution of the sulphonated product is removed. The solvent may consist of an excess of the aromatic hydrocarbon, and the process may be made continuous by continuously removing the extracted sulphonated product. —F. W. A.

*Phenol; Production of*——. A. M. Aylsworth, and Savings Investment and Trust Co., East Orange, N.J., U.S.A. Eng. Pat. 103,664, Jan. 17, 1917. (Appl. No. 883 of 1917.) Under Int. Conv., Jan. 24, 1916.

SEE U.S. Pat. 1,213,142 of 1917; this J., 1917. 382.

*[Production of light] liquid hydrocarbons.* Eng. Pat. 111,933. See II A.

#### IV.—COLOURING MATTERS AND DYES.

*The physical basis of colour-technology.* Luckiesh. See XIII.

##### PATENTS.

*Dyes from natural products; Production of*——. J. B. Duarte, S. Paulo, Brazil. Eng. Pat. 106,815, Jan. 30, 1917. (Appl. No. 1516 of 1917.) Under Int. Conv., May 31, 1916.

WASHED pulverised or unpulverised leaves of one or more of the mangrove species of the genus

*Rhizophora* are infused, macerated, digested, or decocted, and the liquid filtered or separated by a centrifuge, and concentrated *in vacuo* or otherwise, sodium or potassium bisulphite and other salts being subsequently added. The product ("Ingletina C") is composed of tannins, sodium chloride, mucilage, and non-classified colouring and resinous materials, giving when treated with alkalis and metallic salts a range of colours including black and chestnut and a number of coloured lakes applicable to fibres and textiles, and resistant to light and washing.—F. W. A.

*Colouring matters [sulphur dyestuffs]; Manufacture of*—— [from residues from manufacture of trinitrotoluene]. O. Silberrad, Buckhurst Hill, Essex. Eng. Pat. 111,738, Dec. 28, 1916. (Appl. No. 18,547 of 1916.)

RESIDUES obtained in the purification of trinitrotoluene, containing 2,3- and 3,4-dinitrotoluene and 2,3,4- and 3,4,6-trinitrotoluene, are fused with sodium sulphide and sulphur. The product dyes cotton direct a fine deep khaki brown from a sodium sulphide bath.—F. W. A.

*Vat dyestuffs; Process for producing*——. S. Sokal, London. From Kalle and Co. A.-G., Biebrich, Germany. Eng. Pat. 111,904, Dec. 14, 1916. (Appl. No. 17,982 of 1916.)

SEE U.S. Pat. 1,209,212 of 1916; this J., 1917. 211.

*Leuco derivatives of vat dyestuffs; Manufacture of*——. A. Brochet, Paris. U.S. Pat. 1,247,927, Nov. 27, 1917. Date of appl., Sep. 25, 1914.

SEE Fr. Pat. 473,536 of 1913 and Eng. Pat. 19,848 of 1914; this J., 1915, 709, 1047.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Sulphite cellulose; Constitution and stability of various lyes used in the manufacture of*—— and their specific action in the decomposition of wood. T. Marusawa, Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 737–773.

SOLUTIONS of sulphurous acid undergo considerable decomposition even at 130° C., a solution containing 0.6 gm.-mol. per litre of H<sub>2</sub>SO<sub>3</sub> being decomposed to the extent of 25% in 13 hours at that temperature, or in 2 hours at 150° C. After 6 hours at 150° C. about 42% of the acid is decomposed, mainly into sulphur and sulphuric acid, with a small amount of thiosulphuric acid. The addition of selenium has no appreciable influence on the rate of decomposition. A solution of sodium or ammonium bisulphite is very stable, a solution containing 0.6 gm.-mol. per litre, for example, being decomposed only to the extent of a few per cent. after 6 hours at 150° C., and of about 14% at 170° C., but in no case is sulphur separated. Below 150° C. the addition of selenium has practically no influence. A solution of sulphurous acid containing sodium or ammonium bisulphite is fairly stable below 150° C., and a solution containing 0.3 gm.-mol. per litre of each component undergoes only about 10% decomposition after 6 hours' heating at that temperature, whilst no sulphur separates. The rate of decomposition increases rapidly with the rise of temperature, the decomposition being practically complete after 6 hours' heating at 170° C. Selenium has a pronounced influence on the decomposition of the mixture, so that when heated for 6 hours at 150° C., with 0.01 gm. of selenium per litre, it is decomposed to the extent of over 80%, whilst a large amount of sulphur separates. Under parallel conditions the stability of the mixture decreases with the rise in the ratio of free sulphurous acid to bisulphite, independently of the presence or

absence of selenium. If a mixture of sulphurous acid and calcium bisulphite of the composition usually employed in sulphite-cellulose works is heated in a closed vessel calcium sulphite separates. To prevent this, the composition of the mixture must be so regulated that the ratio  $\text{H}_2\text{SO}_3 : 2\text{Ca}(\text{HSO}_3)_2$  exceeds a certain critical point. Other conditions being similar, this value depends upon the temperature and increases with the rise of temperature. For example, in the case of a solution containing 0.13 grm.-mol. per litre of calcium, the critical values at 100° and 140° C. were 1.1 and 1.6 respectively, when the volume of the solution was  $\frac{1}{2}$  that of the vessel. The conditions of decomposition of the mixture of calcium bisulphite and sulphurous acid are similar to those described for sodium and ammonium bisulphites. In course of time the calcium sulphite which first separates, as mentioned above, redissolves, whilst calcium sulphate is formed as a new deposit. The primary decomposition of sulphurous acid probably occurs in its dissociated part,  $\text{HSO}_3'$ , and is accelerated by hydrogen ions, and the stability of bisulphite solution, notwithstanding its high  $\text{HSO}_3'$  content, must be attributed to the want of an accelerator of the decomposition, owing to the very slight concentration of hydrogen ions. The action of selenium may be explained by the assumption that it forms with bisulphite, or more probably with  $\text{HSO}_3'$ , an unknown compound, which in the presence of  $\text{H}^+$  is exceedingly unstable.—C. A. M.

*Potash lye from vegetable ashes; Manufacture of —, and its application for boiling straw in the paper industry.* S. Tanaka. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 844—850.

ASHES from vegetable matters have been investigated from the point of view of the manufacture of caustic potash for the digestion of straw for paper making. Ashes from soy bean pods contained 16.19% of potassium carbonate and from chestnuts, 13.96%. For the manufacture of caustic potash it is necessary that the ashes should contain more than 10% of potassium carbonate. Lime may be added directly to the solution of ash in water without separating the insoluble residue. The yield of caustic potash depends very largely on the perfection of the filtration and lixiviation processes. The potash obtained is quite efficient for the manufacture of straw pulp and its substitution for caustic soda is a question of cost. A constant and sufficient supply of potash from these sources is hardly to be expected and the difficulties of the filtration process increase the cost.—J. F. B.

*Distillation of cellulose and starch in vacuo.* Pictet and Sarasin. See IIB.

*Use of textile fibres in microscopic qualitative chemical analysis.* Chamot and Cole. See XXIII.

#### PATENTS.

*Silk; Process for increasing the strength and elasticity and for preventing or decreasing the subsequent weakening of non-loaded raw or boiled fibres in —.* J. Korseff, Zittau, Saxony. Eng. Pat. 104,684, Mar. 5, 1917. (Appl. No. 3249 of 1917.) Under Int. Conv., Feb. 5, 1916.

THE silk is treated with alkaloids or with products of the decomposition of alkaloids which have a more complex molecule than urea and hexamethylenetetramine, especially with derivatives of pyridine, piperidine, pyrrole, pyrrolidine, quinoline, isoquinoline, phenanthrene, morpholine, or purine, provided these products do not modify the feel and appearance of the silk and provided they do not give any undesirable colour-reaction with iron

compounds. The silk is treated for 30 mins. at the ordinary temperature, or warm (up to 40° C.), in a solution containing from 1 to 10% of the alkaloid or its salt, e.g., cinchonine, caffeine hydrochloride, or carbocinchomeric acid, calculated on the weight of the silk. The increase in strength of the silk may be 10—50% according to the quantity of alkaloid used, and the substances named act in a protective sense against oxidising influences.—J. F. B.

*Textile belting and method of preparing the same.* H. St. L. Buchner, Wollaston, Mass. U.S. Pat. 1,249,019, Dec. 4, 1917. Date of appl., Dec. 18, 1916.

TEXTILE belting, driving tapes, and the like are saturated with a mixture of equal parts of refined oil asphalt and refined petroleum distillate, or asphaltic base lubricating oil by immersion therein at about 200° F. (93.5° C.).—J. F. B.

*Metallic yarn and method of treating same to facilitate weaving and for other purposes.* W. H. Underwood, New York. U.S. Pat. 1,249,252, Dec. 4, 1917. Date of appl., Dec. 30, 1914.

IN the manufacture of woven fabric containing rough metallic yarn, the yarn is coated before weaving with a material to fill the spaces in the surface of the fibres of which the yarn is made. The fibres of the metallic yarn may be coated with lead or the fabric may be made from spun metallic wool coated with a metal which resists the action of sulphur, or, generally, the fibres of the metallic yarn are treated with a corrosion-resisting material before weaving.—J. F. B.

*[Plastic fibrous] material capable of being cast or moulded by extrusion, rolling or other suitable means and the method of manufacturing same.* H. Jackson, Garstang, Lancs. Eng. Pat. 111,946, Jan. 26, 1917. (Appl. No. 1319 of 1917.)

FIBROUS material is reduced to a pulp and the major portion of the water is removed from the pulp by draining or by a hydro-extractor. The moist pulp is mixed with a dry powdered glutinous or gelatinous substance such as gum, starch, or glue, then heated, preferably in a closed vessel at 165°—212° F. (74°—100° C.). A plastic mass of pulped material is thus produced which may be formed in any desired shape by squirting, pressing, or rolling.—J. F. B.

*Fibrous plastic composition and process of making same.* C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,246,806, Nov. 13, 1917. Date of appl., Apr. 30, 1917.

THE solids in acid sulphite-cellulose waste liquor are precipitated in contact with fibrous material.—B. N.

*[Wood] pulp; Manufacture of chemical sulphate —.* C. N. Waite and J. E. Hedin, Wilmington, Del., Assignors to W. H. Sharp, Philadelphia, Pa. U.S. Pat. 1,249,287, Dec. 4, 1917. Date of appl., Feb. 3, 1914.

LIQUOR for the digestion of wood pulp is prepared from the spent liquor of a previous digestion by evaporating the water, burning off the organic matter, and dissolving the sodium carbonate in the ash, at the same time adding lime to causticise the soda and sufficient sulphur to convert part of the caustic soda into sodium sulphide.—J. F. B.

*Process and apparatus for recovering the ether and alcohol vapour contained in the air of factories.* Eng. Pat. 101,723. See I.

*Apparatus for recovering ether and alcohol vapours contained in the air of factories.* Eng. Pat. 101,875. See I.



*Process of making briquettes or other moulded articles. Briquetting process. Weather-resistant briquette.* U.S. Pats. 1,246,805, 1,246,807, and 1,246,808. See IIa.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Printing piece goods; Machines for* —. N. Tidswell, Bihorel, France. Eng. Pat. 111,970. May 3, 1917. (Appl. No. 6302 of 1917.)

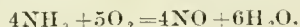
*Bast fibres; Dividing, laniflying, and bleaching* —. L. De W. Wante, Bowdon, U.S. Pat. 1,247,757. Nov. 27, 1917. Date of appl., Jan. 14, 1913. Renewed Feb. 5, 1917.

SEE FR. Pat. 458,289 of 1913; this J., 1913, 1063.

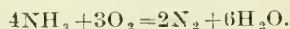
## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Ammonia oxidation process; Analytical control of* —. G. B. Taylor and J. P. Davis. J. Ind. Eng. Chem., 1917, 9, 1106—1110. (See also Fox, this J., 1917, 1005.)

The efficiency of an ammonia converter depends upon the establishment of conditions favourable to the reaction—



and unfavourable to the reaction—



and if nitric acid is to be the ultimate product little ammonia should be allowed to pass unchanged through the converter. The efficiency calculation is based upon the principle that the ratio of the nitrogen in combination as ammonia to the total nitrogen in the intake gas is equal to the ratio of the nitrogen derived from the ammonia to the total nitrogen in the exit gas. Hence the efficiency of a plant may be expressed by the equation—

$$\frac{c(a+b)}{a(c+d+f)} \times 100 = \text{per cent. yield.}$$

where  $a$  represents the nitrogen as ammonia and  $b$  the free nitrogen in the initial mixture of air and ammonia,  $c$  the nitrogen combined as nitrogen oxides,  $d$  the free nitrogen, and  $f$  the nitrogen combined as ammonia escaping oxidation in the exit gas. The most convenient method of controlling the process is to take samples of the gases in exhausted bottles of known capacity, provided with single capillary stopcocks, and with ground glass stoppers. Sufficient  $N/5$  sulphuric acid to neutralise the ammonia is admitted to the ammonia-air sample, and a measured quantity of water after absorption of the ammonia until the residual air is at atmospheric pressure, and the excess of acid is then titrated to obtain  $a$ . A measured volume of water containing hydrogen peroxide is drawn into the bottle containing the sample of reaction products to absorb acid vapours, and analysis of the residual mixture of oxygen and nitrogen gives the proportion of free nitrogen,  $d$ . The acid solution is titrated with  $N/5$  sodium hydroxide solution, and then distilled with sodium hydroxide into  $N/5$  sulphuric acid to obtain the amount of ammonia,  $f$ . Since this ammonia was present as nitrate, its equivalent is added to the free acid already determined for the calculation of  $c$ . To ascertain whether there is a leak between the point where the ammonia-air sample is taken and the point where the nitrous gases are sampled, the amount of oxygen admitted

to the sample of nitrous gas is measured, and water containing a measured quantity of hydrogen peroxide is introduced. The whole of the combined nitrogen will then be present as nitric acid. After determination of the excess of oxygen gas the liquid is divided into two parts, in one of which the acid is titrated with alkali whilst in the other the excess of hydrogen peroxide is determined by means of standard permanganate solution. The amount of oxygen required for the oxidation of the ammonia converted can then be calculated, and any excess will be due to leakage. Analysis of the intake gas of ammonia and air may also be made by the ordinary gas analysis methods, the ammonia being dried by passing it through a U-tube filled with pieces of solid potassium hydroxide, and absorbed in a bulb form of graduated burette by means of concentrated sulphuric acid, dry mercury being used as the confining liquid.—C. A. M.

*Nitrogen-hydrogen equilibrium; Effect of hydrogen chloride on the* —. E. B. Ludlam. Faraday Soc., Feb., 1917. [Reprint.] 10 pp.

THE early experiments of Deville on the vapour density of ammonium chloride at high temperatures suggest that ammonia is far more stable in the presence than in the absence of hydrogen chloride. The large evolution of heat and the marked volume contraction attending the formation of ammonium chloride from its elements suggest also that the conditions are more favourable to a union of nitrogen and hydrogen than when hydrogen chloride is absent. Synthetic experiments, at atmospheric pressure, with nitrogen and hydrogen in presence of hydrogen chloride, at various temperatures from 500° to 1500° C., were carried out with negative results. Equilibrium was approached much too slowly. Attempts were made to attain equilibrium from the other side by heating ammonium chloride alone to various temperatures up to 1200° C., in presence of a number of metallic catalysts such as gold, silver, zinc, copper, iron. The decomposition of ammonium chloride was found to be almost complete after 5 hours at 800° C. in presence of iron, showing that ammonium chloride is not stable at high temperatures. Its apparent stability is due to a very slow rate of decomposition. Synthetic experiments at 450° C. in presence of iron failed to show any advantage due to hydrogen chloride, but since the iron was all removed by volatilisation as ferrous chloride the question still remains open.—H. J. H.

*Potash; A neglected chemical reaction and an available source of* —. E. A. Ashcroft. Inst. Min. and Met., Dec. 20, 1917. [Advance proof.] 20 pages.

THERE is a reversible reaction between dry sodium chloride and orthoclase felspar when heated together in a state of fine division in the absence of air or moisture. In the state of equilibrium the distribution of the alkalis depends on their relative proportions:  $-2\text{NaCl} + \text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 = \text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 + 2\text{KCl}$ . If the felspar (10%  $\text{K}_2\text{O}$ ), crushed to pass a 100-mesh (I.M.M.) sieve and mixed with its own weight of salt, is heated for 2 hours at 900°–1000° C., 85% of the potash will be converted into a soluble form without loss by volatilisation. A temperature above 1100° C. is to be avoided as conducing to fritting, while the presence of air and moisture leads to loss of alkali and destruction of any metal containers by chlorine and hydrogen chloride evolved. The reversibility of the reaction was demonstrated by heating the product of the above reaction with excess of potassium chloride, when the potash felspar was regenerated. The product of the direct reaction as above yields on lixivitation a

mixture containing about 90% of NaCl and 10% of KCl, which can be separated by crystallisation. It is suggested that the reaction should be applied to the extraction of potash from British felspars. Apart from the expensive high-grade felspars used in pottery manufacture, there is a large quantity, estimated at 20,000,000 tons at least, of pegmatite exposed on the extreme north-west coast of Sutherlandshire, Scotland. It contains 8% of K<sub>2</sub>O and its estimated cost, quarried and picked, is 2s. 6d. per ton, on a site well suited for the erection of works for treatment and favourable as regards conditions of water-borne transport. It is suggested that a form of vertical retort might be used in which molten salt percolates a heated charge of felspar. Alternatively the charge might be placed in iron boats, closed with lids, and pushed through a heated horizontal gas retort in which an atmosphere of producer-gas is maintained. The boats would form a continuous train and would remain for 2 hours in the hot zone and 1 hour in the cooling region. It is estimated that 80% KCl could be produced in normal times at a cost of £7 per ton, thus competing favourably with German supplies. The analytical methods for dealing with mixtures of potassium and sodium chlorides are discussed. The "indirect method" based on the estimation of the total chlorine is recommended as accurate to within 1% if care is taken and the mixture titrated against silver nitrate is free from chlorides of other metals. Iron, manganese, calcium, and magnesium are removed by boiling first with ammonium carbonate and filtering, then with a crystal of oxalic acid, and again filtering, and evaporating to dryness with hydrochloric acid. The residue dissolved in water is titrated with N/10 silver nitrate, using weak chromic acid solution as indicator.—H. J. H.

*Sodium cyanide; Hydrolysis of*—F. P. Worley and V. R. Browne, *Chem. Soc. Trans.*, 1917, 111, 1057—1062.

THE hydrolysis of sodium cyanide was measured by comparing the concentration of hydrogen cyanide vapour above solutions of sodium cyanide and of hydrogen cyanide. Air was drawn at the rate of one bubble per second through caustic soda solution to remove carbon dioxide, and then through the following solutions in series in the order given, hydrocyanic acid—indicator—sodium cyanide—indicator—hydrocyanic acid—indicator. The indicator was a solution containing 0.2% of picric acid and 2% of sodium carbonate. With hydrogen cyanide vapour it gives a reddish-brown coloration, the depth of which depends on the concentration of the vapour. The concentrations of the hydrocyanic acid solutions on each side of the sodium cyanide solution were adjusted until one had a vapour pressure just above and the other just below that of the sodium cyanide solution. The degree of hydrolysis of the latter was then defined between narrow limits. From the equation  $\text{NaCN} + \text{H}_2\text{O} \rightleftharpoons \text{NaOH} + \text{HCN}$ , it follows that when the water is in large excess—  
 $\text{conc. of NaOH} \times \text{conc. of HCN} = \text{conc. of NaCN} \times K$ .  
 If neither alkali nor acid is added, and C is the concentration of total sodium cyanide, and P the percentage hydrolysed,

$$\frac{P^2C}{100(100-P)} = K.$$

Experiments at 25° C. on solutions of sodium cyanide varying in strength from 5.2 to 0.0073 gm.-mol. per litre gave the value of  $K \times 10^4 = 0.484$ . The percentage hydrolysis of a solution containing 0.0435 gm.-mol. of sodium cyanide per litre, when different amounts of caustic soda

and hydrochloric acid were added per gm.-mol. of sodium cyanide, varied from 0.11% for 1 gm.-mol. of caustic soda to 2.8% for 0.01 gm.-mol. of caustic soda, and from 10.9% for 0.1 gm.-mol. of hydrochloric acid to 40.175% for 0.4 gm.-mol. of hydrochloric acid. The value of  $K \times 10^4$  varied from 0.135 at 0° C. to 0.72 at 30° C.—F. Sp.

*Polysulphides of alkali metals. Part III. Solidifying points of the systems sodium monosulphide-sulphur and potassium monosulphide-sulphur.* J. S. Thomas and A. Rule. *Chem. Soc. Trans.*, 1917, 111, 1063—1085.

To ascertain the number and the relative stability of the solid polysulphides of sodium and potassium, the freezing-point curves of the systems sodium monosulphide-sulphur and potassium monosulphide-sulphur, and the rate of desulphurisation of the higher polysulphides in a stream of hydrogen at regularly increasing temperatures, have been determined. The results indicate the existence of a complete series of compounds of the general formula  $\text{R}_x\text{S}_x$ , where  $x$  is a whole number having the maximum value 5 in the sodium series and 6 in the potassium series. No evidence was obtained of the existence of intermediate compounds, such as the enneasulphide described by Bloxam (this J., 1899, 684). The variation in the rate of loss of sulphur of sodium tetrasulphide on heating in a current of hydrogen indicates that it undergoes considerable decomposition below 300° C., and then loses sulphur regularly until the composition of the fusion approaches that of the disulphide, between 625° and 650° C. The disulphide is much more stable to heat than the higher sulphides. Potassium pentasulphide shows practically the same behaviour, but there is a slight retardation in the loss of sulphur in the region where the composition of the fusion approximates to that of the trisulphide. The disulphide is the most stable of the potassium polysulphides. None of the results obtained supported Bloxam's view (*loc. cit.*) that the polysulphides should be represented by the formula  $\text{R}_x\text{S}_x$ , and determinations of the molecular weights in alcohol, by the ebullioscopic method, of sodium disulphide, sodium tetrasulphide, and potassium pentasulphide, gave in all cases results lower even than those demanded by the simple formula  $\text{R}_2\text{S}_2$ . To explain the stability of the disulphides, the authors suggest the formula  $\text{R.S.S.R.}$ , the trisulphides being  $\text{S:(R)S.S.R.}$  and the tetrasulphides  $\text{S:(R)S.S(R):S.}$ —F. Sp.

*Rare earths; Observations on the*—IV. *Purification of gadolinium.* L. Jordan and B. S. Hopkins. *J. Amer. Chem. Soc.*, 1917, 39, 2614—2623.

THE fractional crystallisation of dimethylphosphates, bromates, and glycolates of the rare earths has been examined with a view to the separation of pure gadolinium from rare earth mixtures. Fractionation of the dimethylphosphates quickly removes europium and samarium, and from the middle fractions, which are very rich in gadolinium, terbium may be removed by fractionation of the bromates. The method of procedure recommended for the separation of gadolinium from rare earth mixtures is to remove cerium by the bromate method. The residual bromates are fractionally crystallised. The less soluble fractions, containing only europium, samarium, gadolinium, and neodymium, are converted into the dimethylphosphates, and on fractionation, the least soluble fraction consists of high-grade gadolinium. Any remaining traces of samarium can be removed by precipitation with sodium glycolate.—H. M. D.



*Tartrates ; Test for*—depending upon the formation of the copper-tartrate complex. L. J. Curtman, A. Lewis, and B. R. Harris. J. Amer. Chem. Soc., 1917, **39**, 2623—2630.

A SYSTEMATIC examination of the alkaline copper test for tartrates has led the authors to recommend the following procedure. To 5 c.c. of the solution is added 3 c.c. of a 2.5% solution of sodium hyaroxide and 1 c.c. of *N*/5 copper sulphide. The volume is made up to 10 c.c. and the mixture shaken for 1 minute. The mixture is then filtered, the filtrate acidified with 50% acetic acid, one drop of potassium ferrocyanide solution added, and the difference noted after 1 minute. According to the quantity of tartrate present, a pink coloration or a reddish-brown precipitate makes its appearance. It is possible to detect 0.2 mgrm. of tartrate in this way and the use of ferrocyanide, in place of ammonia, makes the test appreciably more sensitive. Solutions which contain ammonium salts, arsenites, borates, or phosphates give a positive reaction in the absence of tartrates. If the quantity of tartrate is small, the reaction is observed in the presence of thiosulphates, arsenates, chromates, fluorides, thiocyanates, nitrites, oxalates, and acetates.—H. M. D.

*Silica ; Solubility of*—V. Lenher and H. B. Merrill. J. Amer. Chem. Soc., 1917, **39**, 2630—2638.

THE solubility of silica in water and acid solutions is a factor of importance in connection with the analytical estimation of silica, and measurements have accordingly been made of the solubility in water, and in solutions of hydrochloric and sulphuric acid at 25° and 90° C. With gelatinous silica, saturation is reached after a few hours or days, but with ignited silica the attainment of equilibrium requires a much longer time. The results obtained show that the solubility of gelatinous silica is independent of the mode of preparation of the substance, and that ignited silica tends towards the same limit in respect of the concentration. The solubility increases with the temperature, and at a fixed temperature, decreases with increase in the concentration of the mineral acid. Carbonic acid has no measurable influence on the solubility of silica. At 90° C. the solubility of gelatinous silica in grms. per 50 c.c. is 0.0212—0.0216 grm. in conductivity water, 0.024 grm. in 1.2% HCl, 0.0028 in 18.7% HCl, 0.0211 in 3.9% H<sub>2</sub>SO<sub>4</sub>, and 0.0004 in 71% H<sub>2</sub>SO<sub>4</sub>. At 25° C., the solubility figures obtained after digesting for 2 days were: water, 0.0032; 6.4% HCl, 0.0044; and 34.9% HCl, 0.004 grm. per 50 c.c.

—H. M. D.

*Manufacture of potash lye from vegetable ashes and its application for boiling straw in the paper industry.* Tanaka. See V.

*Action of caustic soda on steel plates.* Stromeyer. See X.

#### PATENTS.

*Acid-collecting apparatus.* E. T. Keller, City Point, Va. U.S. Pat. 1,247,280, Nov. 20, 1917. Date of appl., Jan. 10, 1917.

AN arrangement of a series of towers is provided with reversely arranged baffles having spiral, radially disposed deflecting blades, in which the acid fumes pass upwards and the washing liquid downwards. The gas outlet of one tower is connected with the inlet of the next, and the acid liquid, collected in a chamber at the bottom of one

tower, is elevated to the distributing chamber of the next and sprayed into the tower under pressure. —B. V. S.

*Nitre-cake ; Utilisation of*—J. Grossmann, Manchester. Eng. Pat. 111,875, Nov. 18, 1916. (Appl. No. 16,555 of 1916.)

By heating a mixture of sodium nitrate, nitre cake, and carbonaceous material in suitable proportions to about 250° C., the nitrogen is evolved almost entirely as nitric acid or oxides easily oxidised to nitric acid, there being no nitrogen or nitrous oxide formed; the residue is an easily friable form of slightly acid sodium sulphate with carbonaceous material. The process may be conducted at ordinary, increased, or reduced pressure, and air may be blown or drawn through part or all of the system. The evolved nitrous gases may be treated with air and steam for the production of nitric acid or passed to a sulphuric acid plant, etc.—B. V. S.

*Ammonia and potassium-containing material from tobacco waste ; Process of producing*—J. E. Zilk. Assignor to Odorless Fertilizer Manufacturing Co., Pittsburgh, Pa. U.S. Pat. 1,247,060, Nov. 20, 1917. Date of appl., Feb. 10, 1917.

TOBACCO waste is decomposed by heat in the absence of air. The charcoal residue contains water-soluble potassium compounds. The gases are withdrawn and heated further to convert the nitrogenous compounds into ammonia, which is recovered by condensation.—B. V. S.

*Sodium silicofluoride ; Method of making*—K. F. Stahl, Pittsburgh, Pa. U.S. Pat. 1,247,165, Nov. 20, 1917. Date of appl., Mar. 17, 1917.

A PHOSPHATE rock containing fluoride is treated with sulphuric acid, a soluble phosphate and silicon fluoride being formed. The silicon fluoride is converted into hydrofluosilicic acid which is treated with sodium sulphate with the production of sodium silicofluoride and sulphuric acid.—B. V. S.

*Manganese carbonate ; Process for the oxidation of*—M. L. Kaplan, Brooklyn, N.Y. U.S. Pat. 1,247,278, Nov. 20, 1917. Date of appl., Apr. 30, 1917.

MANGANESE carbonate is oxidised by heating at a temperature above 300° C. with nitric acid vapour or with air mixed with acid or mixed with nitrogen oxides containing a higher proportion of oxygen than nitric oxide.—B. V. S.

*Alkali [from silicates] ; Recovery of*—J. S. Beckett, Cranford, Assignor to P. R. Moses, New York. U.S. Pat. 1,247,619, Nov. 27, 1917. Date of appl., Apr. 24, 1916.

A SILICATE containing an alkali metal (potassium) is heated in a furnace with a basic compound and a chloride. The alkali (potassium) chloride thus formed is extracted with water and the solution electrolysed. The resulting chlorine is passed into a mixture of lime and potassium chloride; chlorate is formed and the greater part of it separated by crystallisation. The residue containing calcium chloride and the remainder of the potassium salt is used for the treatment of more silicate.—B. V. S.

*Molybdenum disulphide ; Process for producing*—D. L. Johnson, Los Angeles, Cal. U.S. Pat. 1,248,867, Dec. 4, 1917. Date of appl., July 7, 1917.

A MIXTURE of finely-divided sodium molybdate and sulphur, in the proportion of 200 parts of molybdenum trioxide and 250 parts of sulphur, is heated. The insoluble molybdenum disulphide is washed to free it from soluble substances, including compounds of phosphorus and arsenic which may have been present in the molybdate. —B. V. S.

*Magnesia; Process of obtaining* —. H. McLoughlin. Assignor to F. D. Carthew, San Francisco, Cal. U.S. Pat. 1,218,880, Dec. 4, 1917. Date of appl., Feb. 9, 1916.

MAGNESITE containing silica is calcined to remove carbon dioxide and the resulting magnesia disintegrated by quenching while still hot. The coarse silica is removed by sifting and the residue is ground to pass a 30-mesh screen. A relatively pure magnesia is then separated by washing with a stream of water regulated to carry away the part of the mass having sp. gr. about 2.2 and to leave the part having sp. gr. 3 or higher.—B. V. S.

*Aluminium compounds; Process for producing* —. F. Langford, Eureka, Cal. U.S. Pat. 1,249,125, Dec. 4, 1917. Date of appl., Aug. 5, 1916.

CRUDE aluminium ores are extracted first with sulphurous acid and then with dilute sulphuric acid. The resulting solutions are mixed, filtered through redwood fibre, and evaporated to dryness, and the residue is calcined.—B. V. S.

*Chlorine; [Electrolytic] process for the extraction of* —. P. L. Hulin, Grenoble, France. U.S. Pat. 1,246,099, Nov. 13, 1917. Date of appl., Sep. 15, 1916.

A GASEOUS mixture containing chlorine is washed with a solution of ferrous chloride or other suitable lower chloride. The higher chloride thus produced is acidified with hydrochloric acid and electrolysed, with the formation of chlorine and the lower chloride which is utilised again.—B. V. S.

*Process of producing alkali carbonate and hydraulic cement from alkaliferous silicate minerals.* Eng. Pat. 103,290. See IX.

*Stable carbonic acid solution and method of producing same.* U.S. Pat. 1,247,334. See XI.A.

## VIII.—GLASS; CERAMICS.

*Chemical stoneware; Standardisation of* —. H. Nielsen and J. R. Garrow. Chem. Trade J., 1918, 62, 3—5, 23—25.

THERE is no valid reason why almost every article made of chemical pottery should not be standardised almost as closely as are articles made of iron or steel, and the authors appeal to makers and users to adopt standard sizes. Stoneware is less homogeneous than a metal and less is known of its resistance to stress. The ultimate tensile strength of high-class vitreous pottery lies between 600 and 842 lb. per sq. in., 600 lb. being a safe figure for almost any vitreous stoneware used in this country for acid-resisting pipes. The wall-thickness,  $w$ , of a pipe of diameter,  $d$ , required to resist an internal pressure,  $p$ , per unit of area, the stoneware having an ultimate tensile strength,  $u$ , is

$$w = \frac{pd}{2u}$$

This wall-thickness is usually multiplied by a safety factor of 4. The force resisting the tendency to fracture a pipe in direction of its length, owing to the presence of right-angle bends, stopcocks, etc., is

$$\frac{\pi}{4} d^2 \times \rho$$

$$\frac{\pi}{4} \left\{ (d + 2w)^2 - d^2 \right\}$$

and this quotient divided into the ultimate tensile strength of the stoneware gives the factor of safety in a longitudinal direction.

Tables and diagrams are given of the suggested principal dimensions for standard pipes, bends,

T-pieces, cross-pieces, stopcocks, bib-cocks, and iron connecting clips. The wall-thickness of pipes of 1 and 1½ in. diameter are made slightly greater than the calculated values in order to facilitate manufacture. A conical buttressed flange is preferred to a flat one. Bends should have as large a radius as conveniently possible and as it is often desired to replace a bend by a T-piece, or *vice versa*, the same centre-distances should be used throughout. A centre distance equal to the internal diameter + 4 in. is suggested. The flanges of bends should be the same as those on straight pipes. For T-pieces and cross-pieces with unequal branches, the centre-distance for the unequal branch should be  $\frac{1}{2}(a+a_1)+4$  in., where  $a$  is the internal diameter of the equal branches, and  $a_1$  that of the unequal branch. The wall-thickness and flanges of T-pieces and cross-pieces should be the same as in straight pipes. Stopcocks for liquids under pressure should be of as simple design as possible, and preferably of the same length as standard T-pieces. The plugs should be well secured by an adaptable bridge, as there is a considerable force tending to blow them out. They should be carefully ground by machinery and polished by hand. All cocks should be immersed in water, tested by compressed air, and then marked. In bibcocks, the spigot should be of ample size with a standard conical flange; the tangent to the angle of the cone to be ground into the container should be about 0.05. Three-way and other special cocks should be avoided where possible. An arrangement of standard T- and cross-pieces and plain cocks will serve the same purpose, be more reliable, and will cost less for repairs. Iron jointing flanges or clips may conveniently consist of two pieces bolted together, with a suitable packing between the metal and the stoneware. The faces of the two pieces should be machined to ensure an accurate fit, and the whole device should be boiled in pitch before use. All holes should be drilled and not cored. Packing rings for use between the stoneware pipes should be made from woven asbestos cloth or asbestos millboard which has been boiled for three hours in soft paraffin wax or soaked in a strong solution of sodium silicate. For hydrochloric acid, pure soft Para rubber rings may be used.—A. B. S.

*Chemical stoneware; Standardisation of* —. H. Griffiths. Chem. Trade J., Jan. 12, 1918.

THE formula suggested by Nielsen and Garrow (see preceding abstract) for calculating wall-thickness is applicable to thin cylinders such as boiler shells, but not to pipes the thickness of whose walls is of the same order of magnitude as the internal radius. The quality of chemical stoneware is so variable that standardisation of sizes in accordance with calculated values is impracticable unless an excessively high factor of safety is used. The first essential is the standardisation of material rather than of form.—A. B. S.

*Porcelain glaze: The application of talc for* —. S. Kaneshima. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 850—860.

THE influence of talc as a constituent of porcelain glaze has been studied by making up glazes in which MgO replaced CaO in equimolecular proportions, the other constituents being identical. The talc employed was obtained from S. Manchuria and had the composition SiO<sub>2</sub>, 65.32; MgO, 32.47, CaO, 0.72, Fe<sub>2</sub>O<sub>3</sub> and Al<sub>2</sub>O<sub>3</sub>, 0.12%. The calcium glazes employed for comparison were prepared with precipitated calcium carbonate, containing only a trace of iron. The other ingredients of the glazes were felspar, Gairome clay, and silica. A large number of mixtures were



made affording a wide range of comparative variations. The experiments showed that the talc glazes were more fusible than the calcium carbonate glazes but not so liable to overfuse, and the formation of bubbles in the finished glaze was of rarer occurrence. Talc glaze was generally more lustrous than calcium carbonate glaze, but the development of colour of underglaze pigments was less brilliant.—J. F. B.

*The physical basis of colour technology.* Luckiesh.  
See X111.

#### PATENTS.

*Forming molten glass or like material in cylindrical form; Apparatus for*—. E. C. R. Marks, London. From The Libbey Glass Co., Toledo, Ohio, U.S.A. Eng. Pat. 111,521, Nov. 29, 1916. (Appl. No. 17,132 of 1916.)

A SLIGHT modification of Eng. Pat. 110,642 of 1916 (this J., 1917, 1277). The molten glass flows on and around an inclined rotating mandrel contained within a heated shell made of refractory material, the sections of the shell and mandrel being adjusted to regulate the thickness of the walls of the cylinder. After leaving the mandrel the glass is drawn out to the required diameter. A hollow mandrel may be used and air blown through it to produce a tube.—A. B. S.

*Strengthened glass; Manufacturing*—. S. Goldreich, C. A. McKerrow, and Splinterless Anti-Mist Glass Syndicate, Ltd., London. Eng. Pat. 111,539, Dec. 2, 1916. (Appl. No. 17,346 of 1916.)

STRENGTHENED glass is made by immersing a celluloid sheet in methylated spirit or its vapour for about 2 mins. and then placing it between two specially cleaned sheets of glass and pressing them together in a spring press under a pressure of about 100 lb. per sq. in. The press and its contents are then heated in an oven to 120° C. for 1 hour and are afterwards removed and allowed to cool very slowly. The celluloid will adhere uniformly to the glass and the edges may be sealed with a coat of lead paint or lacquer, or by other suitable means.—A. B. S.

*Glass; Method of gathering*—. C. F. Cox, Bridgeton, N.J., U.S.A. Eng. Pat. 111,899, Dec. 11, 1916. (Appl. No. 17,791 of 1916.)

A GATHERING machine of the type described in Eng. Pat. 2992 of 1907 is used, comprising a system of suction devices or moulds mounted on the circumference of a rotating table. Each mould is brought in turn over an extension of the furnace containing the molten glass and is lowered below the surface of the glass, filled by suction, moved forward, and then withdrawn. This dipping and withdrawing of the moulds produces a circulation of the molten glass in the furnace, whereby the portion which has been chilled by the mould is moved into a hotter part of the furnace and re-heated. Drippings of surplus glass from the moulds are similarly circulated and re-heated.—A. B. S.

*Glass manufacture.* T. J. McCoy, Kane, Pa. U.S. Pat. 1,246,764, Nov. 13, 1917. Date of appl., Oct. 4, 1911.

A GLASS-MELTING tank-furnace is connected with a second tank. A counterbalanced device operated automatically maintains the surface of the glass at a constant level in the second tank and in gathering basins on its circumference. Means are provided for maintaining the glass in the second tank at a suitable temperature.—A. B. S.

*Sheet-glass; Apparatus for making*—. J. Barnes, Assignor to S. R. Seybold, New Kensington, Pa. U.S. Pat. 1,247,617, Nov. 27, 1917. Date of appl., Mar. 21, 1917.

AN apparatus for making sheet glass comprises a furnace and an arrangement of rolls for withdrawing the molten glass from the furnace, partially forming it into a sheet, and passing it through a rubber having adjustable casings forming water-jackets and solid wooden blocks. The rubbing blocks may be brought to bear on the sheet of glass or removed from its path without affecting its position in the rolls. A Lehr, with conveyor, for receiving the sheet of glass, is placed near the outlet of the furnace, the conveyor and rolls being driven at the same speed.—A. B. S.

*Sheet glass; Method and apparatus for making*—. I. W. Colburn, Assignor to The Toledo Glass Co., Toledo, Ohio. U.S. Pat. 1,248,809, Dec. 4, 1917. Date of appl., Jan. 8, 1916.

A SHEET of glass is drawn vertically from a molten mass, through a cooling space and thence into a heated space in which the sheet is bent at right angles over a roller, and the temperature of the horizontal portion of the sheet is then raised until it softens. The sheet is then gradually cooled. Previous to bending the sheet, one face of it may be hardened by cooling.—A. B. S.

*Casting pottery and other like articles.* B. J. Allen, Blythe Bridge, Staffs., and R. Y. Ames, Gidea Park, Essex. Eng. Pat. 111,643, July 14, 1917. (Appl. No. 10,238 of 1917.) Addition to Eng. Pat. 110,649, Dec. 4, 1916 (this J., 1917, 1274.)

THE ware is cast in moulds surrounded by a vacuum chamber, as previously described but, in addition, pressure is applied to the casting slip by means of compressed air or steam or by a suitable head of slip, so as to enhance the effect of the vacuum, expedite the manufacture, and improve the quality of the ware.—A. B. S.

*Depositing clay, plumbago and other like products on absorbent moulds. Manufacture of clay, plumbago, and other like articles by electro-deposition on metal moulds.* B. J. Allen, Stoke-on-Trent. Eng. Pats. (A) 111,762, Feb. 15, 1917 (Appl. No. 2252 of 1917), and (B) 111,775, Mar. 3, 1917 (Appl. No. 3160 of 1917).

(A) THE material to be deposited is made into a slip with the suitable addition of acid or alkali and water, and is poured into a plaster mould provided with a vacuum chamber as described in Eng. Pat. 110,649 of 1916 (this J., 1917, 1274). An electric current is then passed through the mould and slip by connecting one wire to the outside of the mould and the other to an electrode immersed in the slip. The current causes the particles in suspension in the slip to travel towards the outside of the mould, and they are eventually deposited on the interior of the mould. A clear liquid accumulates around the immersed electrode and may be siphoned off, leaving the clay or other material deposited in the required shape. The density of the deposit may be reduced by reversing the electric current, or the attracting electrode may be applied to the parts of the deposit which are required to be thickest and the repelling (internal) electrode to those which are required to be thinnest. Reversing the electric current also facilitates the removal of the article from the mould. (B) In making articles of silica and plumbago by the process described above, it is not necessary to use absorbent moulds, and the material may be deposited directly on the metal electrode, which is shaped to give the formation of the desired article. The current required is of the order of 50 to 70 ampères at 50 to 100 volts.

being dependent on the material to be deposited. The process is applicable to the manufacture of crucibles, muffles, tubes, cores for furnaces, etc.—A. B. S.

*Refractory [material] and process of making same.* J. O. Handy and R. M. Isham. Assignors to A. S. Davison Co., Pittsburgh, Pa. U.S. Pat. 1,248,486, Dec. 4, 1917. Date of appl. Mar. 29, 1917.

DEAD-BURNED dolomite is converted into a sintered material which resists the action of boiling water by mixing it with a temporary binder, moulding it into bricks, and burning at a high temperature.—A. B. S.

*Bricks and other moulded articles.* H. W. Charlton. Jonespoint, N.Y. U.S. Pat. 1,249,028, Dec. 4, 1917. Date of appl., Apr. 28, 1917.

BRICKS or other articles made by moulding a mixture of rock material with a binder, obtained by digesting silicate material, such as feldspar, with lime, are subjected to a steaming operation.—A. B. S.

*Process and apparatus for pulverising and treating ores, clag, and other mineral substances.* U.S. Pat. 1,246,514. See 1.

## IX.—BUILDING MATERIALS.

*Cement clinker; Microscopical examination of —.* T. Hattori. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 701—708.

SURFACES of cement clinker etched with water, or with dilute hydrochloric or hydrofluoric acid, show two predominating constituents, viz., alite, which is composed of crystals with square or hexagonal cross-section, and celite, the ground mass between these crystals, which is composed of two distinct substances, one white and the other grey. Eutectic structures may also be observed in the celite, especially in the case of clinker from stationary kilns. Alite is more readily attacked by water and dilute hydrochloric acid than the ground mass (celite), but offers greater resistance to dilute hydrofluoric acid. Some clinkers after being etched with dilute hydrofluoric acid show unattacked clusters of white granules, which, however, behave like alite when the specimens are etched with water or dilute hydrochloric acid, and have therefore been mistaken for a constituent of alite. For this reason hydrofluoric acid is preferable to hydrochloric acid or water as an etching reagent. The difference in the rate of cooling accounts for the presence of finer alite grains in clinker from a stationary kiln than in that from a rotary kiln. The alite grains in well-burnt clinker have sharp outlines, whereas in the case of insufficiently burnt clinker the forms are round and indefinite. The quantity of alite increases with the rise in the proportion of lime in the clinker. The quantity of alite is not the sole criterion of the quality of cement clinker, for its distribution must also be taken into account. In the case of good clinker there is uniform distribution, whilst poor clinker shows irregular distribution of groups of alite and celite. Hence chemical analysis does not afford sufficient data for judging as to the quality of cement clinker.—C. A. M.

*Portland cement briquettes; Tensile strength test of neat —.* T. Hattori. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1018—1026.

OBJECTION is taken to the official Japanese method of testing the tensile strength of neat cement briquettes after 7 days instead of testing after 7 and 28 days the mortar prepared from it. Since the value of cement for mortar cannot be inferred from a test applied to the neat cement, and there

is no proportional relationship between the strength of a cement and of its mortar, it is suggested that for the future all tests should be applied to a mixture of cement and sand. The value of a cement depends upon the proportion of "flour" which it contains, for the grit (coarse particles) does not undergo hydration when mixed with water. By increasing the proportion of "flour" the tensile strength of neat cement briquettes is decreased, whilst that of the mortar briquettes is increased, and the reverse effects are produced by increasing the proportion of grit. Under the microscope hardened cement shows a mass of non-hydrated grains embedded in the hydrated mass, and as these grains are harder than the rest of the mass, the hardness of the section of neat cement when set is proportional to the percentage of grit therein. This would account for the low tensile strength of neat briquettes made from fine cement, whilst the proportion of sand which can be carried by fine cement causes it to yield a dense mortar which produces briquettes of high tensile strength.—C. A. M.

*Cement floors subjected to injurious liquids; Protective treatments for —.* W. P. Anderson. Met. and Chem. Eng., 1917, 17, 667—668.

TESTS were made on six different concretes prepared by the use, in varying proportions, of cement with sand or pebbles or both. Of the protectors tested nine were chemical hardeners (aluminium sulphate solutions varying in strength from 4% to 35%), three were oil hardeners, six were paints, two "blank" acid-proof coatings, and the others linseed oil with turpentine, a liquid preservative, paraffin wax, and a cement floor filler. The slabs were treated with acetic, sulphuric, nitric, and hydrochloric acids, sweet milk allowed to turn sour, and sugar solution. Some of the liquid hardeners afford good protection against dilute hydrochloric or acetic acid, and to a less extent against dilute nitric or sulphuric acid. Some paints and cement fillers, but not liquid hardeners, give good protection against strong hydrochloric acid. None of the protective agents tested was efficient with strong sulphuric acid. The milk tests were not complete but suggested the inadvisability of using paint as a protection against milk. Sugar was without effect on the concrete or the protective coverings.—B. V. S.

*Asphalt industry; The colloidal state of matter in its relation to the —.* C. Richardson. St. Paul (Minn.) Eng. Soc., May, 1917. Met. and Chem. Eng., 1917, 17, 650—653. (See also this J., 1917, 204.)

AN asphalt pavement may be regarded as a suspension of suitably graded sand and a finely-divided mineral filler in bitumen, which, from a physical point of view, may be treated as a liquid. In such a system an important factor is the surface energy, involving the relative surface, i.e., the smallness of grain, and the surface tension of the bitumen. This confirms the empirical conclusions from practical experience with pavements. An asphalt pavement is a colloidal solution in which the suspended matter, owing to the great viscosity of the continuous phase, may be of considerable coarseness. Clay can be introduced into asphalt by making a suspension in a liquid oily bitumen which can then be incorporated with a harder bitumen. The preliminary emulsification may be made in water which must then be removed by evaporation from the final product. Without some such treatment the clay grains cannot be wetted with bitumen owing probably to air films. Great differences were found in the colloidal capacity of bitumen samples when 1 part of colloidal clay was suspended in 2 parts of bitumen



and maintained at 325° F. (163° C.) for 24 hours. The percentage deposition of clay varied from nil in the case of Trinidad residual to 35.8% from a Mid-Continental semi-paraffin residual. Similar relations were found when the bitumens were diluted with carbon bisulphide. When solutions of varying concentration were made by dissolving Trinidad asphalt in benzol it was found that the capacity for carrying clay in suspension varied with the concentration and viscosity of the solution.—H. J. H.

#### PATENTS.

*Alkali carbonate and hydraulic cement from alkaliferous silicate minerals; Process of producing* — E. W. Jungner, Kneippbaden, Sweden. Eng. Pat. 103,290, Dec. 8, 1916. (Appl. No. 17,708 of 1916.) Under Int. Conv., Jan. 7, 1916.

A FINELY-DIVIDED mixture of feldspar or similar silicate, lime or other calcium compound, and carbon or suitable organic substance is heated in the presence of carbon dioxide or combustion gases or (if an electric furnace be employed) in the presence of an adequate supply of air, until complete volatilisation of the alkali is effected without melting the charge. The alkali is recovered, as carbonate, by conducting the gaseous products of the reaction into water and evaporating the resulting solution. The ratio of lime (CaO) to silica plus sesquioxides ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) in the raw mixture should be at least 1.7 to 1 for the production of a satisfactory cement clinker.

—W. E. F. P.

*Cementitious material; Calcareous — and process of making the same.* W. E. Carson, Riverton, Va. U.S. Pat. 1,248,455, Dec. 4, 1917. Date of appl., Jan. 27, 1917.

THE plasticity of commercial fine ground cement is improved by the addition of a further quantity of the cement ground to a much greater degree of fineness.—W. E. F.

*Concrete flooring and composition therefor.* C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,246,827, Nov. 13, 1917. Date of appl., Apr. 26, 1917.

A CONCRETE floor is coated with a hydrocarbon solution of coumarone resin having a melting point of 55°–75° C. and substantially free from saponifiable substances. The resin gives a slightly tacky surface which is non-slipping but not seriously dust-absorbing.—W. E. F.

*Wood; Substitute for — in building construction, and method of producing the same.* W. H. Wheeler, New York. Eng. Pat. 111,686, Oct. 5, 1916. (Appl. No. 14,177 of 1916.)

A CEMENT containing about 60% of calcium oxide, such as Portland cement, is mixed with a binder such as sodium silicate and then with a fibrous material or filler such as asbestos or sawdust, so as to produce a plastic mass. Suggested proportions are filler 30%, cement 10 to 40%, and silicate 30 to 60%. The plastic mass is heated between former-plates or in moulds in a press as described in Eng. Pat. 1390 of 1912, until all moisture is removed. The cement makes the final product resistant to water and frost and enables it to be painted without special preparation. The silicate gives resilience to the product and facilitates its being worked with tools used for wood.—A. B. S.

*Wood; Solution for preserving* — F. Seiden-schnur, Charlottenburg, Germany. U.S. Pat. 1,248,022, Nov. 27, 1917. Date of appl., Feb. 3, 1912.

A SOLUTION for preserving wood is composed of water, an insoluble basic zinc compound, such as zinc hydroxide, and aluminium sulphate.—A. B. S.

*Lumber; Process for rendering — resistant to sapstain.* C. H. Teesdale, Madison, Wis. U.S. Pat. 1,248,711, Dec. 4, 1917. Date of appl., Apr. 9, 1917. (Dedicated to the public.)

LUMBER is rendered resistant to sap-stain—a discoloration caused by organisms—by covering it with a solution of sodium fluoride.—A. B. S.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steel plates; Action of caustic liquors on* — C. E. Stromeyer, Annual Report of the Chief Engineer to the Manchester Steam Users' Assoc., 1916–1917. Chem. Trade J., Dec. 22, 1917, 533.

ATTEMPTS by Heyn and by Parr to reproduce the author's earlier results on this subject have been unsuccessful. The author records further recent observations. Evaporator tubes exposed to caustic soda liquors showed cracks where the steel had been stressed in tension but none where it had been under compression. This was confirmed by experiments in which pairs of rings were cut out of solid plate, one with taper outer edge and the other with taper inner edge so that it could be hammered over the former to leave the two concentric. The inner one was then in compression and the outer in tension. It was found that after exposure to caustic soda varying up to 4 months, the rings which had been in tension, whether annealed or not, developed numerous cracks on deformation, while those which had been in compression could be bent double without cracking. Further experiments in which it was intended to apply the stresses to steel in the form of a stressed spring proved abortive, because the metal on exposure to caustic soda lost its elasticity. The metal had not been maintained in the stressed condition during the test and afterwards it was found to have retained its ductility. Furthermore it had become extremely difficult to work either by sawing or milling. The loss of elasticity makes it difficult to carry out experiments under tension but in practice this may be maintained by steam pressure in boilers. The author approves of Parr's suggestion that the steel absorbs hydrogen when in contact with caustic soda and so acquires brittleness, but this is not yet clearly established.—H. J. H.

*Electrolytic zinc; Future of* — T. French. Amer. Electrochem. Soc., Oct., 1917. Met. and Chem. Eng., 1917, 17, 647–649.

THE author compares the prospects of retort smelting of zinc with those of electro-deposition in view of the rapid development of the latter in recent years. The yields obtained by electrolysis are very variable, but 90% is sometimes attained. Electro-zinc is usually a by-product of copper or lead smelting where the ores are zinciferous. In such cases the zinc is removed to facilitate the recovery of the principal metals and as the price of the zinc is not a prime consideration to the smelter, it will continue to compete more and more seriously with the retort product from high-grade zinc ores. The Wedge and Herreshoff furnaces are recommended for roasting complex zinc-lead ores, and a properly roasted product now presents no difficulties in the electrolytic process where current is cheap. The electrolytic process can be worked by less skilled and cheaper labour. Furthermore it is possible to recover precious and other metals at small cost. These aids to the financial economy are absent in the retort process. The presence of manganese is essential in the electrolytic process. In the bath it prevents sulphating and destruction of the anode. Manganese dioxide of marketable

quality is deposited at the anode at practically no cost. Large quantities of complex ores and zinc concentrates which are not available for the retort process are now sold very cheaply or tipped to waste. With cheap power, improvement of ore flotation methods, and electrolytic processes, the author looks forward to the exploitation of these and the ultimate displacement of the retort zinc.—H. J. H.

*Brass pipe; Dezincification of* ——. E. B. Story. *Met. and Chem. Eng.*, 1917, 17, 653—655.

Brass pipe used in water services has been found to deteriorate in a few years so as to become brittle and weak, and finally to fail completely. This is due to dezincification. Samples of the dezincified brass can be easily scraped off the affected portions of pipes and it has been found to contain up to 94.5% Cu when the original alloy was a 60:40 brass. The zones of varying decomposition in the metal can be brought out by etching with an ammoniacal solution of hydrogen peroxide. The author believes that potential differences are set up in brass as a result of strain, impurities, segregation, etc., and in presence of water some of the alloy goes into solution. When the copper ions come into contact with copper, deposition occurs, leaving the zinc in solution, so that ultimately a mass of loose copper accretions replaces the original alloy.—H. J. H.

*Zinc; The sampling and assay of Chinese* ——. F. Browne. *Chem. News*, 1918, 117, 1—2.

Tin from the Yunnan mining district is refined in Hongkong in native foundries. When purchases are made, the Hongkong Government undertakes the analysis and certification of the metal. Every tenth slab is bored with a drilling machine, the borings being melted down under palm oil at a low temperature in an iron ladle. The cleaned sample ingot is filed, 1 gram. of the filings dissolved in hydrochloric acid, and the solution titrated with ferric chloride according to Parry's method, which has proved entirely trustworthy. The London Metal Exchange has decided that the Hongkong Government certificate shall be binding on all purchasers.—W. R. S.

*Blast furnace slags; Viscosity of* ——. A. L. Foell. *Trans. Faraday Society*, Dec., 1916. 33 pages. (See this J., 1916, 1237.)

BLAST-FURNACE processes must be worked at temperatures at which the slag is sufficiently fluid to permit an easy separation of metal but not so high as to cause excessive wear on the furnace linings. The slag has a desulphurising action in which diffusion probably plays an important part, and therefore low viscosity is desirable. Thus the temperature-viscosity relations of the slags in the liquid condition have a decisive influence on the fuel economy and working of the process. Fusibilities in terms of Seger cones, as hitherto employed in the study of slags, are of less importance, as they are not necessarily related to the viscosity of the slag in the liquid state. Furthermore, the conditions of the cone test lead to the oxidation of calcium sulphide to sulphate, which is not without influence on the fusibility of the slag. The viscosity apparatus previously described was modified to deal with slags up to 1600° C. The slag was melted in a graphite crucible which formed the outer rotating cylinder, and was attached to a vertical spindle which could be turned at a measurable speed. An inner cylinder of graphite was suspended so as to dip into the slag, the combination being enclosed by a vertical electric furnace, in which was maintained an atmosphere of nitrogen and carbon monoxide to

correspond with that of the blast-furnace. Temperatures were observed by a Holborn-Kurlbaum optical pyrometer sighted on the surface of the slag. The measurement of viscosity resolved itself into a determination of the speed of rotation necessary to be applied to the outer cylinder to produce a given torque on the inner one. Results are given of observations on eight commercial and two synthetic slags and an artificial diopside: the same results were found whether the temperature was rising or falling during the test. The curves of temperature-viscosity are apparently rectangular hyperbole, while those of the fluidity—i.e., the reciprocal of the viscosity—are rectilinear. At 1500° C. the average viscosity of the eight slags was 301 (water at 20°C. = 1), the extremes being 230 and 480. The cone softening temperature of the latter was found to be 1279° and of the former 1410°, thus emphasising the shortcomings of this method of studying slags. The high viscosity (480) was shown by a slag high in silica and low in alumina. The author suggests that this influence of silica is due to its molecular complexity which has been demonstrated at low temperatures by the researches of the Braggs and which probably persists in the liquid state.—H. J. H.

*Hydrolysis of organic halides and the corrosion of metals*. Doughty. See X.

#### PATENTS.

*Furnace for steel melting; Electric* ——. S. Steinberg and I. Gramolin, Motowilichi, Russia. *Eng. Pat.* 111,679, June 27, 1916. (Appl. No. 9055 of 1916.)

IN an electric resistance furnace for the continuous treatment of steel, the carbon resistance rods extending across the heating chamber abut against block contacts, the latter being held by hollow, movable, metallic sleeves within which water is circulated. Spring and screw devices are provided for pressing the sleeves upon the block contacts and thus causing the latter to press upon and support the resistance rods.—W. E. F. P.

*Steel; Process of treating — to prevent rusting*. R. L. Gooding, Bridgetown, Barbados, U.S. *Pat.* 1,247,668, Nov. 27, 1917. Date of appl., Apr. 14, 1916.

THE steel is subjected to the action of strong phosphoric acid, and finely powdered iron is applied to the treated surface. The treatment is repeated at intervals and finally a thin coat of oil is applied.—T. H. B.

*Furnace; Metallurgical* ——. U. Wedge, Ardmore, Pa., U.S.A. *Eng. Pat.* 111,580, Jan. 25, 1917. (Appl. No. 1295 of 1917.)

IN a furnace consisting of a series of superposed hearths supported by rings attached to a central shaft, the hearths and rings are composed of separable segments which may be removed laterally through an opening in the outer wall which is normally closed by a movable section. The rabble blades have enlarged heads and project vertically through sockets in the horizontal supporting members, the upper part of each socket being enlarged for the reception of the blade head.—W. E. F. P.

*Metals; Method of hardening* ——. H. Scholey, London. *Eng. Pat.* 111,734. Dec. 22, 1916. (Appl. No. 18,380 of 1916.)

THE metal to be hardened is mounted as one of the electrodes, preferably the cathode, in an electrolytic bath (e.g., dilute sulphuric acid), and an electric current is passed through the liquid until the immersed portion of the metal has attained the necessary temperature. The current is then cut off and the metal allowed to cool while immersed.—W. E. F. P.



*Metal-tempering compound.* A. S. Carlson, Denver, Colo. U.S. Pat. 1,246,802, Nov. 13, 1917. Date of appl., Feb. 3, 1917.

A MIXTURE suitable for use in tempering metals is composed of approximately 10 galls. of kerosene and 1 gall. of creosote.—B. N.

*Metal wire and strip; Electrolytic production of* —. S. O. Cowper-Coles, Sunbury-on-Thames, Eng. Pat. 111,757, Jan. 31, 1917. (Appl. No. 1555 of 1917.)

THE metal is deposited on the disc-shaped, exposed surface of a cathode plate, the latter having a spiral engraved upon it and being provided with a central hole. The deposit is then detached from the plate and mounted on a spindle passed through the central hole, when, owing to the presence of weak lines occasioned by the spiral groove, the metal can be drawn off in the form of an approximately square wire or tape. The parts of the cathode plate upon which deposition is not required are provided with insulating plates or coated with insulating varnish.—W. E. F. P.

*Electroplating.* H. N. Gilbert, Brookline, Mass., Assignor to American Optical Co., Southbridge, Mass. U.S. Pat. 1,246,083, Nov. 13, 1917. Date of appl., Apr. 24, 1915.

A STATIONARY tank of non-conducting material is provided with a peripheral flange or shoulder near its upper end, a ring resting on the shoulder, and a perforated container of non-conducting material is fitted centrally within the ring. The container is suspended by a shoulder extending outwards and interlocking with the ring. A rotatable non-conducting stirrer, supported by a cap plate, is provided below with exposed cathode contacts, located within and near to the bottom of the perforated receptacle for making contact with the articles to be plated.—B. N.

[Copper] alloys; *Process of making metallic* —. H. Eldridge, New York. U.S. Pat. 1,246,571, Nov. 13, 1917. Date of appl., Dec. 22, 1916.

COPPER is alloyed with more fusible metals, e.g., zinc, lead, etc., by melting the copper in one vessel, and conveying the vapour of the alloying metal from a separate chamber so as to bring it into contact with the molten copper.—T. H. B.

*Cadmium; Apparatus for removing* — from zinc ores. C. P. Fiske, Palmerton, Pa., Assignor to The New Jersey Zinc Co., New York. U.S. Pat. 1,246,576, Nov. 13, 1917. Date of appl., Aug. 24, 1917.

THE ore charge is agitated in a rotary kiln and is heated by a current of heating gases (products of combustion), supplied directly to the interior of the kiln from a fire-box. A reducing atmosphere is maintained in the kiln by supplying a reducing gas, in regulated quantity, to the upper portion of the fire-box, so that it mingles with the products of combustion. Means are provided for recovering the cadmium expelled from the ore.—T. H. B.

*Ore concentration process.* W. A. Scott, Chicago, Ill. U.S. Pat. 1,246,665, Nov. 13, 1917. Date of appl., Aug. 17, 1917.

A MIXTURE of a vaporized "modifying agent" and air is introduced through a porous medium of fine texture into a body of ore pulp in the form of bubbles. The bubbles rise through the pulp and form a column of froth above the pulp, and the mineral matter which adheres to the bubbles is removed from the upper part of the column of froth.—T. H. B.

*Sintering ores; Method and apparatus for* —. J. Gayley, New York. U.S. Pat. 1,247,661, Nov. 27, 1917. Date of appl., Mar. 30, 1917.

A GRATE-BAR platform for a connected layer of

material is formed by a train of abutting cars; the cars are moved first under an igniter and then over a suction chamber. The cars are dropped at the end of their travel, without changing their upright position, thus breaking off a portion of the sintered material from the mass.—T. H. B.

*Aluminium alloy.* W. A. McAdams, Bayshore, N.Y. U.S. Pat. 1,247,977, Nov. 27, 1917. Date of appl., Apr. 5, 1914. Renewed May 1, 1917.

AN alloy of Al 100 parts by weight, Cu 17 parts, Zn 5 parts, Sb 1.5 parts, Ag 1 part.—T. H. B.

*Etching metals; Method of* —. J. L. Jones, Oakmont, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,248,499, Dec. 4, 1917. Date of appl., June 11, 1915.

THE metal to be etched is immersed in a molten metal, e.g., zinc, of lower melting point, which is capable of alloying with it. The etched metal is withdrawn, adherent material is broken off, and the last traces removed by a suitable solvent, e.g., dilute sulphuric acid.—W. F. F.

*Metals [e.g., copper]; Process of purifying* —. 1. Ladoff, Pittsburgh, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,248,503, Dec. 4, 1917. Date of appl., May 11, 1914.

IN purifying molten metals, e.g., copper, an intimate mixture of copper, or a conglomerate containing copper, and titanous oxide, is added to the molten metal and acts as a gas-removing agent. The resulting slag is removed.—W. F. F.

*Apparatus for separating substances of different specific gravities, such as fine coal or ores and the like.* Eng. Pat. 111,826. See 1.

*Process and apparatus for pulverising and treating ores, clay, and other mineral substances.* U.S. Pat. 1,246,514. See 1.

*Metallic yarn and method of treating same to facilitate weaving and for other purposes.* U.S. Pat. 1,249,252. See V.

*Testing or determining the hardness of metals and other substances.* Eng. Pat. 108,460. See XXI11.

## XI.—ELECTRO-CHEMISTRY.

### PATENTS.

*Furnace; Polyphase electric* —. J. L. Dixon, Detroit, Mich. U.S. Pat. 1,248,632, Dec. 4, 1917. Date of appl., Jan. 20, 1917.

THE crucible of the furnace, containing the material to be treated, is provided with three electrodes connected to a two-phase three-wire system of current supply. The position of the common return electrode, or the one connected to the neutral point, is adjusted by two opposing electromagnets, so that its position is arranged in accordance with the proportion of the current carried relative to the total amount of current entering the furnace.—B. N.

*Insulation; Flame-proof* —. C. Ellis, Montclair, N.J., Assignor to New Jersey Testing Laboratories. U.S. Pats. 1,246,809 and 1,246,810, Nov. 13, 1917. Dates of appl., (A) July 26, 1915; renewed Sept. 14, 1917, (B) July 2, 1917.

(A) AN insulating composition, adapted for use in the coating of wire, is composed of a plastic insulating material incorporated with a non-inflammable chlorinated product of heavy petroleum, e.g., an "asphaltic oil," sufficient of the

latter being present to render the composition fire-proof. (B) A combustible insulating material is incorporated with a resin-like non-inflammable chlorinated wax, the material being sufficiently plastic to be used for the coating of wire.—B. N.

*Insulating composition; Flame-proof*—C. Ellis, Montclair, N.J. U.S. Pat. 1,248,638, Dec. 4, 1917. Date of appl., Aug. 5, 1915.

A READILY plastic and flexible insulating composition is composed of a combustible organic plastic insulating material incorporated with a mixture of non-inflammable chlorinated hydrocarbons of solvent naphtha. Sufficient of the latter must be present to render the composition flame-proof, without materially detracting from the flexibility of the insulated wire to which the composition is applied.—B. N.

*Electrical-resistance material*. C. Ruzicka, Gravesend. U.S. Pat. 1,246,165, Nov. 13, 1917. Date of appl., May 16, 1911.

SEE Eng. Pat. 11,975 of 1913; this J., 1914, 650.

[*Electrolytic*] process for the extraction of chlorine. U.S. Pat. 1,246,099. See VII.

*Recovery of alkali [from silicates]*. U.S. Pat. 1,246,619. See VII.

*Depositing clay, plumbago and other like products on absorbent moulds. Manufacture of clay, plumbago, and other like articles by electrodeposition on metal moulds*. Eng. Pats. 111,762 and 111,775. See VIII.

## XII.—FATS; OILS; WAXES.

*Globe-fish and angler-fish liver oils*. M. Tsujimoto. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 709—711.

GLOBE-FISH liver oil (from *Spherooides porphyreus*) is a yellow liquid, which has a characteristic odour and deposits "stearine" at the ordinary temperature. It gives a red coloration, with purplish shade, on treatment with sulphuric acid. Angler-fish liver oil (*Lophomus setigerus*) is a yellowish-orange liquid which deposits "stearine," has a peculiar odour, and gives a reddish-purple coloration with sulphuric acid. These oils had the following characters:—

Oil from:	Sp. gr. at 15°/4° C.	Acid value.	Saponif. value.	Iodine value (Wijs).	Refractive index at 20° C.	Unsap. matter.	M. pt. of fatty acids.	Insol. bromide from fatty acids.	Bromine in insol. bromide.
Globe-fish liver .....	0.9269	0.88	182.2	159.8	1.4785	% 1.47	° C. 31—32	% 46.65	% 70.22
Angler-fish liver .....	0.9268	1.1	188.6	154.1	1.4790	1.00	32.5—34	44.13	70.65

The unsaponifiable matter from both oils melted at 100° C., and was free from the hydrocarbon, squalene, present in shark liver oil (see this J., 1916, 609, 1121).—C. A. M.

*Fresh-water fish oils (carp, loach, and trout oils)*. M. Tsujimoto. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 711—720.

THE following characters were possessed by these oils:—

Oil from :	Sp. gr. at 15°/4° C.	Acid value.	Saponif. value.	Iodine value (Wijs).	Refractive index (20° C.)	M. pt. of fatty acids.	Insol. bromide from fatty acids.	Bromine in insol. bromide.
						° C.	%	%
Carp ( <i>Cyprinus carpio</i> ) .....	0.9262	1.1	193.7	137.04	1.4756	29—30	28.29	63.77
Loach ( <i>Misgurnus anguillicaudatus</i> ) ..	0.9212	—	206.1	121.69	1.4740	31—32	25.45	68.01
Trout (a variety of <i>Oncorhynchus nerka</i> )	0.9305	1.8	196.7	176.98	1.4796	22	46.31	68.10
Trout ( <i>Salmo iridens</i> ) .....	0.9229	1.3	198.1	125.90	1.4736	33.5	22.97	68.63

The bromide from carp oil consists mainly of linolenic hexabromide with a small proportion of polybromides. The insoluble bromides from the fatty acids of the other oils behaved in the same way on heating as the bromides from marine animal oils, but contained a smaller proportion of bromine.—C. A. M.

*Pulza-oil [curcas-oil] from the seeds of the physic-nut tree (*Jatropha curcas*)*; *Industrial and economic use of*—Investigation by the Colonial Garden of France. Rev. agric. et vétérin. de Madagascar, 1917, 2, [8], 37—38. Bull. Agric. Intell., 1917, 8, 1303—1304.

THE physic nut is at present obtained almost exclusively from the Cape Verde and Comoro Islands, and before the war about 500—600 tons was imported into France annually. Analysis of samples from Madagascar gave the following results:—1000 seeds weighed 535 grms.; the seeds consisted of 39.2% of shell and 60.8% of kernel, and the kernels contained 7% of moisture and 52% of oil. The oil-content of the whole seeds was 31.6%, corresponding to a commercial yield of 27—28%, since 1—8% of oil would remain in the cakes. Pulza-oil (curcas-oil) is used in soap-making as a substitute for low-grade peanut oil, and it might also be employed in stearin factories. Its low acidity should render possible its application as a lubricant. It may also be used as a purified oil. Before the war the current price of physic-nut seeds was 13s.—20s., but at the present time they should command 40s. per 100 kilos, net at Marseilles.—J. H. L.

*Oil of Câ-y-doc (*Garcinia tonkinensis*)*. F. Heim. Bull. économique de l'Indochine, 1917, 20, [123] (new series), 135—139. Bull. Agric. Intell., 1917, 8, 1304—1305.

THE seeds of the Câ-y-doc or Câ-y-gioe, a tree of the family *Clausiaceae*, have long been the object of an important trade in Tonkin, and are used for the extraction of a burning oil. The oil is a mixture of true fats and oleo-resin containing a small proportion of ethereal oil of agreeable odour. The oleo-resin is probably of no commercial value, as it does not solidify on desiccation or oxidation. The oil has the following constants:—acid value, 93; saponif. value, 198; iodine value, 67.14;

Reichert value, 0.53. Saponified with caustic soda, it furnishes about 90% of fatty acids, brown in colour, melting at 22° C., and composed of a mixture of solid fatty acids (mainly stearic and palmitic) and oleic acid. It is easily saponified and yields a brown coloured soap which, in appearance and to some extent in odour, resembles palm-oil soap. The proportion of essential oil is small (4.58%), and the presence of resin is not harmful but promotes the formation of lather, even in water



containing calcium sulphate. *Garcinia tonkinensis* is grown in Tonkin; it matures in 8—10 years and would yield a full crop of seed only after 20 years. The annual crop of seed per tree should be worth a Tonkin piastre (about 2 shillings).

—J. H. L.

*Oils and fats; Valenta number as a discriminating test for*—. P. J. Fryer and F. E. Weston. Analyst, 1918, 43, 3—20.

THE turbidity temperatures obtained in the ordinary method of applying the Valenta test are greatly affected by the presence of water or free fatty acids in the oils. Acetic acid is extremely hygroscopic and the Valenta results are affected by leaving the bottle unstoppered for a short time before using the acid. In the case of almond oil containing 1.1% of free acids the Valenta value increased about 1.5° C. for each 0.1% of moisture absorbed. Absolute alcohol is less affected by the absorption of moisture, the Valenta value being increased by about 0.75° C. for each 1.1% of added water. A mixture in equal vols. of 90 to 92% alcohol and amyl alcohol absorbs much less water than either acetic acid or alcohol. Thus in the case of the same almond oil the Valenta values before and after exposure of the reagent for 16 hours in an unstoppered bottle were 69.2° and 69.8° C. respectively. To obviate the effect of water in the oil the heated sample may be filtered through dry cotton-wool. For standardising the solvent the use of pure almond oil with an iodine value below 100 is suggested, and the solvent is diluted so as to give a turbidity temperature of about 70° C. with this oil. About 0.11% of water corresponds with a rise of 1° C. in the Valenta figure. The effect of free acids in the oil or fat is to cause a fall in the turbidity temperature, the amount of depression caused by each 1% of acidity (as oleic acid) being as follows:—Marine oils, 1.95°; drying oils, 2.05°; semi-drying oils, 2.03°; non-drying, 2.07°; rape type of oils, 1.61°; vegetable fats (except coconut oil), 1.72°; coconut group, 2.01°; animal fats (except butter fat), 2.13°; and butter and milk fats, 1.54° C. Heating the oil before applying the test has a pronounced influence on the results when acetic acid is used as solvent, but has only a slight effect in the case of the amyl-ethyl alcohol solvent. Slight oxidation of an oil does not materially affect the result, but pronounced oxidation causes pronounced lowering of the temperature. After determining the value of an unknown oil the tube is washed out with hot alcohol, the acidity determined with  $N/10$  alkali solution, and the appropriate correction for the class of oil applied. In the case of acetic acid as solvent the standard temperature with neutral almond oil is fixed at 80° C., and the results may be expressed either as  $V = t + (80 - t_1)$ , where  $t$  is observed temperature and  $t_1$  the temperature of the same acid with the standard oil; or as  $X = \frac{V \times 10}{80}$ , where  $V$  represents the true Valenta figure. By the use of the ethyl-amyl alcohol solvent it is possible to distinguish between animal and vegetable waxes (other than insect wax) and mineral waxes. For example the following values were obtained:—Carnauba wax, 82°; candelilla wax, 63°; beeswax, 76°; spermaceti, 44°; insect wax, insoluble; montan refined wax, 70°; ozokerite, insoluble; paraffin wax, insoluble; beeswax + 10% ceresin, 82°; candelilla wax + 10% paraffin wax, 72°; and candelilla wax + 33% paraffin wax, 83° C.—C. A. M.

*Fats; Relationship between the chemical constitution and refractive index of*—. C. Chénveau. Comptes rend., 1917, 165, 1060—1062.

ASSUMING a fat in the liquid condition to consist of a solution of one or more solid glycerides and one or more liquid glycerides, the law of solutions

would hold good, and the refractive index of the fat could be calculated from the percentage proportions and refractive indices of the component glycerides by means of the formula:—

$$\Sigma p \frac{n-1}{d} + \Sigma p^1 \frac{n^1-1}{d^1} = 100 \frac{N-1}{D}$$

Solid glycerides      Liquid glycerides      Fat

Again, assuming the iodine value,  $I$ , to afford a measure of the unsaturated (liquid) glycerides, then  $p^1 = kI$ , and the law of solution may be expressed as:—

$$N-1 = A + BI,$$

in which  $A$  and  $B$  are two coefficients expressing the relationship between the refractive index and the iodine value of a fat. In the case of non-drying oils the line expressing this relationship graphically is more inclined towards the axis of the abscissæ than in the case of drying and semi-drying oils, but as the temperature rises the two lines tend to become identical.—C. A. M.

*Utilisation of waste tomato seeds and skins.* Rabak. See XIXA.

#### PATENTS.

*"Foots" or soap-stock; Process whereby neutral oils can be profitably recovered from*—. E. E. Ayres, jun., Chester, Pa., Assignor to The Sharples Specialty Co., West Chester, Pa. U.S. Pat. 1,247,782, Nov. 27, 1917. Date of appl., May 21, 1917.

THE "foot" is treated with an amount of water equal to at least half its weight together with a reagent capable of reducing the stability of the emulsion. The whole is then heated, allowed to settle, the bulk of the soap solution separated from the bulk of the oil, the latter centrifuged, and the separated emulsified oil treated with salt. The oil is then separated from the salted product by gravity.—A. de W.

*Saponifiable oils; Process of refining*—. C. Baskerville, New York, Assignor to The Baskerville Processes Corporation. U.S. Pat. 1,246,379, Nov. 13, 1917. Date of appl., Aug. 2, 1915.

SEE Eng. Pat. 101,097 of 1916; this J., 1917, 1054.

*Soap and candle and process of making the same.* B. E. Reuter, Chicago, Ill. U.S. Pat. 1,248,402, Nov. 27, 1917. Date of appl., Apr. 9, 1914.

SEE Eng. Pat. 9394 of 1915; this J., 1916, 1163.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Colour-technology; The physical basis of*—. M. Luckiesh. Met. and Chem. Eng., 1917, 17, 631—637.

THE author deals in a general way with the value of an analytical method of considering technical problems in colour. (For a detailed statement of apparatus, methods, and data, see "Colour and its applications," 1915, D. Van Nostrand Co., and for additional spectro-photometric data, see Luckiesh, J. Franklin Inst., 1917, 184, 73, and 227.) The first factor to consider is the colour value of the illuminant used which should be considered as almost a part of the coloured medium. Curves showing the spectral reflection factors of ultra-marine blue and its spectral luminosity curves for skylight, sunlight, and a tungsten vacuum lamp illustrate the point. There are three main methods in use for the analysis of colour. The most analytical is by means of the spectro-photometer, which determines the reflection or transmission factors of the colour for all wave lengths of the

visible spectrum. By combining these factors with the corresponding spectral values of the illuminant and the visibilities of radiation, the spectral luminosity curve is obtained. A series of curves for chrome yellow illustrate this method. The monochromatic colorimeter expresses the value of a colour in terms of its dominant hue, its percentage of saturation (= unity minus percentage white), and a transmission or reflection factor; chrome yellow has a dominant hue,  $0.583\mu$ , a saturation of 75%, or 25% white, and a reflection factor, as determined by the spectro-photometer analysis, of 82%. The trichromatic colorimeter is based on the Young-Helmholtz theory; chrome yellow by this method is expressed as red 58, green 37, blue 5, as compared with a white standard of 33.3 for each component. In considering pigments the reflection factor curves are given for a number of dry powders. Further sections deal with dyes and coloured glasses and with the laws of combination and of variation of colour with thickness for both of these. Numerous suggestions are made throughout the paper as to applications of the results already obtained and as to further information required.—B. V. S.

## PATENTS.

*Paint or composition and process of manufacturing the same.* G. Peacock, Gravesend. Eng. Pat. 111,507, Nov. 24, 1916. (Appl. No. 16,853 of 1916.)

A COMPOSITION for use in screening glass, etc., is composed of size (7 lb.), dark green or other dry pigment (5 lb.), whitening (2 lb.), and potassium bichromate ( $1\frac{1}{2}$  oz.); it is prepared by stirring the pigment and whitening into the melted size, adding the bichromate dissolved in water, and thoroughly mixing.—W. E. F. P.

*Paint. Submarine paint.* L. S. Tarrant, Hawera, New Zealand. U.S. Pats. (A) 1,247,900 and (B) 1,247,901, Nov. 27, 1917. Date of appl., Jan. 12, 1916.

(A) A PAINT composition comprises defibrinated animal blood, raw linseed oil, driers, and pigment. (B) A submarine paint comprises defibrinated animal blood, linseed oil, resin, and pigment.

—A. DE W.

*Pigment; Process of making—and product thereof.* P. J. Oakes, New York. U.S. Pat. 1,248,355, Nov. 27, 1917. Date of appl., Dec. 10, 1913.

A HOMOGENEOUS amorphous neutral pigment is obtained by precipitating an acid solution containing a colour lake, e.g., from logwood, including an oxidised colour principle, with an alkaline solution of a resin. The mass is then boiled, and the pigment separated and dried.—A. DE W.

*Lead sulphate pigment: Process of making—* R. E. Kroher, Collinsville, Ill., Assignor to St. Louis Smelting and Refining Co., St. Louis, Mo. U.S. Pat. 1,248,591, Dec. 4, 1917. Date of appl., Apr. 23, 1917.

WHITE lead pigment is made by heating a mixture of lead-bearing materials, metallic lead in the form of flakes, a flux, and fuel, in a blast furnace. The vapour produced is condensed and the pig lead is tapped off and poured into a stream of water to flake it for subsequent use.—W. F. F.

*Resin and like material: Apparatus for melting—and filling receptacles therewith.* J. W. Westwood, sen., and J. W. Westwood, jun., Birmingham. Eng. Pat. 111,905, Dec. 14, 1916. (Appl. No. 17,984 of 1916.)

AN apparatus for melting resin and discharging it into receptacles, specially adapted for filling shrapnel shells, consists of a melting tank heated by superheated water, and a support for

the tank to which is fitted a filling platform adjustable vertically. The melting tank is fitted with one or more discharge taps, of which the portion bearing the valve projects into the tank.—A. DE W.

*Hydrogenated resin; Process of making—* C. Ellis, Montclair, N.J. U.S. Pat. 1,249,050, Dec. 4, 1917. Date of appl., Feb. 2, 1916.

THE tackiness of rosin is reduced by treating a solution of the same in a hydrocarbon inert to hydrogen, with arsenic-free hydrogen at about 10 lb. pressure in the presence of a nickel catalyst at a temperature above that of the decomposition of nickel abietate.—A. DE W.

*Impregnating process.* A. L. Brown, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,248,447, Dec. 4, 1917. Date of appl., Dec. 10, 1914.

ABSORBENT material is impregnated by successively applying to it a polymerisable liquid, such as a raw drying oil, and a polymerising agent such as a drier.—A. DE W.

*Manufacture of colouring matters [from residues from manufacture of trinitrotoluene].* Eng. Pat. 111,738. Sec IV.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## PATENTS.

*Rubber and other products; Manufacture of—* from latices and the treatment of raw, inferior or scrap rubbers and rubber gums. R. B. Ransford, London. From C. A. Hecken and St. V. B. Down, Singapore. Eng. Pat. 111,906, Dec. 14, 1916. (Appl. No. 17,991 of 1916.)

LATICES, inferior or imperfectly coagulated rubbers, raw rubbers, tacky rubbers, etc., are treated in the cold with a solution of asphalt, bitumen, pitch, tar, or similar material in a suitable solvent, by which treatment the rubber is rendered harder and stronger, and impervious to bacteria or moulds. In treating latices the addition of the asphalt solution is followed by treatment with a suitable coagulant, whereas with the various grades of rubber the application of the solution is merely superficial. In either case the treatment may be combined with the processes described in Eng. Pats. 8487 of 1915 and 110,566 and 111,907 (this J., 1916, 854; 1917, 1280, and following).—D. F. T.

*Rubbers; Process for purifying raw inferior or scrap—* R. B. Ransford, London. From C. A. Hecken and St. V. B. Down, Singapore. Eng. Pat. 111,907, Dec. 14, 1916. (Appl. No. 17,992 of 1916.)

INFERIOR, dirty, or imperfectly cured rubbers or scrap rubbers are treated with an aqueous solution containing 1% to 3% of a salt such as a sulphite, bisulphite, or hydrosulphite, which by the addition of a small amount of sulphuric acid can be made to yield sulphur dioxide. The rubber, thus treated, is preferably soaked or sprayed subsequently with a mixture of methylated spirit and benzine, the purified rubber then being ready for blocking or creping and drying.—D. F. T.

*Vulcanised rubber composition and method of preparing same.* H. A. Gardner, Washington, D.C. U.S. Pat. 1,247,257, Nov. 20, 1917. Date of appl., July 31, 1917.

A COMPOUND of high tensile strength and toughness is obtained by incorporating a finely divided insoluble zirconium compound with rubber and then vulcanising.—B. V. S.



*Vulcanisable plastics; Process for regulating treatment of* —. E. E. A. G. Meyer, Detroit, Mich., Assignor to Morgan and Wright. U.S. Pat. 1,247,983, Nov. 27, 1917. Date of appl., Jan. 31, 1917.

THE plastic is placed in a mould and a heating medium at a vulcanising temperature is applied in such a way that the rate of expansion of the plastic in the mould may be regulated.—D. F. T.

*Cumaron-rubber product; Vulcanised — and process of making same.* A. A. Wells, Caldwell, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,248,226, Nov. 27, 1917. Date of appl., Apr. 25, 1917.

A MIXING containing natural and reclaimed rubber together with coumarone-resin and various fillers, is vulcanised.—D. F. T.

*Caoutchouc; Treating products resembling* —. E. Münch, Assignor to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,248,888, Dec. 4, 1917. Date of appl., Feb. 26, 1914.

RUBBER-LIKE polymerisation products of butadiene are heated with a mixture of solid caustic alkali and an alkali carbonate, the material being thereby improved; it may be subsequently vulcanised. (See also Fr. Pat. 463,437 of 1913; this J., 1914, 365.)—D. F. T.

*Vulcanising rubber; Process for — and products obtained thereby.* I. Ostromislenski, Petrograd, Assignor to New York Belting and Packing Co., New York. U.S. Pats. (A) 1,249,180 and (B) 1,249,181, Dec. 4, 1917. Dates of appl., July 28 and Nov. 24, 1916.

(A) RUBBER is vulcanised by mixing with a compound containing nitrogen and oxygen, e.g., trinitrobenzene, which is capable of yielding oxygen to the rubber; on heating, the oxygen induces vulcanisation. (B) Vulcanisation of rubber is effected by the action of a peroxy-compound such as benzoyl peroxide.—D. F. T.

*Vulcanising rubber with selenium or its compounds; Process for* —. C. R. Boggs, Arlington Heights, Mass., Assignor to Simplex Wire and Cable Co., Boston, Mass. U.S. Pat. 1,249,272, Dec. 4, 1917. Date of appl., Mar. 16, 1916.

A QUANTITY of selenium or selenium compound is incorporated with the rubber, together with an artificial accelerator such as  $\beta$ -naphthylamine, and the mixture is then vulcanised by heat.—D. F. T.

*Rubber; Process for the complete removal of — from the canvas of worn-out pneumatic tyres.* H. Debaugé, Paris. U.S. Pat. 1,248,463, Dec. 4, 1917. Date of appl., July 20, 1914.

SEE Eng. Pat. 100,961 of 1916; this J., 1917, 1019.

## XV.—LEATHER; BONE; HORN; GLUE.

*Sulphide; Estimation of — in [tannery] lime liquors. Part 2.* H. G. Bennett, J. Soc. Leather Trades Chem., 1917, 2—S. J. Amer. Leather Chem. Assoc., 1917, 12, 626—632.

It is maintained against McCandlish and Wilson that a certain amount of ammonia is necessary in the zinc sulphate and ammonium chloride reagent used for titrating sodium sulphide in lime liquors. Precipitation of zinc hydroxide and escape of hydrogen sulphide are thereby prevented, and though an error is caused by the retention of zinc-ammonium ions in solution, it is negligible in the titration of ordinary lime liquors if the  $N/10$  zinc sulphate solution contains 5% of ammonium chloride and 25 c.c. per litre of concentrated ammonia. Experiments are described in support of the above.—F. C. T.

*Sulphide; Estimation of — in [tannery] lime liquors.* D. McCandlish and J. A. Wilson. J. Amer. Leather Chem. Assoc., 1917, 12, 633—634.

REPETITIONS of Bennett's experiments (see preceding abstract) do not support his contentions. A pure lime-sulphide solution of which 50 c.c. required about 6 c.c. of reagent, was titrated with  $N/10$  solutions of zinc sulphate containing 5% of ammonium chloride and amounts of ammonia varying from 0 to 30 c.c. of concentrated ammonia solution per litre. In no case (even in the absence of ammonia) was there any odour of hydrogen sulphide. On the other hand, with a liquor containing 0.5% of sodium sulphide there was a distinct evolution of the gas.—F. C. T.

*Preparation of tannic acid, gallic acid, and pyrogallol.* Milo. See XX.

### PATENT.

*Leather; Process of producing artificial* —. C. E. Arnold, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,247,610, Nov. 27, 1917. Date of appl., Sept. 3, 1914. Renewed May 16, 1917.

A COATING of a nitrocellulose cementing composition is applied to a fabric and a coating of a nitrocellulose plastic is then applied by means of calender rolls before the first coating has dried.

—J. F. B.

## XVI.—SOILS; FERTILISERS.

*Rarer elements in soils and plants; Relation of some of the* —. W. O. Robinson, L. A. Steinkoenig, and C. F. Miller. U.S. Dept. of Agriculture, Bull. 600, 1917. 25 pages.

THE results of the analyses of the ashes of a large number of plants, bushes, and trees showed that lithium was present in all the plants examined. Rubidium was present in most cases and in larger quantities than the other rare alkalis, caesium being present in only a few cases. These two elements are apparently absorbed by the plant when present in the soil solution, the amount absorbed increasing with the amount present in the soil. Chromium and vanadium were only found occasionally and then only in traces, and there was no indication that vanadium could replace phosphorus in its functions in the plant. Molybdenum was never detected in the plants. Barium and strontium were found in all cases and titanium was invariably present though in very small amounts. There is no indication that these rare elements need be considered in fertilising practice. In an appendix a full description is given of an electric furnace, with an automatic regulator, suitable for incinerating plants at a comparatively low temperature (525°C.) in order to avoid loss of alkali by volatilisation.—W. G.

*Peaty soils; Hydrogen ion concentration of extracts of — and of plants which form peat and humus.* H. Kappen and M. Zapfe. Landw. Versuchs-Stat., 1917, 90, 321—374.

ALTHOUGH aqueous extracts of peaty soils have a marked conductivity and contain organic and inorganic substances, the hydrogen ion concentration only slightly exceeds that corresponding with the neutral point, so that soluble acids can be present in traces only. The power of these soils to decompose neutral salts is probably due to an exchange of ions rather than the liberation of free acid. In the case of peat-forming plants, the hydrogen ion concentration of their aqueous extracts is usually higher than corresponds with the neutral point, and the true acidity is marked.

—W. P. S.

*Plants; Action of dicyanodiamide on the growth of* —. T. Pfeiffer and W. Simmermacher. Landw. Versuchs-Stat., 1917, 90, 415—430.

WHILST the germination of oats is not affected by the presence of considerable quantities of dicyanodiamide in the soil, the growth of the plants and the yield of grain are affected injuriously. The quantity of dicyanodiamide usually found in nitrolim (crude calcium cyanamide) is, however, quite small, but the substance should be considered as an impurity which may possibly have an injurious effect on the crops.—W. P. S.

#### PATENTS.

*Calcium cyanamide; Manufacture of crude* —. Elektrizitätswerk Lonza, Gampel and Basle, Switzerland. Eng. Pat. 107,751, Dec. 21, 1916. (Appl. No. 18,326 of 1916.) Under Int. Conv., July 6, 1916.

IN the manufacture of crude calcium cyanamide from calcium carbide, an inclined, cylindrical, rotary furnace is employed through which nitrogen is circulated, the temperature of reaction being regulated by heating or cooling the gas as required. The furnace has an extension at each end, and forms part of a closed system provided with a fan and with separate heating, cooling, and dust chambers. The extension at the lower end, through which the nitrogen is admitted, forms a cooling chamber for the crude cyanamide and is connected with a sealed discharge receptacle. The nitrogen exit pipe of the furnace is disposed co-axially within the upper extension, through which the carbide is fed, the annular space between the two thus forming a chamber which is not traversed by the circulating gas, so that excessive "dusting" of the charge is avoided.—W. E. F. P.

*Fertiliser; Process of making* —. J. E. Zilk, Assignor to Odorless Fertilizer Manufacturing Co., Pittsburgh, Pa. U.S. Pat. 1,247,059, Nov. 20, 1917. Date of appl., Feb. 10, 1917.

A MIXTURE of phosphate rock, 2000 lb., coke, 200 to 300 lb., powdered limestone, 200 to 300 lb., and nitre cake, 400 lb., is heated to incipient clinkering.—B. V. S.

*Process of recovering ammonia and potassium-containing material from tobacco waste.* U.S. Pat. 1,247,060. See VII.

### XVII.—SUGARS; STARCHES; GUMS.

*Inulin; Comparative examination of pure and commercial* —. J. Wolff and B. Geslin. Ann. Chim. Analyt., 1917, 22, 241—242.

FERMENTATION experiments with a Burgundy yeast, Annanite yeast (see Will, this J., 1913, 1123), and *S. Pombé*, showed that contrary to the statements of some authors, pure inulin is quite unfermentable. A sample of commercial inulin prepared by Dragendorff's method was attacked strongly by *S. Pombé* and to a smaller extent by the other yeasts. It is concluded that inulin prepared by Dragendorff's method contains inulides (see this J., 1917, 1286).—J. H. L.

*Distillation of cellulose and starch in vacuo.* Pietet and Sarasin. See IIB.

*Polarimetric determination of starch in grain, milling products, etc.* Hals and Heggenhougen. See XIXA.

#### PATENTS.

*Syrup evaporator.* P. A. Yoder, Washington, D.C. U.S. Pat. 1,248,237, Nov. 27, 1917. Date of appl., Apr. 3, 1916. (Dedicated to the public.)

A SYRUP evaporator comprises a vat provided

with suitable supports and having an impervious floor consisting of a number of tubes fastened securely together, heads secured to the open ends of the tubes, baffle-plates adjusted in the vat, and troughs arranged along the sides thereof, the tubes and heads providing a passage-way for heating medium for heating the syrup.—J. F. B.

*Vegetable glue or adhesive [from starch].* R. W. Tunnell, Philadelphia, Pa. U.S. Pat. 1,248,039, Nov. 27, 1917. Date of appl., May 3, 1917.

HYDROLYSED starch or a derivative is treated with an alkali aluminate or with alum in excess of caustic soda to form sodium aluminate in the hydrolysed starch.—J. F. B.

### XVIII.—FERMENTATION INDUSTRIES.

*Yeast; Contribution to the knowledge of* —. J. J. Van Hest. Woch. Brau., 1917, 34, 327—328, 341—343. (See also this J., 1904, 30, 831, 945.)

COMPARATIVE laboratory fermentations of sterilised malt wort by a mixture of four top-fermentation yeasts were carried out under different conditions, viz., (A) with continuous rousing by means of a current of sterile air, (B) with the wort covered by a layer of olive oil to exclude air, and (C) under ordinary conditions. In each case 4 c.c. of pressed yeast per litre was used, and the fermentations were carried out to completion at 17.5° C. The wort nitrogen assimilated by the yeast was 0.4, 0.28, and 0.36 gm. per litre in the respective cases, and the amounts of yeast produced were respectively 16.4, 8.2, and 8.8 times that used for seeding. The crop produced by aeration was thus much larger than the others but its nitrogen content was much lower; the individual cells were also about 25% smaller, so that the aerated yeast contained only about half as much nitrogen per cell as that formed under ordinary conditions (C). The nitrogen-content of the aerated yeast, referred to dry substance, was 6.3%, practically the same as that of colonies grown under aerobic conditions on the surface of wort-gelatin (the seeding of the latter having been effected by pouring a yeast suspension over its surface). As the multiplication of the yeast in the colonies was incomparably greater than in the wort, the author considers that 6% represents about the minimum nitrogen-content possible for the yeast. The maximum possible nitrogen-content is probably about 10%, this being the value for the seed-yeast and also for the crop formed under normal conditions (C). To study the effect of prolonged anaerobic growth on the yeast, 13 successive fermentations were carried out under the same conditions as in (B), each fresh lot of wort being seeded with a small quantity of sedimentary yeast from the preceding one and fermented for 5 days at 17.5° C. The results are given in tables. The action of the yeast was irregular, but on the whole the later fermentations proceeded as far as the earlier ones. However, the yeast, even from the earlier fermentations, contained a high proportion of dead cells and abnormal forms indicating degeneration. The author concludes that it would be inadvisable to use a top-fermentation yeast in brewing, after even one fermentation under anaerobic conditions. Copious aeration promotes the biological activities of yeast at the expense of fermentative action, whilst lack of air, or the use of large proportions of seed yeast, promotes fermentative activity at the expense of reproduction.—J. H. L.

*Auxoamylases; Some nitrogenous* —. E. W. Rockwood. J. Amer. Chem. Soc., 1917, 39, 2745—2752.

$\alpha$ -AMINO-ACIDS, whether aliphatic or cyclic,



increase the hydrolytic activity of saliva on starch and are called auxoamylases. In the cyclic compounds the effect is the same whether the amino group is in the benzene ring or the side chain, and is independent of its position in the ring. Amides are not auxoamylases and the introduction of an amino group into the carboxyl group destroys the effect of an amino group already present elsewhere in the molecule. Imides and aminosulphonic acids are not auxoamylases, but the replacement of a hydrogen in the amino group of an amino-acid by an acyl group does not destroy its stimulating effect. Proteins act as auxoamylases, their action being increased as the number of free amino groups is increased by the hydrolysis of the protein. Similar results are obtained with pancreatic amylase as with the ptyalin.—W. G.

*Bran as a new raw material for cereal beverages.*

R. Wahl. Amer. Brewers' Review, 1917, 31, 323—324.

In experiments at the Wahl-Henius Institute it was found that, contrary to the opinion of many authorities, the aleurone layer of seeds generally, including those of all the cereals examined, contains fairly large quantities of diastase. Wheat-bran, for example, will under suitable conditions convert 5—8 times its weight of gelatinised starch, and as it is rich in proteins and mineral constituents it is proposed to use it, together with starchy materials, for the manufacture of alcoholic beverages and for kindred purposes (see Eng. Pat. 101,406: this J., 1917, 936, and U.S. Pat. 1,249,259, p. 69A). The nitrogenous and mineral constituents may be rendered soluble by mashing at a suitable temperature in presence of a sufficiently high acidity produced either by addition of lactic acid or by development of lactic bacteria. Using wheat bran and gelatinised cereal starch, and modifying somewhat the ordinary methods of operation, it was found possible to produce pale and dark bottom-fermentation beers, products of the character of ales and stouts, and also syrups resembling malt extracts and having a stimulating action on yeast in baking. The diastase of bran (translocation diastase) is believed to have a somewhat lower optimum temperature of saccharification (45°—50° C.) than that of malt diastase (50°—60° C.), and in using the former it is possible so to control the mashing process as to produce a wort containing less sugar than ordinary brewers' wort.—J. H. L.

*Alcoholic ferments; Study of — [in apple must].*

E. Kayser. Comptes rend., 1917, 165, 1020—1022. (See also this J., 1917, 608, 902.)

THE author investigated the products of the slow fermentation of sterilised apple must by a wine yeast, a cider yeast, and a perry yeast, and also in each case the effect of adding ammonium phosphate, equivalent to 1 gm. of nitrogen per litre, before fermentation. In each case the must (of sugar-content 11.4% and acidity 0.616% as malic acid), contained in a long-necked flask filled to the neck so as to expose only a very small surface to the air, was inoculated with a drop of yeast culture, and kept at 5°—18° C. for a year, after which analyses were made of the fermented must and the spirit (*eau-de-vie*) obtained from it. The results are given in tables. In the musts which had not been treated with phosphate, the alcohol-content ranged from 6.85 to 6.93%, the total acidity (as malic acid) from 5.84 to 7 grms. per litre, and the volatile acidity (in terms of acetic acid), from 0.05 gm. (for the perry yeast) to 0.368 gm. (for the cider yeast) per litre. The volatile acidity consisted of acetic and butyric acids with traces of formic acid, and was in most cases lowered as the result of the addition of

phosphate, referred to above. The distillates from the musts not treated with phosphate contained in all cases large amounts of aldehydes (323—394 mgrms. per 100 c.c. of alcohol) and higher alcohols (from 167 mgrms. for the cider yeast, to 364 mgrms. for the wine yeast); the phosphate treatment reduced these amounts considerably, especially in the case of the wine yeast. The ester-content of the distillates was in all cases relatively small, ranging from 43 to 67 mgrms. per 100 c.c. of alcohol.—J. H. L.

*Wines; Constitution of the non-volatile acidity of sound and sick —.* J. Laborde. Comptes rend., 1917, 165, 1017—1020. (See also this J., 1918, 36 A.)

THE author divides the non-volatile acidity into two parts: that soluble in a mixture of alcohol and ether, due to lactic, succinic, malic, and citric acids, and that insoluble, due mainly to acid potassium tartrate. Analysing six French and Algerian wines, he was able to account for nearly the whole of the non-volatile acidity by determining the amounts of the various acids present and subtracting the alkalinity of the ash, everything being expressed in terms of sulphuric acid. The actual amounts of the various acids found were, in grms. per litre: lactic acid 0.84—1.90, succinic acid 0.61—0.75, malic acid 0.45—4.26, tartaric acid 1.66—3.47, and citric acid (in the two Algerian wines, to which it had probably been added) 1.10—1.66. Sound wines may thus contain considerable amounts of lactic acid. Analyses of some sick wines are also given. In one sample which underwent de-acidification the malic acid diminished from 4.99 to 1.20 grms. per litre in six months, whilst the lactic acid increased from 1.05 to 2.74 grms. and the tartaric acid remained constant. Two wines suffering from *tourne* were found to contain no tartaric acid, 0.36—0.67 gm. of malic acid, and 2.24—2.40 grms. of lactic acid per litre, and the volatile acidity was high. Two bitter wines contained 1.14—1.97 grms. of tartaric acid, 1.08—1.20 grms. of malic acid, and 3.06—3.68 grms. of lactic acid. In all these wines the succinic acid was remarkably constant at about 0.6—0.8 gm. per litre. The amounts of malic and tartaric acids vary according to the origin of a wine and the influence of filiform organisms such as those which produce *tourne* and bitterness; the former acid appears to be more readily decomposed than the latter by these organisms.—J. H. L.

*Wines; Bitter —.* E. Kayser. Rev. Viti-culture, 1917, 47, 70—75. Bull. Agric. Intell., 1917, 8, 1295—1297.

ANALYSIS of three French wines suffering in different degrees from "bitterness" indicates that the disease is associated with a reduction in the content of fixed acids and of total tartaric acid but not in that of volatile acids; the amounts of both volatile and non-volatile esters also increase whilst the glycerol-content diminishes (cp. Voisinot, this J., 1910, 1126; 1911, 1026; 1913, 503, 620). The volatile acids and esters were investigated by the method of Duclaux; acetic and butyric acids were found to be present, and in one case formic acid. The two samples most strongly attacked contained 0.0267 and 0.0187 gm. of ammonia per litre, as compared with 0.006 gm. in the third sample which was only slightly bitter.—J. H. L.

*Saké; Pasteurisation (hiire) of — by electric heating.* T. Tadokoro, F. Ibuki, and S. Iguchi. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 860—872.

THE pasteurisation of saké by the ordinary method often involves exposure to air infection and is attended by a loss of saké of about 2%, owing to the operation being carried out on small quantities

at a time; there is also a loss of alcohol by volatilisation. The authors have designed an electrical heating apparatus which should show a considerable economy and an improved product. The saké is heated for 2½ to 5 hours at 55°–60° C. The consumption of electric power is about 0.06–0.07 kilowatt per litre, showing an average efficiency of 83.4%.—J. F. B.

*Methyl alcohol; Detection of — in alcoholic beverages and its formation by the several kinds of yeasts.* T. Takahashi, M. Gunke, and T. Yamazaki. J. Amer. Chem. Soc., 1917, 39, 2723–2726.

In the distillates of the beverages examined, namely, wines, whisky, "saké," spirits, and beer, no formaldehyde could be found directly by distillation below 80° C., and after oxidation of the distillates with permanganate and sulphuric acid (this J., 1915, 812), differences were found in the quantities of formaldehyde from methyl alcohol present in the various beverages. The smallest amount of methyl alcohol was found in "saké" except in one sample which contained methyl lactate. In some cases it is necessary to take large samples and concentrate by two or more distillations. All the yeasts tested, "saké", beer-, wine-, and distillery-yeasts, formed methyl alcohol in saccharine fluids (koji extract, raisin extract, Hayduck's solution), the quantity formed being increased by the addition of glycine.—W. G.

*Polarimetric determination of starch in grain, milling products, etc.* Hals and Heggenhougen. See XIXA.

*Comparative examination of pure and commercial inulin.* Wolff and Geslin. See XVII.

#### PATENT.

*Beverage; Maltless, non-alcoholic — and process of producing the same.* R. Wahl, Chicago, Ill. U.S. Pat. 1,249,259, Dec. 4, 1917. Date of appl., Mar. 9, 1916.

A MASH is made of a material which is devoid of germination diastase, but contains translocation diastase, for instance bran, which is mashed with water and lactic liquor of an acidity of about 2%, containing active lactic acid bacteria. The temperature is maintained favourable to the activity of the bacteria; gelatinised starch is added and the temperature raised to a point favourable to the conversion of the starch. After mashing, the wort is drawn off, boiled with the addition of hops and yeast cells, filtered, and carbonated.—J. F. B.

#### XIXA.—FOODS.

*Milk; Reductase test for —.* P. S. Arup. Analyst, 1918, 43, 20–31.

In the modified reductase test as used by Barthel and Orla-Jensen (Milchwirtschaft. Zentralbl., 1912, 14), 40 c.c. of the milk is heated with 1 c.c. of methylene-blue solution, in a covered tube in a water-bath at 38° to 39° C., and the time required for complete decolorisation is noted. As the reducing power of the micro-organisms depends not only on their number, but also on their vitality and capacity for growth (cf. Fred, Analyst, 1913, 38, 62), the author uses a solution containing only 0.015% of methylene blue (1 in 270,000 of milk) so as to reduce the time of action and minimise the possible toxic effect of the dye. From the results obtained in the test in comparison with the

estimation of the number of micro-organisms by Barthel and Orla-Jensen's method of making plate counts, it was found that errors of under-estimation were especially frequent in cold weather. This may be attributed to the reducing power and vitality of organisms which have become predominant at a low temperature being weakened at 38°–39° C.; and this view gains support from the fact that better results are obtained by making the test at 28°–29° C. This lower temperature is also best in the case of pasteurised milk, possibly because it is less favourable to the development of organisms which have survived the pasteurising process. The average optimum temperature of the organisms which survive pasteurisation at a low temperature is higher than that of the organisms in raw milk, whilst in the case of milk which has been heated to higher temperatures the surviving organisms show increased reducing power, probably owing to the elimination of lactic acid bacteria, and the consequent more rapid development of peptonising organisms which grow freely at 38°–39° C. To prevent the results being affected by dissolved oxygen in the milk, it is essential, as suggested by Orla-Jensen, that the samples should be shaken before being placed in the water-bath. By the use of these modifications the efficiency of pasteurisation may be checked, and good, bad, and indifferent samples of milk may be differentiated, but the test is not capable of distinguishing between different grades of milk containing less than half a million organisms per c.c. For distinguishing between "flash-pasteurised" milk (i.e., heated for 30 seconds at the definite temperature), and bulk-pasteurised milk, information would be obtained by the fermentation test at 38°–39° C. In the case of milk which had been heated above 80° C., peptonisation would occur, whereas milk which had been heated at lower temperatures would yield a fine gelatinous curd. A comparison of the times required for reduction at 38° and 28° C. would probably afford information concerning the past treatment of a sample of milk. (See also Ayers and Johnson, this J., 1915, 373.)—C. A. M.

*Starch; Polarimetric determination of — in grain, milling products, etc.* S. Hals and S. Heggenhougen. Landw. Versuchs-Stat., 1917, 90, 391–414.

EWERS' method (this J., 1916, 432), with slight modification, is recommended for the purpose. The substance is ground to pass a 1 mm. sieve and 2.5 grms. of the powder is placed in a 100 c.c. flask together with 25 c.c. of 1.124% hydrochloric acid. After the mixture has been shaken, the sides of the flask are rinsed down with a further 25 c.c. of the hydrochloric acid, and the flask is placed in a boiling water-bath for exactly 15 mins.; during the first 3 mins. the flask is shaken. Cold water is then added so as to make the volume up to about 90 c.c., the mixture is cooled to room temperature, from 0.25 to 1.5 c.c. of sodium or ammonium molybdate solution (containing 120 grms. of MoO<sub>3</sub> per litre) is added, the whole is diluted to 100 c.c., filtered, and the filtrate polarised in a 200 mm. tube. The reading in angular degrees is multiplied by 10.94, or in Ventzke degrees by 3.79, to obtain the percentage quantity of starch. A correction for water-soluble optically active substances is made by digesting 12.5 grms. of the sample with 250 c.c. of cold water for 1 hour, filtering the mixture, treating 50 c.c. of the filtrate with 2.1 c.c. of hydrochloric acid (sp. gr. 1.125) and heating this mixture in a boiling water-bath for 15 mins. The mixture is then cooled, clarified, and polarised as before, the reading obtained being deducted from that observed in the main determination.

—W. P. S.



*Tomato seeds and skins; Utilisation of waste* —.

F. Rabak. Bull. No. 632. Bureau of Plant Ind., U.S. Dept. Agric., Nov. 30, 1917. 15 pages.

In the preparation of tomato pulp the fresh tomatoes are washed by a stream of water under pressure, then cooked with steam, and pulped in a cyclone machine which separates the seeds and skins; or a cold process is used in which, after removal of the "culis," the washed tomatoes pass directly to the cyclone machine. It is estimated that in the U.S.A. about 300,000 tons of tomatoes is annually pulped, and that the moist waste amounts to about 16,000 tons, corresponding to approximately 3300 tons of dry waste, yielding about 1500 tons of dry seeds and 1800 tons of dry skins. This estimate is based upon the data that American tomatoes yield on the average 0.52% of dry seeds and 0.61% of dry skins. In Italy the wet seed and skins are pressed and then dried in a desiccating machine heated by means of steam pipes and containing horizontal conveyors. About 10 tons of the residue is dried in 24 hours, and the seeds are then separated from the skins in a machine containing a series of sieves and fans. The yield of oil from the seeds by extraction with ether or carbon tetrachloride is about 22% of the ground seeds. The extracted oil contains more impurities than the expressed oil, and in the crude state has a slightly rancid odour and a slightly bitter taste, but it can be readily deodorised by the action of steam, and to a large extent decolorised by treatment with fullers' earth and filtration. The refined oil is of a pale yellow colour, and has a pleasant nut-like odour and taste. A sample of extracted refined oil had the following characters:—Sp. gr. at 24° C., 0.9184; refractive index at 25° C., 1.4715; solidification pt., -10°C.; acid value, 2.5; saponification value, 188.6; and iodine value, 114.2. Fatty acids:—Sp. gr. at 25° C., 0.9100; refractive index at 25° C., 1.4655; solidific. pt., 21.5° to 20.5° C.; neutralisation value, 180; iodine value, 104.3; and iodine value of liquid fatty acids, 130. The oil was free from soluble fatty acids, but contained 96.2% of insoluble fatty acids, which were separated by the lead-ether method into 17.54% of solid and 75.84% of liquid fatty acids. The solid fatty acids consisted of about 68% of stearic and 32% of palmitic acid, whilst the liquid fatty acids consisted of about 57% of oleic and 43% of linolic acid. Under the present conditions about 343 tons per annum of tomato seed oil would be available in the United States. Experiments have shown that the oil has a digestibility value of 97, which compares favourably with the value of olive, almond, and cottonseed oils. It would be useful as a culinary and salad oil and would probably yield a satisfactory hydrogenation product for margarine. By cold saponification with caustic soda it yields a soap of good texture with excellent lathering properties. It is a semi-drying oil, yielding a soft sticky film in about 16 days. The residual seed meal had the following composition:—Moisture, 7.15; ash, 4.64; proteins, 37.0; nitrogen-free extract, 29.10; and crude fibre, 22.11%. It thus ranks with cottonseed meal and linseed meal as regards proteins, but the crude fibre is relatively high although lower than that of palm-nut cake. It has been found by Scarpitti that the meal is a valuable feeding-stuff, and has a better effect than linseed cake upon the weight and lactal secretion of cows. The dried skins could be incorporated with the meal, as in Italy, thus increasing the annual available quantity to about 3000 tons.—C. A. M.

*Grape syrup ["honey"]*. F. Martinotti. Giorn. Vinicolo, 1917, 43, 362. Bull. Agric. Intell., 1917, 8, 1307.

In a process invented by Monti (cp. U.S. Pat.

1,167,006, this J., 1916, 270) grape must is concentrated, first by freezing, which removes the excess of acid, the impurities, and about one-fourth of the water, and then by evaporation under greatly reduced pressure. The product is syrup or "honey" having the nutritive and therapeutic value of grape juice. The must obtained by pressing the grapes, and the liquid obtained from the skins, are mixed either before or after the freezing process.—J. H. L.

*"Woody fibre" in feeding stuffs; Determination of* —. J. A. and E. W. Voelcker. Analyst, 1918, 43, 31.

In this country the usual method of determining "woody fibre" is that devised by Voelcker, in which the hydrolysis is effected by means of 2% sulphuric acid and 2% caustic alkali solution. In the American and Continental official methods, however, solutions of only 1.25% strength are used, and in the case of oil-cakes from seeds with hard "husks" considerable differences are obtained by the two methods. For example, it is not possible to effect the hydrolysis of palm nut cakes and the like with solutions of lower strength than 2%, and even then vigorous boiling for 30 mins. is required. After extraction of the oil from 3 grms. of the ground sample the residue is boiled for 30 mins. with 125 c.c. of 2% sulphuric acid, the volume of the liquid being kept constant during the boiling. The liquid is then diluted to 400 to 500 c.c. and allowed to settle, the supernatant liquid decanted through a linen filter, and the residue washed on this and returned to the original vessel, where it is boiled for 30 mins. with 2% caustic alkali solution. The liquid is again decanted as before, and the insoluble fibre washed free from alkali, then treated with a little dilute acid, and again washed with water, and finally with alcohol, and dried at 100° C. until constant in weight. Any mineral matter left on ignition is deducted from the weight of fibre.—C. A. M.

*Sweet chestnuts; Analytical examination of* —. J. L. Baker and H. F. E. Hulton. Analyst, 1918, 43, 32–33.

The average weight of an English chestnut (*Castanea sativa*) was found to be, including the brown skin, 5.45 grms. The skins (17.4% of the nut) contained 49.3% of water, and the kernels (82.6%) contained 59.86% of water. The peeled nuts after being dried and finely ground had the following composition:—Moisture, 4.7; ash, 2.68; ethereal extract (oil), 2.9; proteins, 7.44; reducing sugars (as dextrose), 5.36; sucrose 9.0; starch (Lintner), 50.6; starch (taka-diastase), 41.08; pentosans, 3.06; crude fibre, 2.28; and substances soluble in cold water, 22.08%. The residual material, after removal of substances soluble in water, and starch, contained all the fibre, 65% of the pentosans, 56% of the total proteins, and 90% of the original proteins insoluble in water; when hydrolysed with acid it yielded a reducing sugar (15.7% of the material calculated as xylose) and was almost completely soluble when treated successively with 1.25% acid and 1.25% alkali solution. It was thus probably of the nature of a "hemicellulose." The substances soluble in water contained:—Water, 4.7; ash, 2.4; proteins, 2.8; sucrose, 8.9; dextrose, 5.36; and pentosans, 1.07%. Of the total matter soluble in water 64.6% consisted of fermentable sugars. The aqueous extract of the undried kernel had a diastatic value (Lintner) of less than 0.5.—C. A. M.

*Marine algæ as fodder for horses; Utilisation of certain* —. Adrian. Comptes rend., 1918, 166, 54–56.

CERTAIN seaweeds of the class *Laminaria*, after

washing to remove salts, have a composition which indicates their suitability for fodder. The following is a typical analysis: Moisture, 14.40; carbohydrates, 52.90; proteins, 17.30; cellulose, 11.50; mineral matter, 3.90%. Although poorer in carbohydrates than oats, the seaweed is much richer in protein and, provided it were acceptable and assimilable, this material should serve as a substitute in times of scarcity. In a mixed ration of oats, hay, and straw, the oats were completely replaced by an equal weight of seaweed and fed to twenty cavalry horses for a period of two months. The results were perfectly satisfactory, showing a considerable gain in weight for the animals fed on seaweed as compared with twenty others on the normal ration. It would appear that 0.75 kilo. of the seaweed is equivalent in feeding value to 1 kilo. of oats. The seaweed used is plentiful on the Breton coast.—J. F. B.

*Turnip-tops; Losses occurring during ensilage of* —. F. Honcamp. *Landw. Versuchs-Stat.*, 1917, 90, 431—442.

WHEN turnip-tops are stored in pits for some months the loss of organic substances amounts to about 30%; the greatest loss is in the carbohydrates, but the proteins are also diminished to a considerable extent. Storage after artificial drying is to be preferred, but cheaper and more efficient apparatus than that at present available would be required.—W. P. S.

*Comparative examination of pure and commercial inulin.* Wolff and Geslin. *See* XVII.

*Bran as a new raw material for cereal beverages.* Wahl. *See* XVIII.

#### PATENTS.

*Meal or flour and milling products; Process for treating* —. Naamlouze Vennootschap Industriele Maatschappij voorheen Noury and Vander Lande, Deventer, Holland. Eng. Pat. 102,967. Dec. 21, 1916. (Appl. No. 18,325 of 1916.)  
• Under Int. Conv., Dec. 22, 1915.

FLOUR is mixed with 0.7% of a 3% solution of hydrogen peroxide and is transported by a belt conveyor through a room containing mercury vapour lamps of 200 volts each. The layer of flour on the belt is 3 cm. deep, and 200 kilos. of flour is treated by each lamp per hour. Decomposition of the peroxide may be brought about also by passing the flour down a shoot between electrodes, and by stirring the flour in a drum by means of zinc plates. Instead of hydrogen peroxide any inorganic or organic peroxide or both may be used, either in solution, suspension, or the solid state. The treatment is claimed to improve both the colour and the baking qualities of the flour.—J. H. J.

*Bread making; Art of* —. R. and A. S. Wahl, Chicago. U.S. Pat. 1,247,349, Nov. 20, 1917. Date of appl., Sept. 12, 1916.

A MASH containing phosphates, proteins, and the peptonising enzyme of malt is digested at 45° C., and then added to the dough. A mash containing the aleurone layer of seeds and lactic acid is maintained under conditions favouring the proteolytic digestion of the proteins, starch is then added and, when this has been hydrolysed by the diastase in the seeds, the mixture is added to the dough. Yeast is added to a solution of yeast food or "bread improver" having a cereal base, and, when the solution has become charged with carbon dioxide, the mixture is incorporated with the dough.—W. P. S.

*Milk and other liquids; Process and apparatus for condensing and reducing to powder* —. E. C. R. Marks, London. From Merrell-Soule Co., Syracuse, N.Y., U.S.A. Eng. Pat. 111,508, Nov. 24, 1916. (Appl. No. 16,858 of 1916.) (See also U.S. Pat. 1,225,348 of 1917; this J., 1917, 633.)

MILK is passed into the bottom of a heated centrifugal chamber and is made to pass spirally up the side of the chamber by the rotation of a beater at a high speed, and in so doing is concentrated by evaporation. The concentrated portion passes out at the top. The vapour given off passes into a condensing chamber and its rapid condensation causes a vacuum of 23—26 inches of mercury in the centrifugal chamber. As the concentrated milk passes out of the centrifugal chamber it may be atomised into a forced current of heated air and reduced to powder form.—J. H. J.

*Milk-serum powder and process of making same.* A. W. Bosworth, Milton, Mass., Assignor to The Boston Floating Hospital, Boston, Mass. U.S. Pat. 1,246,858, Nov. 20, 1917. Date of appl., Aug. 9, 1917.

FAT, casein, and all but a trace of calcium and other inorganic phosphates are removed from milk in such a way that the vitamins are not destroyed, and the liquid is then evaporated to dryness at a low temperature.—W. P. S.

*Butter substitute.* R. H. Adams, Ridgewood, N.J., and H. Beatty, Hinsdale, Ill. U.S. Pats. (A) 1,247,482 and (B) 1,247,483, Nov. 20, 1917. Date of appl., Feb. 10, 1917.

(A) THE water-insoluble solids of ripened milk are mixed with a large quantity of solid coconut oil. (B) Varying quantities of solid coconut oil and the water-insoluble solids of butter-milk are mixed together, e.g., 3 parts of coconut oil to 1 part of butter-milk solids.—W. P. S.

*Rice food product.* A. Matsuo, San Francisco, Cal. U.S. Pat. 1,248,884, Dec. 4, 1917. Date of appl., Apr. 18, 1917.

COOKED rice is fermented with barley malt, the liquid is removed by straining, and the solids are macerated with more cooked rice and then dried in thin flakes. The method may be varied by adding Japanese saké and glucose before macerating.—J. H. J.

*Flesh; Method of preserving* —. G. D. Rogers, Gloucester, Mass. U.S. Pat. 1,249,205, Dec. 4, 1917. Date of appl., Sep. 1, 1917.

FLESH is packed in sodium chloride and a solution of sodium hypochlorite.—J. H. J.

*Carbonic acid solution; Stable — and method of producing same.* E. Rudolff, Assignor to L. E. Elkan Erben Ges. m. b. H., Berlin-Westend, Germany. U.S. Pat. 1,247,334, Nov. 20, 1917. Date of appl., June 25, 1914.

CARBONATED waters containing a relatively large quantity of carbonic acid are prepared by dissolving carbon dioxide in an aqueous liquid containing chlorophyll or a chlorophyll derivative.—W. P. S.

#### XIXB.—WATER PURIFICATION; SANITATION.

*[Sewage] sludge; Nature of activated* —. F. Diénert. *Comptes rend.*, 1917, 165, 1116—1117.

To prepare activated sludge from Paris sewage, it was found necessary to aerate the sewage for 30 hrs. to effect the nitrification of the 10 mgrms. of ammonia per litre which it contained. The liquid was then decanted, a further volume of



sewage was admitted to the residual sludge, and aeration was continued. These operations were repeated for 15–20 days, when the time required for nitrification had been reduced to one and a half hours and the volume of sludge had much increased. The sludge was then in an activated condition. During the aeration the dissolved carbon dioxide is expelled and the calcium bicarbonate is precipitated as carbonate; at the same time the precipitate removes some of the suspended and dissolved organic matter by adsorption. It was found that the alkalinity of the sewage was reduced from 180 parts to 60 parts of lime per million by one and a half hours' aeration. The activated sludge contained 50% of mineral matter, chiefly calcium carbonate, 20% of albuminoid matter, and 30% of non-albuminoid matter. In tests in which the sewage took about 2 hours to travel along the sewers, it was found that in consequence of the aeration which took place during that time, about three-fourths of the albuminoid and ammoniacal matter was adsorbed by the precipitated calcium carbonate and was found in the deposits of calcium carbonate along the sewers.—J. H. J.

*Dichloramine T and chlorinated eucalyptol (sp. gr. 1.2).* R. B. Krauss and E. Crede. J. Amer. Chem. Soc., 1917, 39, 2720–2722.

For the preparation of dichloramine T on a large scale, toluene-*p*-sulphonamide is dissolved in ten parts of 1:10 sodium hydroxide (39° B., sp. gr. 1.345) and diluted with twenty parts of water. The solution is filtered and chlorine is passed in until a voluminous white precipitate is formed, which is collected, washed with 5–8 parts of water and finally and quickly with enough 10% alcohol to make a thin paste. The substance is separated on a vacuum filter and dried at below 55° C. in a vacuum drier. The product has a negligible ash, and good chlorine content, but does not evolve free chlorine on standing. The following specifications are given as most suitable for the material when used for surgical purposes:—White powder or crystals, m.pt. 78°–84° C., soluble in cold chloroform with slight to no turbidity, and soluble in prepared eucalyptol (Dakin) or chlorinated eucalyptol (sp. gr. 1.2). When chlorine is passed into eucalyptol exposed to good daylight, the rate of passage of the gas being controlled so that the temperature does not rise above 80° C., a chlorinated eucalyptol (sp. gr. 1.2) is obtained, which after washing with water, shaking with anhydrous sodium carbonate, and drying over fused calcium chloride, is left as a slightly amber-coloured oil, chlorine content 31%, which will dissolve 20% of dichloramine T and may be used at full strength on the skin or an open wound, without the need for dilution with prepared paraffin oil.—W. G.

*Cresol; Preparation of a preservative from* —. M. Nevin and B. Mann. J. Amer. Chem. Soc., 1917, 39, 2752–2756.

IF commercial cresol is fractionally distilled, the process being repeated three times, a fraction is obtained, having b.pt. 199°–204° C., sp. gr. at 25° C., 1.030, which has a toxicity equal to that of phenol, and only slightly lower than that of Schering's "Trikresol," and a phenol (germicidal) coefficient of 2.55, which is higher than that of "Trikresol."—W. G.

*Guaiacol and benzoic acid; Physiological properties and medico-surgical applications of* —. L. Menciére. Comptes rend., 1917, 165, 1023–1025.

AN antiseptic solution, containing guaiacol and benzoic acid, with or without other aromatic substances, such as eucalyptol, has been extensively applied in France in the treatment of

wounds, and with great success. It has powerful antiseptic properties, but is not toxic towards cellular protoplasm, and unlike hypochlorite it has no solvent action on animal tissue. Pieces of flesh immersed for 15 months in an aqueous solution containing 5 grms. of guaiacol, 4 grms. of alcohol, and 1 gm. of benzoic acid per litre, underwent no apparent change, and when afterwards left in the air they remained immune from putrefaction. Intravenous injection of much stronger solutions had no injurious effect on rabbits.—J. H. L.

#### PATENTS.

*Sewage; Tanks for the purification of* —. G. W. and J. F. Naylor, Denby Dale, Yorks. Eng. Pat. 111,548, Dec. 7, 1916. (Appl. No. 17,569 of 1916.)

THE air-diffusing means along the bottom of a tank for the treatment of sewage by diffusion of air in small bubbles, may consist of non-porous tubes with small perforations on their under sides, covered by strips of cloth, tightly encircling the tube. The whole length of the tube is enclosed by a cloth casing kept from touching the under side of the tube by depending stays. The air from the perforations in the tube diffuses through the cloth casing, mainly through the sides. The tubes may be made also of porous earthenware, the upper surface being painted over so that air escapes from the sides only.—J. H. J.

*Sewage and other impure liquids; Purification of* —. W. Jones, Stourbridge, Worcestershire, and Jones and Attwood, Ltd., Amblescote, Staffordshire. Eng. Pat. 111,720, Dec. 9, 1916. (Appl. No. 17,718 of 1916.)

CRUDE sewage enters a treatment tank through a hydropneumatic appliance whereby a mixture of sewage and air is delivered well below the surface into the body of the liquid in the tank. The bottom of the tank is of inverted conical shape with a sump at the apex, and the dilute sludge passes out by the sump into a separate tank with ridged bottom provided with air diffusers, whereby it is aerated and circulated. This invigorated sludge is then returned by an air-lift to the top of the liquid in the sewage tank. In another form of the tank, the mixture of sewage and air is delivered into radial arms at the level of the conical portion of the tank, and as it issues from these through a series of holes along one side, it causes them to revolve and bring about a perfect distribution. The process may be carried out also in several tanks connected in series.—J. H. J.

*Filtration [of water]; Art of* —. W. M. Jewell, Assignor to Jewell Engineering Co., Chicago, Ill. U.S. Pat. 1,248,127, Nov. 27, 1917. Date of appl., May 12, 1913.

THE process is a combined method of water filtration and filter cleansing, in which, when the filter requires to be cleaned, the flow of the filtered water is reversed through the same path in which it is discharged from the filter.—J. H. J.

*Waters; Method for treating ferruginous* —. L. S. Hughes, Chicago, Ill. U.S. Pat. 1,248,329, Nov. 27, 1917. Date of appl., Oct. 2, 1913.

MINE water is treated by passing over small pieces of broken limestone, and then over relatively larger pieces at a higher velocity.—J. H. J.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Chemical constitution and physiological action; The relation between* —. F. L. Pyman. Chem. Soc. Trans., 1917, 111, 1103–1128.

THE study of the relation between chemical



structure and physiological action is complicated by the almost total absence of general laws, and a substituent group may have a pronounced influence in one case but none at all in a parallel case. Even with such simple nerve reactions as smell and taste, the relations between members of homologous series are not always constant, and stereoisomerism, whether due to asymmetry of C or N atoms, may sometimes have no influence, while in many cases it is associated with a partial or complete reversal of physiological character. Variations in the physiological activity of stereoisomerides may possibly be due to the formation of additive compounds with the organic tissues in the case of one and not the other of the isomerides. Other differences may be traced to simple differences in physical and chemical properties, e.g. solubility, which may influence the distribution of the substance in the animal organism, or the liberation of bases from their salts by the alkalinity of the blood. Instances of profound physiological modification, resulting in total inactivity, are often found by the introduction or liberation of sulphonic or carboxylic acid groups in the molecule of an active base, which may also perhaps be attributed to reaction with the blood alkalinity. Similarly the formation of quaternary salts from alkaloids may very largely neutralise their activity, which returns when the tertiary character is restored, e.g., by hydrogenation. Certain alkaloids are almost identical in physiological action yet have little in common as regards chemical structure, for instance, the group nicotine, lobelline, and cystine or the group muscarine, arecoline, and pilocarpine. For systematic investigation it is necessary to select a particular drug the physiological action of which is capable of quantitative measurement, and to study comparatively the effect of a large series of substituent variations in different groups of its molecule. A convenient series for such a study is that of the tropeines, typified by atropine, the *dl*-tropic ester of tropine, which is characterised by its mydriatic activity. Atropine is the racemic form of hyoscyamine, the pure *l*-hyoscyamine being about 100 times as active as the *d*-constituent. No tropeine of an aliphatic acid has been found to possess mydriatic activity in dilute solution, but the closed ring of the acyl residue need not be of the benzene type, since active tropeines of pyridyl acids exist. It is not essential that a mydriatic tropeine must contain an alcoholic hydroxyl in the side chain carrying the carboxylic group, nevertheless those tropeines possessing a high mydriatic power do contain such hydroxyl. The acid sulphuric ester of this hydroxyl is in all cases inactive; tropeines containing an unsaturated linkage in the carboxylic side chain are also inactive. The local anaesthetic effect characteristic of the alkaloid cocaine has been proved to be associated with the chemical type of esters of amino-alcohols. The acyl group in the majority of cases of practical utility is aromatic, generally benzoyl, but a ring complex does not appear to be essential. Substitution in the benzoyl residue lowers the activity; cinnamoyl-cocaine is inactive. In other cases, e.g., alpine and novocaine, the substitution of the cinnamoyl group for the benzoyl increases the activity. The amino group may be secondary or tertiary or may be associated with simple or bridged rings. Cocaine, tropacocaine, eucaine, alpine, and novocaine all represent different types of nitrogen linking. Norcocaine, in which the  $\text{N.CH}_3$  becomes  $\text{N.H}$ , is more active than cocaine but the primary amine corresponding to novocaine is inactive. The alcohol group may be primary, secondary, or tertiary and may separate the acyl and amino groups by a chain of two or three carbon atoms. Adrenaline, which is  $\beta$ -3,4-trihydroxy- $\beta$ -phenylethylmethylamine, has a vasoconstrictor action which can be measured by the rise of blood pressure.

Although catechol causes a rise of blood pressure, the action of adrenaline is not due to the catechol residue contained therein, since the type of physiological action is entirely different. A large number of amines possess the characteristic property in a more or less slight degree, but the only compound with an activity comparable with that of adrenaline is the primary ethylamine of corresponding constitution; *l*-adrenaline is considerably more active than the racemic form. Protozoacidal drugs are typified by the alkaloids quinine and emetine, and experiments have been made to find derivatives of related constitution which would be more deadly to infusoria (protozoa) and less toxic to mammals. The results obtained *in vitro*, however, have not been borne out in clinical practice. Ethylhydrocupreine hydrochloride appeared in the laboratory to offer the advantages over quinine sulphate which were desired, but in practice it was inferior; hydroquinine hydrochloride was the most valuable. In the emetine series the full amoebacidal action is not obtained unless the emetine nucleus is intact; none of the related alkaloids was superior to emetine in practice. In fact, in none of the cases discussed above are the natural products, hyoscyamine, cocaine, adrenaline, quinine, and emetine, surpassed in point of maximum effect by any of their derivatives, nevertheless useful additions to medicine have resulted from the researches.—J. F. B.

[Essential] oil of *Artemisia annua*. Asahina and Yoshitomi. J. Pharm. Soc. Japan, 1917, No. 424, 1. Perf. and Essent. Oil Rec., 1917, 8, 353–354.

THE work of Imada on the essential oil of *Artemisia annua* has been extended. The oil used had the following characteristics:—Sp. gr. at  $16^\circ/4^\circ\text{C}$ ., 0.90798; optical rotation,  $16-19^\circ$ ; acid value, 1.82; ester value, 34.58; ester value after acetylation, 63.54. A minute quantity of an oil with an odour of cinnic aldehyde was extracted by sodium bisulphite solution. The remainder was steam-distilled, yielding an oil of low specific gravity. The brown residue, amounting to about one-fifth of the original oil, distilled between  $128^\circ$  and  $182^\circ\text{C}$ . at 10 mm. pressure. The fraction of the light oil distilling from  $176^\circ$  to  $181^\circ\text{C}$ . contained cineol. The fraction distilling from  $181^\circ$  to  $190^\circ\text{C}$ . contained a ketone yielding a semicarbazone melting at  $96^\circ\text{C}$ ., from which concentrated hydrochloric acid regenerated the pure ketone, having the formula  $\text{C}_{10}\text{H}_{16}\text{O}$ , boiling at  $71^\circ-75^\circ\text{C}$ . at 11 mm., and at  $182^\circ$  at 760 mm. Its sp. gr. at  $14^\circ/4^\circ\text{C}$ . was 0.8906, refractive index 1.4695 at  $18.5^\circ\text{C}$ ., and it was optically inactive. Catalytic reduction by hydrogen in the presence of platinum black yielded a tetrahydroketone, forming a semicarbazone melting at  $134^\circ-135^\circ\text{C}$ . The pure tetrahydroartemisia-ketone boiled at  $73^\circ\text{C}$ . at 15 mm., and at  $173^\circ\text{C}$ . at 760 mm.; sp. gr. at  $19.5^\circ/4^\circ\text{C}$ ., 0.8262, and refractive index 1.42425. The higher-boiling fractions of the oil ( $190^\circ-210^\circ\text{C}$ .) yielded a semicarbazone melting at  $236^\circ-237^\circ\text{C}$ ., which when treated with dilute sulphuric acid yielded a solid ketone, m. pt.  $175^\circ\text{C}$ ., with all the properties of *l*-camphor.—F. Sp.

[Essential] oils of *Darwinia*. H. G. Baker and R. T. Smith. J. Proc. Roy. Soc. N.S.W., 1918, 1. Perf. and Essent. Oil Rec., 1917, 8, 355.

THE following are the characteristics of the essential oils of three species of *Darwinia*. (1) *D. fascicularis*. Sp. gr. 0.9184; optical rotation  $+1.2^\circ$ ; esters (hot saponification) 60%; esters (cold saponification) 58%; yield, 0.318%. (2) *D. grandiflora*. Sp. gr. 0.915; optical rotation  $+23.1^\circ$ ; refractive index, 1.4773; esters (hot



saponification) 35.1%; esters (cold saponification) 33.8%; yield, 0.12%. (3) *D. larifolia*. Sp. gr. 0.8779; optical rotation. —6.5°; esters (hot saponification). 5.3%; yield 0.313%. The oil from *D. grandiflora* is red and somewhat mobile. The difference of 3.7 between the hot and cold saponification values is said to be due to the presence of geranyl butyrate, the main ester being geranyl acetate. The mixture of barium salts from the liberated mixed acids contained 92.15% of barium acetate and 7.85% of barium butyrate. A considerable portion of the oil distilled between 156° and 160° C., and about 30% below 165° C. This fraction had a terpene-like odour. sp. gr. 0.872, optical rotation 41.6°, and refractive index 1.4685. Its nitrosochloride melted at 104° C. It is therefore *d*-pinene. The pinene in the oil from *D. larifolia* is levorotatory, and in the oil from *D. fascicularis* dextro-rotatory.—F. Sp.

*Lemon grass oil; Constituents of Formosan* —. H. K. Kafuku. *Kōgyō-Kwagaku-Zasshi* (J. Chem. Ind., Tokyo). 1917. 20, 825—833.

THE terpene isolated from lemongrass oil from Formosa (this J., 1916, 865) gave, on reduction with sodium and alcohol, a derivative corresponding closely with dihydromyrcene, yielding dihydromyrcene tetrabromide, m. pt. 87°—88° C. It was further reduced by hydrogen and platinum to 2,6-dimethyloctane. On oxidation with alkaline permanganate it yielded succinic and oxalic acids. The identification of this terpene with myrcene is considered complete. In addition, the oil contains an aldehydic substance, other than citral, which has not yet been isolated.—J. F. B.

*Tannic acid, gallic acid, and pyrogallol; Preparation of* —. M. Mito. *Kōgyō-Kwagaku-Zasshi* (J. Chem. Ind., Tokyo). 1917, 20, 720—736.

TANNIN was extracted from Japanese gall nuts by four successive treatments with water at 20° to 30° C., and the residue was pressed. In this way a 13% solution of tannin containing 82% of the theoretical quantity was obtained. Tannic acid was prepared by concentrating the extract to 20%, extracting it with half its volume of ether, and evaporating the ethereal extract. About 75% of the tannin in the aqueous extract was obtained. For the preparation of gallic acid the residual solution from the tannic acid preparation was concentrated to 20%, and treated with 15 to 20% (by vol.) of sulphuric acid (66° B., sp. gr. 1.84), and the precipitated black mass was dissolved in water, decolorised with animal charcoal, and finally recrystallised from water. The yield was 62% of the tannin contained in the residual solution. Pyrogallol was prepared by heating the crude gallic acid with about 3 times its volume of water in an autoclave at 210° C. The crude product (yield, 63.5% of the gallic acid) was purified by crystallisation from pure benzene.—C. A. M.

*Nitriles; Catalytic method for the preparation of aromatic* —. A. Mailhe. *Comptes rend.*, 1918, 166, 36—38.

THE vapour of an alkyl ester of an aromatic acid, mixed with gaseous ammonia, is passed through a tube containing thoria heated to 450°—470° C., and the nitrile is purified by fractional distillation of the oily layer of the liquid obtained. Good yields of nitriles have been obtained from the alkyl esters of benzoic, toluic, and phenylacetic acids. (See also J. Chem. Soc., Feb., 1918).—J. F. B.

*Corrosion of metals; Hydrolysis of organic halides and the* —. [Colour reaction of tetrachloroethylene.] H. W. Doughty. J. Amer. Chem. Soc., 1917, 39, 2685—2692.

THE corrosion of metal containers (tin cans) by moist carbon tetrachloride cannot be due merely

to the hydrolysis of the organic halide and subsequent action of the hydrochloric acid upon the metal, since in the case of iron and copper the corrosion is very greatly increased by the presence of ammonia. The hydrolysis of carbon tetrachloride is greatly accelerated by iron and copper. It is also accelerated by hydrogen ions and hydroxyl ions, especially at elevated temperatures. A number of organic halides were examined as to their action on copper in the presence of water and ammonia, and the corrosion was found to be most pronounced with compounds containing a trichloromethyl group, monohalogen derivatives having practically no action.

A colour test for tetrachloroethylene is given. When this compound is allowed to stand in contact with water, ammonia, and copper, the aqueous layer develops a rich, red colour.—W. G.

*Test for tartrates depending upon the formation of the copper-tartrate complex.* Curtman and others. See VII.

*Dichloroamine T and chlorinated eucalyptol* (sp. gr. 1.2). Krauss and Crede. See XIXB.

*Physiological properties and medico-surgical applications of guaiacol and benzoic acid.* Menciére. See XIXB.

#### PATENTS.

*Acetaldehyde; Method of preparing* —. H. Hibbert. Pittsburgh, Pa., Assignor to Union Carbide Co., New York. U.S. Pat. 1,247,270, Nov. 20, 1917. Date of appl., Apr. 28, 1916.

ACETYLENE is combined with the elements of water in an acid bath containing a mercury compound, maintained at a suitable temperature for the continuous distillation of acetaldehyde, and the mixture of acetaldehyde and acetylene is conveyed to one or more baths connected in series with, and similar in composition to, the first bath.—F. W. A.

*Vaccin for the prevention of hog-cholera and product therefrom; Manufacturing* —. M. J. Couret, Assignor to Louisiana Biological Products, Incorp., New Orleans, La. U.S. Pat. 1,246,059, Nov. 13, 1917. Date of appl., Feb. 16, 1917.

A VACCIN product for the prevention of hog-cholera is produced by removing from the hog those parts containing the virulent virus, removing solid particles, extracting the liquid residue with chloroform, separating the chloroform at a temperature not above 45° C., and desiccating the residue at about 10° C. The yield is increased by injecting into the animal a liquid germ culture containing dextrose, having an acid reaction and possessing exosmotic properties.—L. A. C.

*Chlor-substitution products of the saturated paraffin hydrocarbons; Preparation of* —. C. Blanc, Assignor to The Goodyear Tire and Rubber Co., Akron, Ohio. U.S. Pat. 1,248,065, Nov. 27, 1917. Date of appl., May 1, 1914.

A LIQUID paraffin hydrocarbon is subjected to the action of chlorine and a chlorine carrier under the influence of actinic light and a temperature-regulating agent. The desired chlorine substitution product is removed and fresh supplies of chlorine and hydrocarbon added to the remaining material.—L. A. C.

*[Aromatic] alcohols; Process of producing* — by hydrogenation. A. Brochet, Paris. U.S. Pat. 1,247,629, Nov. 27, 1917. Date of appl., July 16, 1913.

SEE Fr. Pat. 458,033 of 1912; this J., 1913, 1031.

*Process and apparatus for recovering the ether and alcohol vapour contained in the air of factories.* Eng. Pat. 101,723. See I.

*Apparatus for recovering ether and alcohol vapours contained in the air of factories.* Eng. Pat. 101,875. See I.

*Manufacturing side-chain chlorine derivatives of toluol [benzyl and benzal chlorides and benzo-trichloride].* U.S. Pat. 1,246,739. See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENTS.

*Photographic films [for X-rays].* A. Edwards, Warwick. Eng. Pat. 111,913, Dec. 16, 1916. (Appl. No. 18,104 of 1916.)

AN X-ray film of increased speed and uniformity is obtained by coating roll celluloid on both sides with a suitable emulsion.—B. V. S.

*Photographs; Method of producing multicoloured —.* A. R. Lawrie, Trenton, N.J. U.S. Pat. 1,248,139, Nov. 27, 1917. Date of appl., Feb. 5, 1916.

COLOUR-SELECTION negatives are obtained by exposing together two plates or films, placed face to face, through a suitable light-filter. From these, two coloured positives, red and blue, are obtained and the third colour is introduced by dyeing the film of the blue positive yellow and then removing the dye from the parts of this picture corresponding with the blue parts of the original. The positives are then superposed in register on a reflecting base.—B. V. S.

*Colour photographs or films; Method of producing —.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,248,861, Dec. 4, 1917. Date of appl., Feb. 4, 1916.

COLOURED positives, taken from red and blue-green selection negatives reversed with respect to one another, are fixed permanently together face to face in register. One of the celluloid backings is then dissolved away, the other being first coated with a gelatin or other protective covering.—B.V.S.

## XXII.—EXPLOSIVES; MATCHES.

### PATENTS.

*Nitrocellulose; Utilising the wash-water of the manufacture of —.* C. E. B. Seyffert, Troisdorf, Germany. U.S. Pat. 1,248,933, Dec. 4, 1917. Date of appl., July 22, 1915.

THE same water is used for washing successive charges of nitrocellulose containing adhering nitric and sulphuric acids which are progressively stronger in acid. The acids are recovered from the concentrated wash-water.—L. A. C.

*Process and apparatus for recovering the ether and alcohol vapour contained in the air of factories.* Eng. Pat. 101,723. See I.

*Apparatus for recovering ether and alcohol vapours contained in the air of factories.* Eng. Pat. 101,875. See I.

## XXIII.—ANALYSIS.

*Textile fibres; Use of — in microscopic qualitative chemical analysis.* E. M. Chamot and H. I. Cole. J. Ind. Eng. Chem., 1918, 10, 48—51. (See this J., 1918, 21 A.)

III. *Detection of boron by means of turmeric viscose-silk fibres.* To dye the fibres, a solution of turmeric is prepared by boiling about 20 grms. of ground turmeric root with 50 c.c. of alcohol and adding

to the filtered solution an equal volume of water and  $\frac{1}{2}$ —1 c.c. of 10% sodium hydroxide solution. The fibres are immersed in this solution, which is then evaporated to a syrupy consistency in a water-bath. The fibres are removed and immediately dipped in 95% alcohol, pressed between filter paper, dipped in dilute sulphuric acid, washed with water, and dried. Of all fibres tested, viscose-silk gives by far the best colour reaction. To apply the test, a drop of solution of the material to be tested is placed upon an object slide and acidulated with dilute hydrochloric acid. In this drop a turmeric fibre is placed, and the liquid evaporated carefully to complete dryness. The fibre is examined under the microscope. A rose or violet-rose colour indicates boron. To confirm, a drop of a 1% solution of sodium hydroxide is placed upon the fibre. The rose colour immediately turns to Prussian blue, changing to violet. Care must be taken that boron is not present in the object glass and that strong bleaching agents such as hydrogen peroxide or hypochlorites are destroyed. Boric acid can be distinguished from borates in the absence of inorganic salts capable of decomposing a borate by omitting the addition of the mineral acid. Boric acid gives the characteristic colour, but borates fail to do so. A drop of a solution containing 0.000,025 mgrm. of boron gives a positive test by this method.

IV. *Detection of the heavy metals by means of zinc sulphide wool fibres.* Wool, defatted by treatment with a mixture of alcohol and ether, is soaked overnight in a 1% solution of sodium hydroxide. It is then washed and dipped five or six times alternately in solutions of 10% zinc acetate and 10% sodium sulphide, pressing but not washing between the dippings. The wool is then washed and dried between filter papers. A drop of the solution to be tested is placed on the object slide, a drop of dilute hydrochloric acid added, a treated wool fibre introduced, and examined under the microscope. The solution is then evaporated to dryness, a drop of dilute ammonia added, and the fibre examined again, a new fibre also being placed in the drop. The colour of the fibre indicates the following metals: Straw-yellow—tin; lemon-yellow—arsenic, cadmium; orange—antimony; reddish-brown—bismuth; brown or yellow-brown—platinum, copper, mercury (ic), antimony (sometimes cobalt, iron, manganese, nickel); black (brown in very dilute solutions)—silver, lead, gold, mercury (ous). Colourless in acid solution, in alkaline solution the fibre may be brown or yellow-brown if cobalt, iron, manganese, or nickel is present. The colour in all cases depends to a certain extent upon the quantity of metal present.—L. A. C.

*Calcium; Rapid method of determining — as calcium sulphate.* L. G. Willis and W. H. MacIntire. J. Ind. Eng. Chem., 1917, 9, 1114—1116.

IN the methods in which calcium is determined as sulphate, sulphuric acid is usually employed for the conversion, but, owing to high temperature, loss occurs through dissociation of sulphate, whilst the organic matter contained even in "pure" sulphuric acid causes reduction of sulphate to sulphide. These sources of error may be obviated by using recrystallised ammonium sulphate to convert the calcium salt into sulphate, as first suggested by Schrötter (Fresenius, Quant. Chem. Anal., 1 (1906), 272). The calcium is first precipitated as oxalate, the precipitate ignited, and the residue of calcium carbonate incorporated with a dried, finely powdered mixture of equal parts of ammonium sulphate and ammonium chloride in sufficient quantity to give an excess of about 0.3 grm. of sulphate for each 0.2 grm. of the calcium salt. The mixture is then heated



over a small flame in a crucible inserted in a circular opening in a horizontal asbestos board, until the excess of ammonium salts is expelled. Duplicate determinations should agree within a small fraction of a mgrm.—C. A. M.

*Calcium; Volumetric determination of*—. J. Grossfeld. *Chem.-Zeit.*, 1917, **41**, 842. (See also *J. Chem. Soc.*, March, 1918.)

THE method depends mainly on the fact that non-crystalline, finely-divided calcium oxalate (such as is obtained from a slightly acid solution in the cold) may be separated by filtration through a "kieselguhr filter-paper" (see this *J.*, 1916, 1105). The slightly acid solution containing about 0.2 gm. of calcium oxide is treated with 10 c.c. of phosphoric acid solution (250 grms. of concentrated phosphoric acid per litre). 15 c.c. of 4% ammonium oxalate solution is added, and the excess of acid is partially neutralised by the addition of 10 c.c. of 10% sodium hydroxide solution; the mixture should now be acid towards litmus paper but not to Congo-red paper. After dilution to 100 c.c. the mixture is filtered through a "kieselguhr filter-paper," and an aliquot portion of the filtrate is acidified with sulphuric acid and titrated with N/10 permanganate solution. The difference between the quantity of permanganate used for this titration and that required for the titration of 10 c.c. of the ammonium oxalate solution, gives the amount of oxalate combined with the calcium and the quantity of the latter is found by a simple calculation.—W. P. S.

*Rubidium and cesium; Approximate quantitative method for the determination of*— in plant ash. W. O. Robinson. *J. Ind. Eng. Chem.*, 1918, **10**, 50—51.

At least 20 grms. of the dry plant is incinerated below 525° C. The ash is dissolved in hydrochloric acid, the excess evaporated off, and an excess of freshly slaked lime added. The liquid is boiled for a few minutes and filtered. Calcium in the filtrate is precipitated with ammonia and ammonium carbonate, and filtered off, and for precaution a second precipitation is made. The combined filtrates are evaporated to dryness in a muffle just below redness. The residue is treated with hot water, a few drops of hydrochloric acid added, and then about 0.05 gm. of platinum chloride. The solution is stirred well and evaporated to pastiness. The unchanged chlorides of sodium and potassium are rapidly dissolved in the minimum amount of hot water, the chloroplatinates washed with 80% alcohol on to a filter consisting of an asbestos pad on perforated platinum foil and reduced by gently heating in a current of hydrogen. The chlorides formed are washed through the filter with hot water, and the filtrate evaporated to pastiness in a small platinum dish. The mass is treated with four drops of strong hydrochloric acid and filtered into a vial of 2—3 c.c. capacity, a number of these being made to hold the same volume. The rare alkali chlorides are treated with two more portions of acid of four drops each, each portion being blown through the filter and the solution then made up to volume. Standards are made up by treating known amounts of cesium and rubidium chlorides and excess of potassium chloride with strong hydrochloric acid as above. These solutions are then examined spectroscopically by evaporating large drops on a coil of platinum wire and holding the residue in a Bunsen flame. Cesium is identified by the doublets 4215.6 and 4201.9 and rubidium by the double lines 4593.3 and 4555.4. The unknown solution is matched with the standards according to the brilliancy of the lines. An accuracy of 5—10% is easily obtainable.—L. A. C.

*Iron; Colorimetric determination of*—. E. R. Dovey. *Analyst*, 1918, **43**, 31—32.

THE following method obviates errors of comparison caused by differences in the concentration of thiocyanate or in the general composition of the two liquids:—A measured volume of the liquid under examination is treated with the thiocyanate solution, and divided in the proportion of one-third and two-thirds between two Nessler cylinders. The standard iron solution is then added to the smaller portion until its colour matches that of the larger.—C. A. M.

*Laboratory methods for benzol recovery plant operation.* Sperr, jun. See III.

*Analysis of crude benzols, etc.* Adam. See III.

*Determination of phenol in crude carbolic acid and coal-tar oils.* Skirrow. See III.

*Analytical control of ammonia oxidation.* Taylor and Davis. See VII.

*Test for tartrates depending upon the formation of the copper-tartrate complex.* Curtman and others. See VII.

*Solubility of silica.* Lenher and Merrill. See VII.

*Tensile strength test of neat Portland cement briquettes.* Hattori. See IX.

*Sampling and analysis of Chinese tin.* Browne. See X.

*Valenta number as a discriminating test for oils and fats.* Fryer and Weston. See XII.

*The physical basis of colour technology.* Luckiesh. See XIII.

*Estimation of sulphide in [tannery] lime liquors.* (1) Bennett. (2) McCandlish and Wilson. See XV.

*Detection of methyl alcohol in alcoholic beverages, and its formation by the several kinds of yeasts.* Takahashi and others. See XVIII.

*Reductase test of milk.* Arup. See XIXa.

*Polarimetric determination of starch in grain, milling products, etc.* Hals and Heggenhøgen. See XIXa.

*Determination of "woody fibre" in feeding stuffs.* Voelcker and Voelcker. See XIXa.

*Hydrolysis of organic halides and the corrosion of metals.* [Colour reaction of tetrachloroethylene.] Doughty. See XX.

#### PATENT.

*Hardness of metals and other substances; Testing or determining the*—. G. Boyelle-Morin, Paris. Eng. Pat. 108,460, Apr. 24, 1917. (Appl. No. 5781 of 1917.) Under Int. Conv., Apr. 25, 1916.

AN apparatus for testing the hardness of metals and other substances by comparison of the indentations produced in the substance and in a body of standard hardness, by the pressure of a hardened steel ball or the like, comprises a cylindrical case having a suitable detachable socket at the lower end for the hardened ball, and a hammer rod within the casing and projecting from the upper end. An aperture is provided for the insertion of a small block of the standard substance between the ball and the hammer rod, and a blow of any desired force is applied to the top of the hammer rod when the implement is held with the ball resting on the body to be tested. An impression is thus produced both on the metal to be tested and on the standard block. To

facilitate computation of the hardness, a slide rule is provided in which two fixed scales are graduated in hardnesses and resistance respectively, and the slide between the scales is graduated in diameters of impressions obtained in a body of standard hardness.—W. F. F.

*Calorimeter; Adiabatic*——. S. W. Parr. Urbana, Ill. U.S. Pat. 1,247,998, Nov. 27, 1917. Date of appl., Aug. 4, 1916.

AN inner vessel containing the calorimeter bomb immersed in a liquid is completely surrounded by an outer chamber also containing liquid. The outer vessel is provided with suitable stirring and heating appliances to maintain it at a temperature approximately the same as the external temperature of the inner vessel.—B. V. S.

## Patent List

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Anderson. Installation for extraction of mineral, animal, and vegetable substances. 1369. Jan. 24.

Barclay. Means for filtering gases. 1495. Jan. 25.

Borstein and Ratner. Drying-chambers. 1563. Jan. 26.

Davidson, and W. C. Holmes and Co. Apparatus for bringing liquids and gases, vapours, fumes, etc., into intimate contact. 1025. Jan. 18.

Donald. Kilns. 1595. Jan. 28.

Donald. Treatment of waste gases from furnaces, kilns, etc., and recovery and utilisation of products. 1596. Jan. 28.

Garrow. Chemical condenser of the Hart type. 1202. Jan. 22.

Johnson. Pumps for corrosive fluids. 1338. Jan. 23.

Kestner Evaporator and Engineering Co., and Reavell. Apparatus for concentrating, distilling, and/or elevating liquids. 1822. Jan. 31.

Lloyd and Wild. Recovery of solvents and volatile liquids. 1500. Jan. 26.

Lynde. Mechanism for vibrating and consolidating powders or plastic material, etc. 1106. Jan. 19.

McClellan. Separating oil from volatile refrigerating-fluid. 1392. Jan. 24.

Morison. Means for maintaining vacuum in condensing plant. 1020. Jan. 18.

Morris. Apparatus for extracting aqueous, etc., vapours from thick or viscid liquids by means of a vacuum. 1718. Jan. 30.

Prigleau. Apparatus for drying moisture-laden substances before distillation. 801. Jan. 14.

Rasch. Centrifugal machines. 875. Jan. 15.

Rothwell. Extracting vapours from stills, boiling and vacuum pans, etc. 1084. Jan. 19.

Smallwood. Furnaces. 1925. Feb. 2.

Soc. l'Air Liquide. Effecting exothermic chemical synthesis under pressure and at a high temperature. 1007. Jan. 17. (Fr., Mar. 31, 1917.)

Soc. l'Air Liquide. Process of effecting exothermic chemical syntheses. 1072. Jan. 18. (Fr., Aug. 7, 1917.)

Soc. d'Exploit. de Procédés Evaporatoires Système Prache et Bouillon. Evaporating and crystallising apparatus for obtaining large crystals, etc. 1171. Jan. 21. (Fr., Jan. 23, 1917.)

Stansfield. Treating smoke, furnace gases, exhaust gases, etc. 1523. Jan. 26.

### COMPLETE SPECIFICATIONS ACCEPTED.

151 (1917). Spratt. Means for separating solid from liquids. (112,791.) Feb. 6.

285 (1917). Semmler. *See under X.*

827 (1917). Marshall. Apparatus for cooling and drying compressed materials and removing dust therefrom. (112,512.) Jan. 23.

1192 (1917). Sepulchre. Separation and recovery of liquids of different densities and separation of light solid particles from a heavy liquid. (103,670.) Jan. 30.

1557 and 2484 (1917). Hall. Furnaces and the like. (112,827.) Feb. 6.

4562 (1917). Rothenbach. Apparatus for the fractional uninterrupted distillation of liquid mixtures. (106,088.) Jan. 23.

12,646 (1917). Wallwin. Gas-heated furnaces or muffles. (112,747.) Jan. 30.

16,389 (1917). Mathys. Apparatus for cooling and otherwise treating gases with liquids. (111,113.) Feb. 6.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Adams and Greenwood. 1383. *See under VII.*  
Baker. Eliminating sulphur from oils and spirits. 925. Jan. 16.

Barclay. 1495. *See under I.*

Bouillon. Process for dehydrating peat. 987. Jan. 17.

Burdon, and Burdons, Ltd. Oil-gas, etc. furnaces. 966. Jan. 17.

Burdon, and Burdons, Ltd. Liquid-fuel furnaces. 1042. Jan. 18.

Comp. pour la Fabr. des Compteurs et Matériel des Usines à Gaz. Manufacture of producer gas. 1159. Jan. 21. (Fr., Jan. 31, 1917.)

Comp. Générale de Construction des Fours, and Rivière. Eliminating slag in gas-producers. 982. Jan. 17. (Fr., Jan. 19, 1917.)

Glover and West. Destructive distillation of coal, etc. 1815. Jan. 31.

Hiller. Apparatus for low-temperature distillation of coal, etc. 1746. Jan. 30.

Leadbeater. Obtaining carbon from peat for decolorising. 1779. Jan. 31.

Litwin. Coke furnace for gas-generator. 1166. Jan. 21.

Lucas, and Palmer. Retorting systems for distilling coal, shales, etc. 806. Jan. 14.

Mascord. Vaporiser for internal-combustion engines applicable in manufacture of gas and distillation of hydrocarbons. 1343. Jan. 23.

Pease. 1053 and 1418. *See under VII.*

Penrose and Penrose. Manufacture of vegetable charcoal. 1276. Jan. 22.

Peters. Coke ovens. 1274. Jan. 22. (Fr., Feb. 28, 1917.)

Prigleau. 801. *See under I.*

Rebs. Process of purifying mineral oil or its distillates and concentrates. 1767. Jan. 30. (Ger., Jan. 30, 1917.)

Rollason. Retorts, and utilising therein the emanations from carbonates of alkaline earths to increase the yield of gases and hydrocarbons from coal during distillation. 1724. Jan. 30.

Russell. Coal-washing. 1170. Jan. 21.

Soc. l'Air Liquide. Extraction of hydrogen from gaseous mixtures. 1278. Jan. 22. (Fr., Apr. 16, 1917.)

Soc. l'Air Liquide. Manufacture of compressed hydrogen. 1533. Jan. 25. (Fr., Aug. 9, 1917.)

Soc. d'Exploitation des Appareils Râteau. Apparatus for production of water-gas. 1114. Jan. 19. (Fr., Jan. 24, 1917.)

Waite. Apparatus for obtaining gas from combustible materials. 1257. Jan. 22.



## COMPLETE SPECIFICATIONS ACCEPTED.

13,428 (1916). Bostaph Engineering Co. Process for transforming hydrocarbons into other hydrocarbons relatively poorer in hydrogen. (106,080.) Jan. 23.

18,295 (1916). Smith. Process and apparatus for treating coal. (106,084.) Jan. 23.

576 (1917). Bilbrough. Distillation of coal, oil shales, and other carbonaceous materials, and retorts therefor. (112,793.) Feb. 6.

862 (1917). Tollefsen. Production of fuel briquettes. (112,514.) Jan. 23.

11,478 (1917). King and Stoneham. Method of rendering alcohol more readily ignitable when used as fuel in internal-combustion engines. (112,741.) Jan. 30.

13,770 (1917). Thuman. Gas producers. (112,909.) Feb. 6.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Francis. 1501. *See under V.*  
Hintikka. Manufacture of *p*-nitrotoluol-*o*-sulphuric acid. 1004. Jan. 17.

Lessing. Separation of oils and pitch from tar. 1635. Jan. 28.

## COMPLETE SPECIFICATION ACCEPTED.

5568 (1917). Thuman (Evans). Manufacture of toluol and like hydrocarbons. (112,878.) Feb. 6.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATION.

Soc. Chem. Industry in Basle. Manufacture of chlorinated derivatives of 1,2,2'-anthraquinone-azine. 1663. Jan. 28. (Switz., Feb. 27, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

14,390 (1916). Malcomson. Dyestuffs and dyes. (112,474.) Jan. 23.

1228 (1917). Malcomson. Manufacture of dyestuffs and dyes. (112,813.) Feb. 6.

7105 (1917). Boake, Roberts, and Co., and Rendle. Preparation of colouring-bodies for use in food, pharmaceutical, and other preparations. (112,886.) Feb. 6.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Aitken. Paper-making machines. 832. Jan. 14.  
Francis. Tar proofing for canvas, sackings, ropes, etc. 1501. Jan. 26.

Johnston and Sutherland. Treatment of stalk fibres for removal of gum or mucilage. 904. Jan. 16.

Marks (Du Pont de Nemours and Co.). Production of pyroxylin-coated fabrics. 1180, 1182, 1183. Jan. 21.

Marks (Du Pont de Nemours and Co.). Solvents used in connection with pyroxylin compositions. 1181. Jan. 21.

Schmid. 805. *See under XII.*

## COMPLETE SPECIFICATIONS ACCEPTED.

204 (1917). Oman. Preparation of nitroso compounds from ligninesulphonic acid or its salts. (103,653.) Jan. 30.

869 (1917). Sanderson and Sanderson. Manufacture of coated paper or other fabrics. (112,660.) Jan. 30.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Ashton, Nelson, and Calico Printers' Association. Printing cotton fabrics or yarn. 1137. Jan. 19.

Bright and Bros., and Evans. Machines for boiling, bleaching, dyeing, sizing, etc., yarns or fabrics. 1225. Jan. 22.

Dudley. Dyeing machine. 1592. Jan. 28. (U.S., Jan. 31, 1917.)

Henshilwood and Thornber. Apparatus for use with machines for dyeing, bleaching, finishing, etc., fabrics. 846. Jan. 15.

Huebner. Machines for coating fabrics. 1519. Jan. 26.

Leather. Ltd., and Longthorne. Textile fibre printing processes. 1941. Feb. 2.

Marks (Du Pont de Nemours and Co.). Production of coated fabrics. 830. Jan. 14.

Marks (Du Pont de Nemours and Co.). 1180, 1182, 1183. *See under V.*

Poulson. Sizing textile fabrics, etc. 1585. Jan. 28.

Stutely. Means for producing ornamental effects on textile materials. 924. Jan. 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

17,497 (1916). Farrar and Whitehead. Dyeing machines. (112,482.) Jan. 23.

869 (1917). Sanderson and Sanderson. *See under V.*

9118 (1917). Mohs. Fulling and materials for use therein. (110,343.) Jan. 30.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Adams and Greenwood. Manufacture of hydrogen. 1383. Jan. 24.

Ashcroft. Manufacture of alkali-metal salts and by-products from alkali-metal alloys. 1628. Jan. 28.

Backström, Cederberg, and Kyhlberger. Fixation of nitrogen for producing ammonia or ammonium salts, etc. 1558. Jan. 26. (Sweden, Jan. 3, 1917.)

Backström, Cederberg, and Kyhlberger. Production of ammonia. 1559, 1560. Jan. 26.

Bailey, Denny, and Dunk. Production of ammonium perchlorate. 1058. Jan. 18.

Baker. Recovery of ammonium chloride from ammoniacal liquor with the extraction of cyanides, etc. 1211. Jan. 22.

Barbet et Fils et Cie. Producing chemically-pure oxygen and nitrogen from the air. 1698. Jan. 29. (Fr., Jan. 31, 1917.)

Bayeux and Richard. 933. *See under XI.*

Bertoya. Manufacture of alkaline tungstates and molybdenates. 1970. Feb. 2.

Blackier. Machine for breaking up caustic soda, etc. 1154. Jan. 21.

Boake, Roberts, and Co., and Durrans. Production of sulphuryl chloride. 1520. Jan. 26.

British Potash Co., and Chance. Method of obtaining potash compounds. 1887. Feb. 1.

Calvert. Production of cyanide and cyanogen compounds. 1248. Jan. 22.

Dutt and Dutt. Manufacture of magnesia and calcium chloride. 1708. Jan. 29.

Dzierzgowski and Polzeniusz. Production of nitrates. 1762. Jan. 30.

Eden. 815. *See under XVI.*

Ekeley and Stoddard. Extraction of acids of heavy acid-forming elements. 1390. Jan. 24.

Ekeley and Stoddard. Process for obtaining pure tungstates. 1391. Jan. 24.

Heap and Newbery. Manufacture of metal halides. 1677. Jan. 29.

Jacoby. Production of chlorosulphonic acid. 1622. Jan. 28.

Jones and Partington. Manufacture of ammonium nitrate. 1874. Feb. 1.

Marks. (Hüttenwerk Niederschöneweide A.-G. vorm. J. F. Ginsberg). Manufacture of copper compounds. 1817. Jan. 31.

Marks (Hüttenwerk Niederschöneweide A.-G. vorm. J. F. Ginsberg). Manufacture and use of copper protoxide. 1878. Feb. 1.

Mills, Packard, and Packard and Co. Chambers used in manufacture of sulphuric acid. 872. Jan. 15.  
Norske Aktieselskab for Elektrokemisk Industri. Manufacture of aluminium compounds poor in iron. 1403. Jan. 24. (Norway, Jan. 24, 1917.)

Pease. Extraction of ammonia from gases and vapours, and production of nitrogen compounds. 1053. Jan. 18.

Pease. Extraction of nitrogen from gases, and its conversion into nitrogen compounds. 1453. Jan. 24.

Soc. l'Air Liquide. Conversion of liquid synthetic ammonia into a solid product. 1713. Jan. 29. (Fr., Aug. 25, 1917.)

Soc. Industrielle de Produits Chimiques. Process for isolating salts of chromium. 1466. Jan. 25. (Fr., Aug. 2, 1917.)

Soc. Industrielle de Produits Chimiques. Transforming alkaline monochromates into bichromates. 1467. Jan. 25. (Fr., Aug. 31, 1917.)

Twynam. 1149. *See under X.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

266 (1917). Ehrhardt and Ehrhardt. Manufacture of anhydrous sodium hydrosulphites. (112,491.) Jan. 23.

1291 (1917). Tucker and Kelley. Reclamation of bichromates. (105,222.) Jan. 23.

6687 (1917). Llewellyn, Spence, and Spence and Sons. Production of aluminous compounds. (112,881.) Feb. 6.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Howard. Method of delivering glass from furnaces. 1186. Jan. 21.

#### COMPLETE SPECIFICATION ACCEPTED.

2437 (1916). Koepf und Co. Enamels for enamelling iron. (100,777.) Jan. 31.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Brazier and Green. Roofing materials. 1969. Feb. 2.

Surie, and Suristone, Ltd. Insulating-material for cold storage. 992. Jan. 17.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Annable. Extraction of tin from its ores. 1271. Jan. 22.

Armstrong. Metallurgy of zinc. 1603. Jan. 28.

Ashcroft. Treatment of lead and zinc ores or products. 910. Jan. 16.

Ashcroft. 1628. *See under VII.*

Balfour, Bray, and British Oxygen Co. Treatment of iron ores, etc., for use as catalysts. 1337. Jan. 23.

Balfour. Treatment of graphitic, etc., refractory ores. 1446. Jan. 25.

Barfield and Wild. Indicating condition of steel, iron, or other magnetisable metal during heat treatment. 1905. Feb. 1.

Brookfield, Gaunt, White, and Tylor and Sons. Smelting furnaces, etc. 802. Jan. 14.

Brookfield, Gaunt, White, and Tylor and Sons. Furnaces for smelting and refining metallic swarf, etc. 803, 804. Jan. 14.

Donald. Open-hearth, etc., metallurgical furnaces. 1229. Jan. 22.

Fletcher. Puddling iron. 920. Jan. 16.

Martin and Richards. Furnaces for roasting ores. 1174. Jan. 21.

Metalloids, Ltd., and Perry. Annealing or scaling apparatus. 981. Jan. 17.

Phillips. Means for protecting metals from destructive action of sea water. 1799. Jan. 31.

Rouse. Manufacture of iron and steel. 1557. Jan. 26.

Stabilimenti Biak, Ing. A. Pouchain. Manufacture of alloys containing nickel and zinc. 1710. Jan. 29. (Ital., Oct. 23, 1916.)

Thermalloy, Ltd., and Vautin. Ignitable metallic mixtures. 1003. Jan. 17.

Twynam. Recovery of potash salts in manufacture of pig iron. 1149. Jan. 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,996 (1916). Considere and Bethell. Apparatus for recovering tin from waste material. (112,471.) Jan. 23.

18,565 (1916). Maass. Zinc alloys. (112,487.) Jan. 23.

285 (1917). Semmler. Method of utilising the waste heat of slag. (112,792.) Feb. 6.

447 (1917). Gill (Nathan). Flux for use in brazing cast iron and other metals. (112,500.) Jan. 23.

843 (1917). Foster. Blast-furnaces. (112,513.) Jan. 23.

935 (1917). Cowper-Coles. Electrodeposition of copper. (112,516.) Jan. 23.

4955 (1917). Norsk Elektrisk Metalindustry Aktieselskab. Condensers for zinc vapours. (105,559.) Feb. 6.

5216 (1917). Hall. Melting furnace. (112,559.) Jan. 23.

5453 (1917). Lockwood. Obtainment of gold and silver from their ores. (112,876.) Feb. 6.

9169 (1917). Wild and Barfield. Electrical furnace for hardening steel and the like. (112,731.) Jan. 30.

9179 (1917). Wild and Barfield. Indicating the condition of steel, iron, or other magnetisable metal during heat treatment. (112,894.) Feb. 6.

10,532 (1917). Fisher and Fisher. Blast furnaces. (112,589.) Jan. 23.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Bayeux and Richard. Ozonising apparatus. 933. Jan. 16. (Fr., Feb. 5, 1917.)

Etchells and Greaves. Steadying the arc discharge in electric furnaces. 1012. Jan. 18.

Hornby. Electric accumulators. 1313. Jan. 23.

Johnson (Commercial Truck Co.). Secondary or storage cells or batteries. 1907. Feb. 1.

Marks (Hüttenwerk Niederschöneweide A.-G. vorm. J. F. Ginsberg). Electrolytic baths. 1879. Feb. 1.

Thorowgood. Renovation of electric batteries. 1219, 1220. Jan. 22.

White (Industrial Electric Furnace Co.). Electric furnaces. 1896. Feb. 1.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,439 (1916). Saville. Electrodes for storage batteries. (112,476.) Jan. 23.

935 (1917). Cowper-Coles. *See under X.*

9169 (1917). Wild and Barfield. *See under X.*

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Bellwood, Downs, and Turnill. Extraction of oil from vegetable seeds, nuts, etc. 1783. Jan. 31.

Harman. Soap or soap powder, etc. 1203. Jan. 22.

McComb and McComb. Detergents or washing-compounds. 1413. Jan. 24.

Meyer. Conversion of fatty acids with several double compounds or of their glycerides into oleic acid-like fatty acids or their soaps. 1644. Jan. 28.



Schmid. Manufacture of a substitute for sericin soap. 805. Jan. 14. (Switz., Sep. 4, 1917.)

Winters. Extracting oil from seeds, beans, nuts, offal, etc. 1412. Jan. 24.

Witter. Treatment of linseed oil residual fatty acids and utilising same as oil for paint or for oxidation for manufacture of linoleum. 1750. Jan. 30.

#### COMPLETE SPECIFICATION ACCEPTED.

10,586 (1917). Harrison (Bartlett and Snow Co.). Percolators for use in extracting oil by solvents. (112,590.) Jan. 23.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Bailey. Manufacture of anti-fouling paints. 1372. Jan. 24.

British Enallite Co., and Goldsmith. Production of dopes. 1554, 1555. Jan. 26.

British Thomson-Houston Co. (General Electric Co.). Manufacture and application of japan. 988. Jan. 17.

Craven, and Yorkshire Dyeware and Chemical Co. Linoleum and linoleum cement. 1600. Jan. 28.

Farnell. Composition for coating surfaces to protect them from atmospheric, etc., conditions. 1546. Jan. 26.

Reihl. Manufacture of paint oils, waterproofing oils, and lithographic varnishes. 1856. Feb. 1.

Wiedemann. Dopes, varnishes, paints, etc. 1382. Jan. 24.

Witter. 1750. *See under XII.*

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATIONS.

Carper and Tulloch. Solvents for india-rubber, etc. 1630. Jan. 28.

Dunlop Rubber Co., and Twiss. Reclaiming rubber. 980. Jan. 17.

#### COMPLETE SPECIFICATION ACCEPTED.

15,342 (1916). Lintner and Co. Process and apparatus for coagulating latex. (108,298.) Jan. 30.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Buchanan. Waterproofing-compound for leather and hides. 812. Jan. 14.

Dickson. Machine for facilitating treatment and handling of hides, skins, etc., in tanning, etc. 1668. Jan. 29.

Grime. Manufacture of gelatins, animal size, and glue. 1224. Jan. 22.

Marks (Du Pont de Nemours and Co.). Process for producing artificial leather. 1178, 1179. Jan. 21.

Smith. Substitute for leather. 1838. Feb. 1.

### XVI.—SOILS; FERTILISERS.

#### APPLICATIONS.

Brown, and Simon-Carves, Ltd. Apparatus for manufacture of artificial manures. 1793. Jan. 31.

Eden. Apparatus for manufacture of superphosphate, etc. 815. Jan. 14.

#### COMPLETE SPECIFICATION ACCEPTED.

1881 (1917). Lipman. Fertilisers. (104,174.) Jan. 30.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATION.

Anderschou, Lambshead, and Ramsay. Treatment of carbohydrates to render them suitable for fermenting or re-fermenting yeast. 1670, 1671. Jan. 29.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATIONS.

Anderschou. Treatment of brewers' yeast to render it suitable for baking purposes. 1325, 1326, 1327. Jan. 23.

Anderschou, Lambshead, and Ramsay. 1670, 1671. *See under XVII.*

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Anderschou. 1325, 1326, 1327. *See under XVIII.*  
Beeton, and Trufood, Ltd. Drying milk, etc., into powders. 1873. Feb. 1.

Ferguson and Gillespie. Food product and beverage. 770. Jan. 14.

Ferguson and Peyton. Method of food production, distribution, and preservation. 1943. Feb. 2.

Hildebrandt. Manufacture of meat extract, etc. 1816. Jan. 31.

Hunter, and Simon-Carves, Ltd. Settling tanks for clarifying liquids. 1734. Jan. 30.

Mapleton, and Mapleton's Nut Food Co. Preparation of foodstuffs. 1069. Jan. 18.

Rasch. Centrifugal milk-separators. 874. Jan. 15.

Scully. Manufacturing potatoes into potato flour. 775. Jan. 14.

Suric, and Suristone, Ltd. 992. *See under IX.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,138 (1916). Sherman. Antiseptic or sterilising agents for use in the preservative treatment of meat. (112,473.) Jan. 23.

404 (1917). Bart. Sterilising and preserving animal and vegetable products and preparation of inoculating substances and vaccines. (112,498.) Jan. 23.

1061 (1917). Candy. Filtration of water. (112,672.) Jan. 30.

1294 (1917). Evans. Obtaining pure water from salt water. (112,679.) Jan. 30.

4651 (1917). Heyl and Baker. Means for rendering water radio-active. (112,865.) Feb. 6.

7105 (1917). Boake, Roberts, and Co., and Rendle. *See under IV.*

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATION.

Haworth and Irvine. Preparation of alkyl sulphates. 1796. Jan. 31.

#### COMPLETE SPECIFICATIONS ACCEPTED.

204 (1917). Oman. *See under V.*

283 (1917). Momose. Preparation of antigens for tubercle bacilli. (103,298.) Jan. 23.

404 (1917). Bart. *See under XIX.*

1166 (1917). Imray (Soc. Chem. Industry in Basle.) Manufacture of therapeutically valuable compounds of the acridine series. (112,809.) Feb. 6.

2921 (1917). Parke, Davis and Co. Anaesthetic bodies. (105,745.) Feb. 6.

7105 (1917). Boake, Roberts, and Co., and Rendle. *See under IV.*

10,586 (1917). Harrison (Bartlett and Snow Co.). *See under XII.*

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATIONS.

James, Jones, and Lewis. Manufacture of explosives. 1517. Jan. 26.

Landrin. Manufacture of explosives. 1349. Jan. 23. (Fr., Nov. 13, 1917.)

### XXIII.—ANALYSIS.

#### APPLICATION.

Nielsen. Photometers. 1473. Jan. 23.

**I.—GENERAL; PLANT; MACHINERY.**

*Fusion apparatus; Notes on —.* F. Pope. Met. and Chem. Eng., 1917, 17, 704—709.

THE common type of fusion apparatus consists of a cast iron pot with agitator of the horse-shoe, gate, or other shape, but rarely of the cone or propeller type. The agitator and shaft should be cast in one piece, as trouble arises if bolts are used in positions exposed to the melt. Agitators for autoclaves should be strongly made, but the shaft should be as small as possible as it has to carry an unbalanced pressure, tending to push it out of the autoclave. In vertical steel autoclaves working at moderate pressure, the use of sloping shaft agitators has advantages. Bottom discharge can be most easily attained when the pot is gas-fired. In such cases ordinary valves and cocks are impracticable. A plug valve with seat and plug of a suitable metal in a horizontal run-off tube at the bottom of the pot can be used. An alternative method is to have the run-off pipe water-cooled, whereby it is closed by a plug of solidified charge, which melts when the water is drawn off and allows the pot to be discharged. The shaft should be lubricated with hard grease, not with oil. The design of safety valves and pressure gauges requires care to avoid their being plugged and rendered inoperative by the melt. Cast steel autoclaves should be cast bottom down with heavy sink heads to be machined off. Forged steel is better than cast steel for large autoclaves. When an enamelled lining is used, the space between it and the outer shell should be filled by running in molten lead or other suitable metal.—H. J. H.

**PATENTS.**

*Gases; Method and apparatus for separating — and the formation of products thereby.* A. Mond, London. From L. Bradley, East Orange, N.J., and W. A. Schmidt, Los Angeles, Cal., U.S.A. Eng. Pat. 112,153, Dec. 21, 1916. (Appl. No. 18,303 of 1916.)

SEE U.S. Pat. 1,221,505 of 1917; this J., 1917, 538. The process may be applied to the removal of chlorine from gases by injection of finely-divided calcium hydroxide; the removal of sulphur dioxide, ammonia, or the like from gases by injection of finely-divided charcoal; the separation of sulphur dioxide from air by injecting a finely-divided catalyst, such as iron oxide, chromium oxide, or the like, to convert it into sulphur trioxide; the drying of air for blast-furnaces by injecting a dehydrating agent, such as pulverised calcium chloride; and in the synthesis of ammonia from nitrogen and hydrogen by injecting an absorbing agent for the ammonia, e.g., phosphoric acid or boric acid, into the gaseous mixture.

*Filters; Cloth —.* W. Boby, London. Eng. Pat. 112,159, Dec. 22, 1916. (Appl. No. 18,394 of 1916.)

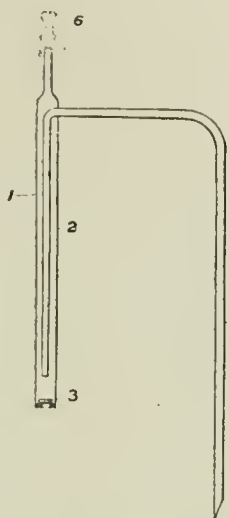
A PERFORATED tube is covered with a casing of filter cloth woven from fine wire or hair and is enclosed in an outer tubular casing. The liquid to be filtered is forced into the casing and the clear liquid passes through the cloth into the interior of the perforated tube to the exit. The filter is cleaned by reversing the flow. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 9206 of 1887, 16,996 of 1894, 18,716 of 1908, and 12,681 of 1910.)—W. H. C.

*Aerating liquids; Machines for —.* C. W. Wallace Bruce and Co., Ltd., and E. S. Jones, Newcastle-upon-Tyne. Eng. Pat. 112,165, Dec. 27, 1916. (Appl. No. 18,517 of 1916.)

THE supply of unaerated liquid to the mixing

chamber is controlled by a float in the reservoir of aerated liquid, so that when the level of liquid in the reservoir reaches a predetermined height the supply of unaerated liquid is cut off.—W. H. C.

*Syphons for corrosive and other liquids.* P. J. Channon, Dornoch, Dumfries, Eng. Pat. 112,188, Feb. 13, 1917. (Appl. No. 2160 of 1917.)



THE leg, 2, of the siphon is inserted into the reservoir of liquid, whereby the liquid raises the valve, 3, and partly fills the space between the tubes, 1 and 2. By blowing through the mouth-piece, 6, with the tap open, the liquid is forced up the tube, 1, and starts the siphon, the valve, 3, closing by the pressure. The tap, 6, is then closed and the siphon continues to act.

—W. H. C.

*Pulps; Process of replacing solvents in —.* G. Moore, Joplin, Mo. U.S. Pat. 1,248,374, Nov. 27, 1917. Date of appl., Mar. 3, 1915.

IN a process of replacing the solvent in pulps, the solids are allowed to settle from the slimes and are then moved in the form of a settled mass, in one direction in the replacing liquid, the latter moving in the opposite direction, but having its motion retarded at the surface so as to form a practically quiescent area into which the lighter solids rising through the advancing liquid are directed. At intervals the advancing liquid is divided into a number of horizontal layers and the solids which separate in the several layers are returned to the mass of settled solids.—J. H. P.

*Centrifugal machine.* E. R. Curtis, Guildford. Eng. Pat. 112,339, Feb. 12, 1917. (Appl. No. 2107 of 1917.)

SEE U.S. Pat. 1,248,821 of 1917; this J., 1918, 46 A.

*Apparatus for mechanically separating materials.* Eng. Pat. 112,227. See 11A.

*Outlet for acid receptacles.* U.S. Pat. 1,249,728. See VII.

*Gas-fired furnaces for pottery and other purposes.* Eng. Pat. 112,365. See VIII.

**IIA.—FUEL; GAS; MINERAL OILS AND WAXES.**

*Coal storage; Modern methods of —.* G. F. Zimmer. Engineering, 1918, 105, 30—32, 57—60.

ESTABLISHMENTS depending upon rail service



require to store at least 33% of their annual coal consumption; those depending on water carriage should store 66%, and a stock equal to 6 or 8 weeks' consumption should be kept even by works situated near collieries. The theory that iron pyrites and marcasite are factors in spontaneous heating of coal is not borne out by experiments though it is possible that they may indirectly help through the production of ferrous oxysulphate. Bruigum (Het Gas; through Gas World, June 24, 1916) states that humin and humic acid are the dangerous constituents. Humin, which is the residue left when the coal has been extracted with benzene or the like to remove bitumen, and then with caustic soda solution, to remove humic acid, consists principally of cellulose, and undergoes gradual combustion during weathering. Humic acid will take up much free bromine and also ozone, the latter forming ozonides, which are decomposed in the presence of moisture with formation of carbon dioxide and liberation of heat. Coal absorbs a good deal of oxygen but holds it simply as an absorbed gas, and chemical combination is very slow at ordinary temperatures, but as the heat increases so does the absorption of oxygen, and progressively rising temperatures result. For an effective start, however, ozone must be present, and this is always formed when water evaporates; consequently a damp heap of coal commences weathering, and if circumstances are favourable the temperature begins to rise, and the hitherto inactive oxygen helps in the chemical deterioration. Hence warm weather after rain, and damp sea air laden with ozone are dangerous, especially in a ship's bunkers. Compression and dumping from a height are also causes of rise of temperature and hence sources of danger. The spaces between "large" and "nut" coal provide channels for the air and so facilitate oxidation. During the development of heat, a vapour escapes from the stack and certain acetylenic gases of distinctive odour are evolved, perceptible at a considerable distance. As coal is a poor conductor of heat and has a low specific heat, its temperature may rise rapidly in parts where no cooling air currents can penetrate. When a fire is once established in a coal pile, it is almost impossible to extinguish it with water or steam, the only plan being to dig it out, though in a hoppers bunker it may be quenched by flooding. Chemical extinguishers containing ammonium carbonate are better than water; in some cases gas-works ammonia liquor is effectual. Screening of coal to eliminate smalls and dust before storage is very advantageous. Air penetration is beneficial in a stack, if abundant, but detrimental once heating has taken place. Coal recently mined or freshly broken heats more readily than older coal and it may be advisable to re-handle such coal after two months' stacking. According to Parr (see this J., 1911, 408) any method of storage to be successful must either check or prevent the absorption of oxygen to such an extent that the generation of heat shall not proceed faster than the dissipation and loss of heat due to absorption or radiation. The actual height of storage with safety depends on the character of the coal. The installation of steam pipes is compulsory in American vessels carrying coal, and the distribution of cylinders of compressed carbon dioxide with fusible plugs throughout a cargo has been suggested. The height of the pile is not such an important factor in silos with bottom withdrawal as in stack heaps on the ground. Where site values are high storage in heaps as high as possible is important, though 15 to 20 ft. is a maximum for safety. The kind of coal and method of deposition, however, must be considered. The practice in some naval yards is to stack 14 to 16 ft. high, but in some cases a 34 ft. stack has been found quite safe. At the English gas works in Berlin 60,000 tons of English

coal has been regularly stacked in piles of 50 ft., whilst German coal stored in piles of 16 to 26 ft. has heated, and in some cases caught fire. In any case, all coal stored in the open depreciates in calorific value, the more so with soft and friable qualities.

White (this J., 1917, 24) found that the chemical change produced in Pittsburgh and Fairmont gas coal which had been kept in the open for five years was too small to be recognised by analysis. Storage in the open for 3½ years has been found to have practically no effect on the gas-making properties of the coal, but has a deleterious influence on the quality of the coke (see this J., 1914, 1144). "Pocahontas" coal stored four years under cover showed a loss of calorific value to the extent of only 0.7%. Tests made by the Bureau of Mines on behalf of the U.S. Navy proved that the deterioration of a properly-stacked coal is quite insignificant (see this J., 1912, 112). Porter, in a report to the Department of Mines in Canada, states that the initial temperature of coal at the time of storage is important, also the loss of coal from fire in the second year of storage is about half that in the first year, due to protection offered by the older surface. The Bureau of Mines, at the instance of the U.S. Navy, found that coal stored for two years in fresh water lost nothing, in salt water 0.395%; indoors exposed to air, 0.383%; outdoors uncovered, 0.997%.

Methods usually employed in storing are:—(1) An ordinary pile, open or covered, sides generally open; (2) in pockets or silos, open at the base and capable of drainage; (3) under water. Method (2) is more rational than (1) and prevents the accumulation of small coal, and the coal at the deepest part of the pile is kept sufficiently in motion for the bulk to be slightly broken every time coal is withdrawn, thus minimising spontaneous heating and making admissible a higher pile; a large outlet is an advantage because of ventilating and cooling and also preventing bridging of large pieces likely to cause blockage. Method (3) is probably the most expensive, but in the end the most economical; fresh water is quite as effectual as sea water. At Chicago 12 pits 80 ft. by 25 ft. by 16 ft. deep were employed, holding together 14,000 tons of coal under water without apparent deterioration; the largest plant for this purpose is at Brimot's Island, Pittsburgh, consisting of a pit of ferro-concrete 800 ft. long, 150 ft. wide, and 25 ft. 6 in. deep, with sides sloping at 45°, which will hold a maximum of 100,000 tons of coal. Coal recovered from the battleship "Maine" which had been submerged 14 years, showed a heating value of 8588 calories and had deteriorated 160 calories, or 1.9%.—T. H. B.

*Hydrogen; Absorption of — by sodium oleate. Reagents for use in gas analysis. VI. I. R. P. Anderson and M. H. Katz. J. Ind. Eng. Chem., 1918, 10, 23—24.*

BOSSHARD and Fischli (this J., 1915, 1079) suggest the use of a solution of sodium oleate containing nickel in suspension for the absorption of hydrogen in gas analysis. The authors prepared metallic nickel by the reduction of nickelous oxide by hydrogen at 340° C., keeping the metal out of contact with air after production, and they employed a 10% solution of sodium oleate for most of their experiments, stronger solutions solidifying too rapidly to be of use. They found that absorption of hydrogen by the above reagent was very incomplete under all the conditions they employed, and they gave up the attempt to find conditions of complete absorption, finding that the method had several objections which render it of little value.—L. A. C.

*Benzene vapour; Determination of* —. *Reagents for use in gas analysis.* VII. R. P. Anderson. *J. Ind. Eng. Chem.*, 1918, 10, 25–26.

THE author summarises various methods for the determination of benzene vapour in gas, and suggests a method in which a measured quantity of gas containing benzene vapour is placed in contact with benzene in a special apparatus, and the increase in volume read. By determining what the increase would have been had there been no benzene vapour present, the amount actually present can be determined. A standard apparatus has not yet been produced.—L. A. C.

*[Oil] refining process; The Trumble* —. N. W. Thompson. *Amer. Soc. Mech. Eng.*, Dec., 1917. *Engineering*, 1918, 105, 63–65.

THE crude oil enters the plant through a 6 in. main, passing first of all through six coolers. These are of the horizontal tubular type, 30 in. in diameter, with sixty-two 2 in. by 18 ft. tubes. The oil, entering at the bottom, flows through the tubes, making four passes, and leaves at the top, passing on to four heat-exchangers, each 48 in. in diameter and containing 178 tubes 2 in. by 18 ft. These heat-exchangers are connected in series, and the oil, on leaving the last, passes on to the heater pipes. Here the flow of oil is split in two, each half passing in series through 72 lengths (18½ ft.) of 4-in. pipe, flowing backwards and forwards, but upwards at all times, and then into the top of the evaporator. Both the heater pipes and the evaporator are heated by flue-gases. The evaporator consists of a closed vertical cylindrical shell. Inside is a central vertical pipe having umbrella-shaped devices ("spreader hoods") attached thereto at intervals. The oil, fed to the apex of these hoods, flows down over their sides in a thin film, striking against the interior wall of the evaporator, which is separated from the lower edges of the hoods, and flowing down the wall in a thin continuous film. The central vertical pipe is provided with perforations under each of the hoods, through which the vapour passes off. Lateral connections through the walls of the chamber convey the vapour through an oil separator similar to a steam separator, the oil passing back to the bottom of the evaporator. The vapour then passes through six large dephlegmators in series, a system of water circulation serving as a cooling medium. From some of these, the condensed liquid is a finished product; from others it is an intermediate product. The residuum in the bottom of the evaporator is drawn off at a constant head and is used as a heating medium in the separators and heat-exchangers, through which it flows counter-current to the crude oil. On leaving the last of the series, it passes on to storage tanks. At the bottom of the evaporators, separators, and dephlegmators are superheated steam coils to agitate the liquid and expel the lower boiling-point fractions. The separators are re-run stills for distillates from the bottoms of the dephlegmators, or from any other source, which need fractionating. The distillate flows through a series of six compartments over pipes through which there is a regulated flow of a suitable heating medium, e.g., evaporator residuum. The vapours generated pass off through openings at the top of the compartments and are condensed. The following advantages are claimed for this process: The oil while being vaporised is never exposed to any action tending to "crack" the constituents. This is particularly advantageous in the production of asphaltum for road work. In passing through the apparatus the oil is not subjected to direct heat for any period of time, nor in the quiescent state, thus preventing burning and cracking. By regulating the fires and flow of oil, a uniform product can be obtained in any

quantity. The system is economical, only 1.1% of the crude oil being used as fuel and the loss of non-condensable gases being only 0.75%. The amount of labour required is small.—L. A. C.

#### PATENTS.

*[Coal:] Apparatus for mechanically separating materials* [—from rock]. A. Lotozky, Charkoff, Russia. *Eng. Pat.* 112,227, May 4, 1917. (*Appl. No.* 6372 of 1917.)

IN an apparatus for separating coal from rock mechanically, a number of polished discs, spaced apart, are mounted on a rotating shaft inclined to the vertical at an angle which is between the angles of friction of the coal on the discs at rest and in motion. The mixed coal and rock are fed on to the discs from tangential shoots inclined downward, parallel to the plane of the discs, and on that side of the discs which is moving in the opposite direction. The mixture is fed into the shoots from a box having distributing partitions, which is suspended by hangers and vibrated by a rotating ratchet below, acting through a spring which may be adjusted to vary the vibration. The coal is not subject to centrifugal force due to the rotation of the discs, but moves downward directly to the outlet at the lower edge of the discs. The rock, owing to its higher coefficient of friction, is carried round by the discs to an outlet opposite the coal outlet. To prevent the rock from being carried past its outlet, a wooden tongue is provided sliding freely between two parallel metal strips, resting freely on the disc, and projecting inwards from the further edge of the outlet. The inclination of the whole apparatus may be adjusted, by means of a worm gearing, about a horizontal axis coinciding with that of the driving shaft which operates the discs through bevel gearing.—W. F. F.

*Fuel; Manufacture of composition* —. C. T. Hopkins, Walthamstow, Essex. *Eng. Pat.* 112,341. (*Appl. Nos.* 2233, Feb. 14, and 10,517, July 21, 1917.) *Addition to Eng. Pat.* 14,771, Oct. 19, 1915 (this J., 1916, 826).

A MIXTURE of small coal and soot, with or without other ingredients, is mixed with water to a semi-liquid condition, allowed to stand for not more than 30 mins., and the floating material skimmed off. The liquid carrying the lighter suspended solids is decanted and allowed to settle for 24 hours, and the clear liquid then drawn off. This liquid may be used as a fertiliser, or as a pickling bath for iron and other metals preparatory to electrolytic treatment. The fine sediment dries into solid blocks without pressure and may be used as a fuel, or, with the addition of water, may be used as a binder for coal, coke, cinders, and the like. The coarse residue from the original mixture may be moulded alone or mixed with the binding material described above, and used as a fuel.

—W. F. F.

*Furnaces for the combustion of pulverulent fuel.* J. E. Muhlfeld, Scarsdale, N.Y., and V. Z. Caracristi, Bronxville, N.Y., U.S.A. *Eng. Pat.* 112,155, Sept. 13, 1915. (*Appl. No.* 18,306 of 1916.)

IN an apparatus for burning pulverulent fuel, the mixed fuel and air are injected tangentially at one end of a horizontal cylindrical preliminary combustion chamber, the nozzle being inclined to the axis of the chamber so as to produce a helical stream. The angle of the fuel jet may be varied so as to vary the time during which the fuel is in the chamber, to suit different varieties of fuel. A trough may be provided in the bottom of the chamber to collect any solid combustion products, which are then blown out by a jet of compressed air. The burning fuel passes into a main



combustion chamber and thence to a flue. In a modification, the fuel is injected downwards along the inclined bottom of the combustion chamber and then passes upwards through the chamber in a tortuous path formed by two baffles projecting alternately from opposite sides of the chamber and extending about halfway across. A zone of retarded flow is thus produced between two zones of accelerated flow, and an additional supply of air is admitted into this middle zone. This may be done by openings in the walls, or by making the projecting baffle hollow and communicating with the atmosphere. The combustion products then pass through any desired heating apparatus, such as boiler tubes, to the flue. The apparatus is especially suitable for use with the apparatus for feeding pulverulent fuel described in Eng. Pat. 13,067 of 1915 (this J., 1917, 200) and is described in detail as applied to a locomotive furnace.

—W. F. F.

*Aromatic bodies; Apparatus for the manufacture of — from petroleum oils.* F. W. Mann, Berkeley, and M. L. Chappell, El Segundo, Cal., Assignors to Standard Oil Co., Richmond, Cal. U.S. Pat. 1,249,444, Dec. 11, 1917. Date of appl., May 3, 1917.

THE oil to be treated, mixed with the necessary volume of air for the desired reaction, is supplied to a converter containing a contact material. A supply of liquid fuel is burnt within the converter under such conditions as to produce its complete combustion and maintain the reaction temperature, and the reaction products are discharged to the suction side of a compressor which maintains a pressure below atmospheric in the converter. The compressor delivers the vapour to a condensing system. A controllable outlet is provided in the converter on the side of the contact material opposite to the air supply inlet.—W. F. F.

*Petroleum oils; Double evaporator and process of treating —.* M. J. Trumble, Los Angeles, Cal., Assignor to Simplex Refining Co. U.S. Pat. 1,250,052, Dec. 11, 1917. Date of appl., Dec. 5, 1914.

THE oil is heated to a moderate temperature in an evaporator, and the vapour drawn off. The residue is transferred to a second evaporator where it is heated to a higher temperature, and the residue and vapour drawn off. The latent heat of the latter vapour is recovered by passing it through a heating compartment in the first evaporator, for the preliminary evaporation of the oil.—W. F. F.

*Coal; Process of and apparatus for treating —.* C. H. Smith, Short Hills, N.J., U.S.A. Eng. Pat. 106,084, Dec. 21, 1916. (Appl. No. 18,295 of 1916.) Under Int. Conv., May 5, 1916.

SEE U.S. Pat. 1,224,424 of 1917; this J., 1917, 636.

*Coal-gas; Manufacture of —.* A. Waddell, Dunfermline, Scotland. U.S. Pat. 1,249,864, Dec. 11, 1917. Date of appl., Feb. 29, 1916.

SEE Eng. Pats. 5611 and 14,142 of 1915; this J., 1916, 625.

*Hydrocarbons; Process of transforming — into other hydrocarbons relatively poorer in hydrogen.* Bostaph Engineering Co., Assignees of A. S. Ramage, Detroit, Mich., U.S.A. Eng. Pat. 106,080, Sept. 21, 1916. (Appl. No. 13,428 of 1916.) Under Int. Conv., Apr. 27, 1916.

SEE U.S. Pat. 1,224,787 of 1917; this J., 1917, 637. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 15,049 of 1913; this J., 1914, 806.)

*Recovery of ammonium chloride from ammoniacal liquor, with extraction of cyanides and other valuable products.* Eng. Pat. 112,329. See VII.

*Gas-fired furnaces for pottery and other purposes.* Eng. Pat. 112,365. See VIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Characteristics of sperm oils used for burning purposes.* Blakeley and Reilly. See XII.

*Preparation by the distillation and by the combustion of wood of acid coagulants and their effect on the quality of rubber.* Eaton and Whitby. See XIV.

### PATENTS.

*Electric [incandescence] lamp or other vacuum tube having a copper leading wire.* T. Yanai, G. Ohtani, and S. Yago, Tokyo, Japan. Eng. Pat. 103,648, Jan. 2, 1917. (Appl. No. 90 of 1917.) Under Int. Conv., Jan. 26, 1916.

COPPER wire, to be used as the leading-in wire of an electric lamp or other vacuum tube, is first hammered or rolled to a suitable form to give a great surface area, and then coated with a layer of oxide on its surface by heating or by immersion in an acid or alkaline oxidising bath. The wire is thrust through a heated glass rod, and the whole afterwards heated to a high temperature to bring about combination between the oxide and the glass.—B. N.

*Process for the purification of crude alcohols.* Eng. Pat. 112,166. See XX.

## III.—TAR AND TAR PRODUCTS.

*Tars; Effects of certain solvents on the determination of "free carbon" in —.* G. S. Monroe and H. J. Broderson. J. Ind. Eng. Chem., 1917, 9, 1100—1101.

TARS were left in contact with various solvents for different periods of time, and the amounts of "free carbon" determined by Weiss's method (this J., 1914, 474). The results showed a gradual increase with the time when benzene, bromobenzene, and chloroform were used. In other experiments the tar was digested with the solvent on the water-bath for definite periods, and then extracted with the same solvent, and the insoluble residue dried and weighed as free carbon. In the case of water-gas tar and gas-works tar the amounts of free carbon gradually approached a maximum, and then decreased, when benzene was used as solvent. In like manner, there was a gradual increase in the free carbon on continued digestion of gas-works tar with toluene, but in the case of coke-oven tar no maximum point was reached. In the digestions with chloroform no maximum point was reached in the case of any of the tars, although there was a gradual rise in the percentage of free carbon. Fluctuations in the boiling points of the solutions during the digestion indicated the probability of the occurrence of chemical reactions during the digestion of tars with chloroform, but these would not account completely for the gradual increase in the free carbon residues on continued digestion. Possibly free carbon present in colloidal form may be precipitated by dilution. Benzene, toluene, and carbon bisulphide do not react chemically with tar, and should therefore be preferred to chloroform for the determination of free carbon.—C.A.M.

*Naphthalene; Solidifying point of —.* G. V. Heyl. Met. and Chem. Eng., 1917, 17, 681.

A SOLIDIFYING point lying between 79° and 80° C., or a few decimal points above, is not always a true

indication of the purity of refined naphthalene. It is of as little value as proof of the absence of phenoloids, etc., as the "acid test." Naphthalene refined from flue-gas tar always shows a characteristic "acid test," although no traces of phenols or cresols are present. It is important that the refiner should furnish the dye manufacturer with a purer material. The failure of the latter to produce certain dyestuffs successfully is largely due to impurity of the prime materials, particularly in the case of naphthol and amine dyes. Samples of naphthalene of the same solidifying point and of nearly the same "acid test," but refined from different tars, may give entirely different yields on sulphonation or conversion to  $\beta$ -naphthol. All traces of aliphatic hydrocarbons must be removed from naphthalene produced from water gas tar. On subliming naphthalene containing these bodies, a pure white brittle flake is obtained giving a fair "acid test," but with a solidifying point not above  $79.3^{\circ}\text{C}$ .; this material gives very unsatisfactory results on nitration and halogenation.

—L. A. C.

*Phenol; Estimation of— in the presence of the three cresols.* G. W. Knight, C. T. Lincoln, G. Formanek, and H. L. Follett. *J. Ind. Eng. Chem.*, 1918, 10, 9—18.

For the determination of phenol in commercial products it is necessary to remove completely hydrocarbons, xylenols and higher homologues, and bases. Hydrocarbons are effectively removed by diluting the original sample with two volumes of benzol before extracting with caustic soda. Bases are removed during the separation of the tar acids. For the separation of xylenols and higher homologues a special still-head has been devised. This consists of three 2-in. bulbs joined by two glass tubes  $\frac{3}{16}$  in. long and having a  $\frac{5}{16}$  in. aperture. The upper bulb is connected with a glass tube open at the top ( $\frac{1}{8}$  in. internal diameter and 3 in. long) having a side tube ( $\frac{1}{8}$  in. internal diameter) bent downwards at a suitable angle. The lowest bulb has a glass siphon tube ( $\frac{1}{8}$  in. internal diameter) joined to the side of the bottom of the bulb, bending at first upwards to about half the height of the bulb and then downwards, passing through and down the centre of another tube,  $4\frac{1}{2}$  in. long and  $\frac{1}{8}$  in. internal diameter along  $3\frac{1}{2}$  in. of the lower part and  $\frac{1}{8}$  in. inside diameter along 1 in. of the upper part, where it is joined to the lowest bulb. A Tirrell burner should be used for heating the distillation flask. One hundred grams of the sample is poured into a 500 c.c. separating funnel, and the flask rinsed out with 200 c.c. of benzol; 100 c.c. of 20% caustic soda is then added; the whole shaken thoroughly for two minutes, allowed to settle and the lower layer drawn off into another funnel of 600—700 c.c. capacity. The benzol layer is washed with three or four 100 c.c. portions of 20% caustic soda. The caustic soda extracts are washed with 30 c.c. portions of benzol until hydrocarbons are all removed. The combined benzol extracts are washed twice with 10 c.c. of 20% caustic soda and the washings added to the caustic soda solution. The solution is then just acidified with dilute sulphuric acid, keeping the temperature below  $40^{\circ}\text{C}$ ., and allowed to settle in a separating funnel. The lower layer is drawn off into another funnel, the acid water layer washed with successive portions of benzol, the mixed benzol extracts then being treated with 10 c.c. of saturated salt solution. The tar acids are poured into a 300 c.c. round-bottom Jena glass distilling flask fitted with a Wurtz still-head, the funnel being washed out with the benzol extract. Benzol and water are distilled off to  $170^{\circ}\text{C}$ . into a separating funnel. The still is then cooled and the still-head removed and rinsed out with benzol which is added to the distillate. This is saturated with salt, shaken separated, and the benzol com-

pletely extracted with 5 c.c. portions of 20% caustic soda solution. The extract is acidified and the tar acids which separate added to the distilling flask. The distillation is now continued, using the Knight still-head and a standardised thermometer accurate to  $\frac{1}{2}^{\circ}\text{C}$ . between  $170^{\circ}$  and  $210^{\circ}\text{C}$ . Correction must be made for emergent stem, the correction being  $0.00016N(T^{\circ}-t^{\circ})$  ( $N$  is number of degrees exposed,  $T^{\circ}$  and  $t^{\circ}$  the temperatures of the thermometer bulb and the exposed stem respectively). The distillate is collected in a 10 c.c. burette until the oil passes over clear, and then in a tared 100 c.c. cylinder up to  $193^{\circ}\text{C}$ . The water layer in the burette is saturated with salt and the tar acids added to the distilling flask, the distillation being continued to  $206^{\circ}\text{C}$ ., with a fresh receiver, at a rate of 0.5 to 1.0 c.c. per min. This portion is redistilled in another flask to  $201^{\circ}\text{C}$ ., the distillate being collected in the cylinder used for collecting to  $193^{\circ}\text{C}$ . The cylinder is again weighed and 4.5 grms. of the distillate mixed with 10.5 grms. of *o*-cresol (sp. gr.  $25^{\circ}/25^{\circ}$ ,  $G_0$ , and solidifying point,  $T_0$ , above  $28^{\circ}\text{C}$ .). The specific gravity  $25^{\circ}/25^{\circ}\text{C}$ . ( $G_{50}$ ) of the mixture is determined in a 10 c.c. Geissler pyknometer, and the solidifying point ( $T_{50}$ ), using a standard thermometer. The specific gravity at  $45^{\circ}/45^{\circ}\text{C}$ . ( $G_p$ ) and solidifying point ( $T_{sp}$ ) of the distillate mixed with phenol (of sp. gr. at  $45^{\circ}/45^{\circ}\text{C}$ .,  $G_p$ , and solidifying point,  $T_p$ , not below  $40^{\circ}\text{C}$ .), instead of with *o*-cresol are then determined. The percentage of phenol is then calculated from the formula: Per cent. phenol =  $100D[(T_0-T_{50})(0.366+0.702L_s)+(G_{50}-G_0)(2970-609L_s)]\div 30w$  where  $L_s=[1000(G_p-G_{sp})/(T_p-T_{sp})]-0.482$   $w$  = wt. of sample used and  $D$  = wt. of distillate below  $199^{\circ}\text{C}$ . This method does not give absolutely accurate results with all possible mixtures of the three isomers with phenol, but in all cases ordinarily met with in commercial practice, the probable error would amount to only a few tenths per cent.—L.A.C.

*Determination of benzene vapour. Reagents for use in gas analysis.* VII. Anderson. See IIA.

#### PATENT.

*Apparatus for the manufacture of aromatic bodies from petroleum oils.* U.S. Pat. 1,249,444. See IIA.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Testing of paper, textiles, etc.; A constant temperature and humidity room for —.* F. P. Veitch and E. O. Reed. *J. Ind. Eng. Chem.*, 1918, 10, 38—44.

THE room is 10 ft. 6 in. by 14 ft. 3 in. by 9 ft. 3 in. high. The walls and ceiling are 8 in. thick and consist of an outer 4-in. layer of ground cork, a thin partition, and two layers of sheet cork  $1\frac{1}{2}$  in. thick, the inner layer being cemented on the inside to prevent absorption of moisture. Air is drawn into one corner of the room near the ceiling and passes out through openings in a flue with hand-controlled dampers. Two fans in the room prevent stratification of the air. The temperature of the incoming air is regulated by steam and brine coils and the humidity by a steam valve, the coils and valve being regulated respectively by a thermostat and a hygrostat situated in the room.—L. A. C.

*Paper; Method for determining the absorbency of —.* E. O. Reed. *J. Ind. Eng. Chem.*, 1918, 10, 44—47.

A 4 in. square of blotting paper is placed over a beaker or tumbler and a 1 c.c. pipette supported vertically above it with the tip  $\frac{1}{2}$  in. above the centre. A pipette with a delivery time of ap-



proximately 4 secs. should be used. The pipette is filled with water or preferably with a standard ink and allowed to drain on to the paper, which should be dishd slightly. The blot should not extend to the sides of the supporting vessel. The average time in seconds of complete absorption for three experiments is recorded. This gives a relative value for the absorptive power of the paper which is claimed to be more accurate than either the mounting test or the measurement of zones formed by blots of ink. For copying paper, filter paper, and very thin blotting paper, 0.5 c.c. of water should be used.—L. A. C.

*Preparation of uniform collodion membranes for dialysis.* Farmer. See XXIII.

#### PATENTS.

*Silk and silk waste ; Process for ungumming* —. Gebr. Schmid, Basle, Switzerland. Eng. Pat. 107,966, June 20, 1917. (Appl. No. 8856 of 1917.) Under Int. Conv., July 10, 1916.

In the process described in Eng. Pat. 100,029 of 1916 (this J., 1916, 733) for the degumming of silk and silk waste, the substitution of a portion of the soap by certain silk wastes known as "gallettamini" or "pelettes" or by silk-worm chrysalides may be increased until as much as nineteen-twentieths of the quantity of soap usually employed has been thus replaced by these natural lather-forming substances. A suitable bath is prepared with 1 kilo. of soap and 10 kilos. of silk-worm chrysalides or gallettamini. The alkalinity of the bath may be increased, if necessary, by the addition of crystallised soda.—J. F. B.

*Fibrous materials ; Process for separating impurities from waste* —. J. J. Werst, Arnhem, Assignor to Naaml. Vennootschap Neo-Cellulose Maatschappij, Rotterdam, Netherlands. U.S. Pat. 1,249,575, Dec. 11, 1917. Date of appl., May 22, 1916.

SEE Eng. Pat. 105,055 of 1916 · this J., 1917, 868.

### VI.—BLEACHING ; DYEING ; PRINTING ; FINISHING.

*Bleaching powder ; Effect of light on solutions of* —. R. L. Taylor. J. Soc. Dyers and Col., 1917, 33, 246—250.

THE conclusions of Lunge and Landolt (J. Soc. Dyers and Col., Nov., 1885), and the character of the reactions has been investigated. Evolution of oxygen as noted by Foerster and Dolch (this J., 1917, 546) in the case of sodium hypochlorite was found to be relatively the greatest in a strong solution of bleaching powder kept in the dark, the solution in which the total oxidising power changed the least. A solution containing a relatively smaller amount of free lime bleaches more rapidly and is more liable to decompose. A solution containing 10 grms. of bleaching powder per litre was unchanged after keeping in the dark for 15 months; the amount of free lime has no influence on the stability of solutions kept in the dark. It is concluded that it is of great importance in keeping bleaching solutions for use to preserve them from the action of light.—F. W. A.

*Ferrocyanides and ferricyanides ; Mordanting action of* —. A. B. Clark. Communication No. 46 from Research Lab. of Eastman Kodak Co. J. Phys. Chem., 1917, 21, 776—784.

MORDANTING action is a function of the nature of the dye, of the acid and metal united in the mordant, and also probably of the concentration of the ions in the solutions, and possibly an effect of exposure to light, e.g., in the case of silver compounds; as in all dyeing processes, the concen-

tration of hydrogen and hydroxyl ions and of neutral salt has a great influence on the result. The results are also greatly affected by the physical state of the mordant, i.e., colloidal or crystalline, and are much complicated in case the mordant is in gelatin. The experiments described were conducted in a system containing only water, the mordant, the inorganic salt resulting from the action of the soluble metal and acid salts, the dyestuff, and a slight excess of acid salt to ensure uniformity. The dyestuffs selected represented the common groups from the standpoint of chemical classification, together with others representing special characteristics of the organic molecule. The mordanting power was tested by preparing suspensions of the insoluble ferri- or ferrocyanide of such molar concentrations as to show clearly the relative mordanting action; the various dyestuff solutions were added, and the amount of dyestuff in solution after the precipitate had settled out was roughly determined by comparison with standards. A relation exists between mordanting power of a metal and its position in the periodic table; the high-valent metals of high atomic weight form ferro- and ferricyanides of high mordanting power, with certain irregularities probably due to difference in physical state of subdivision. All ferricyanides act better as mordants for basic dyestuffs than for acid dyestuffs in presence of a slight excess of ferricyanide ion. All ferrocyanides act as mordants for basic dyestuffs in presence of an excess of ferrocyanide ion, with the exception of lead ferrocyanide, possibly due to formation of a basic salt, and thorium ferrocyanide, probably due to the heavy multivalent ion which tends to precipitate acid dyestuffs. Ferricyanides are better mordants than ferrocyanides. Mordanting power in presence of a slight excess of cyanogen compound is primarily a function of the molecular weight and valence of the metals. Secondary factors are the physical state of subdivision and molecular weight of the mordant. It is probable that the nature of the adsorption complex varies in most cases with the ionic concentration in solutions.—F. W. A.

#### PATENTS.

*Dyeing and bleaching machine.* T. Allsop and W. W. Sibson, Assignors to The Philadelphia Drying Machine Co., Philadelphia, Pa. U.S. Pats. (A) 1,246,835 and (B) 1,246,836, Nov. 20, 1917. Dates of appl., (A) Aug. 7 and (B) Sept. 18, 1916.

(A) IN a dyeing and bleaching machine, a rotary cylinder is mounted axially in a tank on an inlet pipe into which the treating liquor is introduced by a pump, an outlet pipe from the tank leading to the pump, whereby a continuous circulation of liquor is maintained. The liquor is heated whilst passing through the pump by the introduction of steam. (B) A closed steam pipe is contained in the portion of the perforated outlet pipe within the tank.—F. W. A.

*Dyeing and analogous purposes ; Machinery for use in* —. A. Secley, Rochdale. U.S. Pat. 1,249,677, Dec. 11, 1917. Date of appl., Mar. 16, 1916. SEE Eng. Pats. 4957 and 6024 of 1915; this J., 1916, 174.

### VII.—ACIDS ; ALKALIS ; SALTS ; NON-METALLIC ELEMENTS.

*Hydrochloric acid ; Detection and determination of small quantities of free* — in the presence of chlorides and other mineral or organic acids. M. Entat. Ann. Chim. Analyt., 1918, 23, 5—7.

THE electrometric titration method described previously (this J., 1917, 515) for the determination of sulphuric acid may be applied to hydrochloric

acid. The hydrochloric acid solution under examination should contain 2% of nitric acid and the titration made with *N/1* silver nitrate solution. The curve plotted from readings shows an inflexion at the point where all the free hydrochloric acid has been precipitated.—W. P. S.

*Formic acid or formates; Volumetric determination of — in the presence of hydroxides, carbonates, oxalates, and acetates.* F. Tsiropinas. *J. Ind. Eng. Chem.*, 1917, 9, 1110—1111.

Fifty c.c. of the solution containing from 0.5 to 1 gm. of formic acid is introduced into an Erlenmeyer flask, and, if alkaline, is acidified with dilute (1:1) sulphuric acid, and treated with 400 c.c. of chromic acid solution prepared by dissolving 50 grms. of pure sodium bichromate in 500 c.c. of water, adding 80 c.c. of pure sulphuric acid, and boiling for 5 mins. The flask is attached to a reflux condenser the upper end of which is connected with a graduated gas-measuring tube holding about 600 c.c., and having its lower end connected by means of rubber tubing with a levelling bulb. After the measuring tube has been filled with water to the zero mark, the contents of the flask are boiled for about 15 to 20 mins. until the volume of carbon dioxide in the tube is practically constant. The volume is then reduced to standard pressure and temperature, a correction made for the volume of air displaced by the stopper at the beginning of the determination, and the weight of carbon dioxide calculated into the equivalent amount of formic acid. In the presence of carbonates, bicarbonates, oxalates, and acetates, the solution, which should contain between 2.5 and 5 grms. of formic acid, is boiled for a few minutes, made alkaline with sodium hydroxide if bicarbonates are present, and treated with sufficient calcium chloride solution to precipitate the carbonates and oxalates. The filtrate and washings from the precipitate are made up to 250 c.c., and the formic acid determined in 50 c.c. of this solution. Acetates, which remain in solution, are not oxidised by the chromic acid solution. The average result obtained in test determinations of mixtures containing 0.5 gm. of sodium formate was 0.4982 gm.—C. A. M.

*Nebraska potash industry.* E. E. Thum. *Met. and Chem. Eng.*, 1917, 17, 693—698. (See also this *J.*, 1917, 595.)

A MORE detailed account of the Nebraska potash lakes and their exploitation is given. All the plants are working substantially as stated in the previous note. In one case preliminary evaporation by solar heat is employed. In the drying of the saturated brine, different expedients have been employed. In one plant the brine is run down an oil-fired rotary kiln as used for cement burning, and the salts accumulate in small nodules. In another works the brine is sprayed into the oil flame placed axially in the upper end of the furnace. Very efficient and almost instantaneous drying occurs and the salt is discharged in powder form. In another works the drying is carried to the stage of fritting and the salt is discharged as a hot clinker which is sprayed with saturated brine. This is evaporated by the heat of the clinker, which is disintegrated in the cooling process. Flue losses of potash in all cases are considerable. The Nebraska region is now producing potash salts (25%  $K_2O$ ) at the rate of 32,000 tons of  $K_2O$  per annum, whereas in 1916 the total production of the United States was only 9720 tons. The pre-war consumption was 275,000 tons.—H. J. H.

*Potash; Commercial aspect of recovery of — in pig iron manufacture.* K. M. Chance. *Cleveland Inst. Eng.*, Jan. 15, 1918. *Chem. Trade J.*, 1918, 62, 44. (See also this *J.*, 1917, 1006.)

NORMALLY there is from 15 to 30 tons of potash in

the burden charged into iron blast furnaces to produce 1000 tons of pig iron. At this rate 200,000 tons of potash is annually fed into the blast furnaces of this country and of this about 50,000 tons is carried out of the furnaces in the blast-furnace gas. This quantity can be increased by the addition of salt to the charge (see Eng. Pat. 112,338 of 1917; following) and so far as known at present without detriment to the furnace lining. From the dust deposited by the gas it is practicable to produce a potash fertiliser with only 5% of impurities, whereas the German kainit contained only about 20%  $K_2O$ . A very pure potash can also be prepared from the aqueous washings of wool, and there are still other sources available in this country. The exploitation of these would speedily enable the demand for home consumption to be met without importation. A company, half the shares in which are held by the Government, has been formed for the home production of potash, and one factory has already been erected at Oldbury. The author regards the commercial prospects of the industry as very favourable.—H. J. H.

*Potash; Recovery of — from greensand.* H. W. Charlton. *J. Ind. Eng. Chem.*, 1918, 10, 6—8.

FINELY ground greensand is digested in an autoclave with lime and water at a pressure of about 225 lb. for 2—4 hrs., high-pressure steam being admitted to maintain the required temperature and pressure. On completion, the contents are filtered, the filtrate containing potassium hydroxide in a relatively pure state. The residue, on the commercial utilisation of which the success of the method depends, may be successfully employed as a cementing agent in the manufacture of steam-hardened bricks, tiles, etc.; it may also be moulded and dried without admixture with sand to form insulating fireproof blocks. The method may also be employed for the extraction of potash from felspar, this material, however, giving a potash containing soda and alumina, which have to be separated. One ton of greensand is capable of producing about 100 lb. of  $K_2O$ , and cement for twenty to thirty thousand bricks.—L. A. C.

*Phosphate rock; Electric furnace smelting of — and use of the Collrell precipitator in collecting the volatilised phosphoric acid.* J. N. Carothers. *J. Ind. Eng. Chem.*, 1918, 10, 35—38.

WORK done previously (see this *J.*, 1917, 134) has been continued on a commercial scale. The furnace consisted of a water-cooled crucible lined with firebrick, but silica brick is recommended as likely to prove more satisfactory. The gas mains and cooling tower also had a firebrick lining. The graphite electrodes entered through the top of the furnace and were controlled by hand. A current of 220 volts was used for starting, but one of 110 volts was found satisfactory for operating. The  $P_2O_5$  content of the slag was approximately 2%, but it is possible to reduce it to 1.5 or even 1% with regular operation. The average production was 0.3 lb. of  $H_3PO_4$  per kilowatt-hr., but it should be possible to double this figure. The gases on leaving the furnace passed through a cooling tower so designed that they entered the treater at 250°—300°C. The treater contained a header of common brick and 20 treater tubes of vitrified sewer pipes, 2 in. in diameter and 15 ft. in length, all being enclosed at the top in a common hood. Supports for the conductors rested on insulators within the treater hood. The treater was capable of dealing with 2000 cub. ft. of gas entering at 300°C. with a velocity of 3 ft. per sec. It was found that 70 kilo-volts was sufficient to give complete precipitation of the suspended matter in the gases at the above-stated volume and velocity. As the acid fell from the pipes it was caught in a



receiving basin and removed. The concentration of the acid was controlled by the temperature of the gas in the treater; at less than 100° C., the concentration did not exceed 50%; at 250°–300° C., an acid of 85–93%  $\text{H}_2\text{PO}_4$  was obtained. Unscreened rock was found to be undesirable, as dust is likely to be carried over; with a dust-free rock, the only impurities likely to be present in the resultant acid are carbon, silica, fluorine, and arsenic.—L. A. C.

*Effect of light on solutions of bleaching powder.* Taylor. See VI.

*Method for the determination of fluorine, with special application to analysis of phosphates.* Wagner and Ross. See XXIII.

*Estimation of phosphoric acid as magnesium pyrophosphate.* Balareff. See XXIII.

*Transition temperatures of strontium chloride and bromide as fixed points in thermometry.* Richards and Yngve. See XXIII.

*Gravimetric determination of sulphuric acid and barium as barium sulphate.* Karaoglanow. (See XXIIJ).

#### PATENTS.

*Acid receptacles; Outlet for*—. J. J. Dye and W. A. Lucas, Newark, N.J., Assignors to Butterworth-Judson Corporation. U.S. Pat. 1,249,728, Dec. 11, 1917. Date of appl. June 12, 1917.

AN acid receiver of refractory material is provided on the outside with a box of similar material communicating with the receiver. An outlet pipe communicates with the box, and is provided with a flange at a point outside the box. The box is surrounded by a sheet of acid-proof metal, having a tubular portion which forms a sleeve to the outlet pipe, the end of the sleeve being clamped to the flange on the outlet pipe.—F. SP.

*Sodium hydroxide; Production of*—. C. S. Bradley, New York. U.S. Pat. 1,249,314, Dec. 11, 1917. Date of appl. Feb. 26, 1915.

AMMONIUM carbonate is formed by the action of barium carbonate on ammonium chloride, and from it sodium carbonate is produced by the ammonia-soda process, with reproduction of ammonium chloride. The sodium carbonate is causticised first with calcium hydroxide and then with barium hydroxide, the latter being produced from the barium chloride formed in the first reaction by means of a portion of the caustic soda produced in the causticising process.—F. SP.

*Ammonium chloride; Recovery of*—*from ammoniacal liquor, with extraction of cyanides and other valuable products.* H. Baker, Barrow-in-Furness. Eng. Pat. 112,329, Jan. 17, 1917. (Appl. No. 836 of 1917.)

AMMONIACAL liquor is boiled to remove free ammonia, which is recovered as liquid or gaseous ammonia, or as ammonium salts. The liquor is then treated with the oxide or chloride of copper, tin, silver, aluminium, or nickel, either singly or mixed, to remove cyanides as an insoluble precipitate. Excess of the reagents is precipitated by treating the liquor with hydrogen sulphide, or unpurified coal gas, or a mixture of the two. Ammonium chloride of moderate purity is then obtained by concentrating the liquor until it crystallises, or evaporating it to dryness.—F. SP.

*Potassium chloride; Production of*—*[in manufacture of pig iron].* British Cyanides Co., Ltd., K. M. Chance, and E. C. Rossiter, London, and North Lincolnshire Iron Co., Manchester. Eng. Pat. 112,338, Feb. 8, 1917. (Appl. No. 1960 of 1917.)

SODIUM chloride or an alkaline-earth chloride is

added to the ordinary blast-furnace charge in the manufacture of pig iron. The flue gases contain nearly all the potassium of the charge in the form of potassium chloride, which is extracted from the flue gases by a wet or dry cleaning process.—F. SP.

*Cement-kiln dust; Process for increasing the solubility of potash in*—. E. Anderson, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,249,708, Dec. 11, 1917. Date of appl. Mar. 14, 1917.

THE cement dust is exposed to the action of steam under pressure and at a temperature above 100° C., so as to convert the difficultly-soluble potash into a more soluble form. The product is then leached with water to extract the potash.—A. B. S.

*Thorium; Recovery of*—*[from monazite sand] as pyrophosphate.* Welsbach Co., Gloucester City, N.J., Assignees of M. A. Goltz, Winona, Minn., U.S.A. Eng. Pat. 112,380, June 14, 1917. (Appl. No. 8550 of 1917.) Under Int. Conv., Feb. 28, 1917.

THORIUM is obtained from monazite sand as pyrophosphate by treating the sand with concentrated sulphuric acid, heating the mass to a sufficiently high temperature to convert the orthophosphoric acid into pyrophosphoric acid, and treating the product with water. The pyrophosphates of the other rare earths remain in solution, and the insoluble thorium pyrophosphate, which contains small amounts of impurities, can be separated by decantation, filtration, etc.—F. SP.

*Lime mud [from ammonia-soda process]; Process of recovering values from*—. H. A. Galt, Akron, Ohio, Assignor to Columbia Chemical Co., Barberton, Ohio. U.S. Pat. 1,249,739, Dec. 11, 1917. Date of appl. Feb. 5, 1915.

LIME mud from the ammonia-soda process, whilst heated and under pressure, is passed into a tank to allow the steam to escape. The solid portion is dried, heated, and crushed, to produce a fertiliser, and the liquor is evaporated to obtain salts.—F. SP.

*Cyanogen compounds; Process of making*—. H. Philipp and H. Foersterling, Perth Amboy, N.J. U.S. Pat. 1,249,821, Dec. 11, 1917. Date of appl. June 4, 1912. Renewed May 11, 1917.

A METAL capable of forming cyanogen compounds is vaporised from a molten mass containing it, and the vapour is led into a separate chamber where it reacts with nitrogenous and carbonaceous matter; or the metal may be vaporised in a current of nitrogen, and the mixture of vapour and nitrogen led into a retort containing carbonaceous matter.—F. SP.

*Molybdenum compounds; Process of obtaining*—. E. H. Westling (Assignor to N. W. Stern) and E. Anderson, San Francisco, Cal. U.S. Pat. 1,250,063, Dec. 11, 1917. Date of appl. Oct. 9, 1916.

MOLYBDENUM is separated from solutions containing molybdic acid by adding a salt of a trivalent metal, e.g., iron, then adding an alkaline basic compound, if necessary, to neutralise the acidity and precipitate the molybdate of the trivalent metal, separating the precipitate, and digesting it with a soluble base, to produce a molybdate of the latter.—F. SP.

*Iodine; Process for the direct extraction of*—*from fresh or dried marine algae.* V. Vincent, Quimper, France. U.S. Pat. 1,249,863, Dec. 11, 1917. Date of appl. Dec. 26, 1916.

SEE Fr. Pat. 480,014 of 1915; this J., 1917, 137.

*Method and apparatus for separating gases and the formation of products thereby.* Eng. Pat. 112,153, See I.

## VIII.—GLASS; CERAMICS.

*Chemical glassware; Comparative tests of —.*  
P. H. Walker and F. W. Smither. J. Ind. Eng. Chem., 1917, 9, 1090—1092.

COMPARATIVE tests were made with five brands of American glassware and with specimens of Jena and Kavalier glass beakers and flasks. In the evaporation test, sodium chloride solution was repeatedly evaporated to dryness in the beakers. In no case were cracks produced after 12 evaporations. The beakers were also tested for extremes of temperature by filling them with cold water, which was then heated rapidly to boiling, and by plunging them into ice-water when filled with boiling water, and paraffin oil at 150° C., and at 200° C. As a shock test they were dropped bottom downwards upon a thick board from heights increasing by 5 inches. Solubility tests included boiling water in the beakers for 24 hours, and leaving it for 72 hours, and in the flasks boiling the water for 5 hours, and leaving it for 17 hours. Solutions containing sodium chloride and nitrate were mixed with a large excess of sulphuric acid, and boiled and heated for an hour after fumes of sulphur trioxide appeared. In the case of  $N/2$  solutions of sodium and potassium carbonates and hydroxides the boiling was continued for 20 mins., and fresh solutions were subsequently evaporated in the same beakers. Strong ammonia solution was allowed to stand for 24 hours in the vessel, and then diluted and boiled for 30 mins., whilst  $2N$ -solutions of ammonium chloride and sulphide were left for 24 hours and then boiled for 30 mins. The results, given in tabular form, showed that the American glassware was superior to Kavalier and equal or superior to Jena ware for laboratory purposes.—C. A. M.

## PATENTS.

*Crucibles for preparing fused quartz. Method of preparing crucibles for preparing fused quartz.* F. G. Keyes, Boston, Mass., Assignor to Cooper Hewitt Electric Co., Hoboken, N.J. U.S. Pat. (A) 1,249,636 and (B) 1,249,637, Dec. 11, 1917. Dates of appl., (A) July 1, 1913, (B) Apr. 8, 1915.

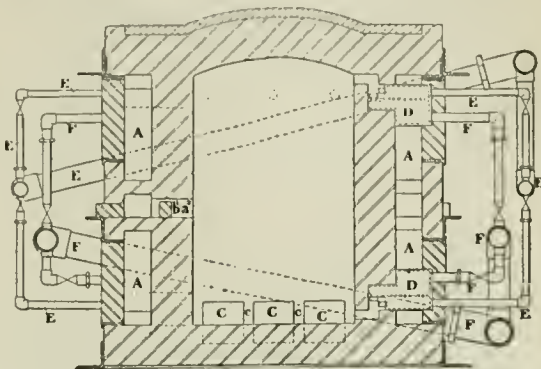
(A) A CARBON crucible is provided with a lining of tungsten or other refractory metal which is inert to fused quartz. (B) The lining is made by preparing a paste of sodium tungstate (or other suitable compound) and an appropriate binder, spreading this paste over the inner surface of the crucible, igniting the crucible and lining so as to harden the latter, and then applying a burnishing tool or roller so as to compress the lining and give to it a firm, tight, and compact surface.—A. B. S.

*Plastic material.* R. B. Ransford, London. From Soc. Française du Céramoïd, Paris. Eng. Pat. 112,196, Feb. 28, 1917. (Appl. No. 2971 of 1917.)

ZINC oxide is added in small quantities to a hot solution of gelatin or other similar material which solidifies on cooling, and a strong solution of calcium chloride, about 32° B. (sp. gr. 1.27), added while the temperature is prevented from rising above 60° C. A small quantity of sulphur may be added to make a more flexible material. The preferred proportions are anhydrous calcium chloride 10 parts, commercial zinc oxide 200 parts, glycerin 100 parts, washed sulphur 25 parts, gelatin or the like 2000 parts, water 1955 parts. A double oxychloride of zinc and calcium is formed within the gelatinous material. The material is specially suitable for moulding the heads and bodies of dolls, etc.—W. F. F.

*Gas-fired furnaces for pottery and other purposes.* H. J. Yates, Birmingham, S. N. and E. R. Brayshaw, Manchester, Eng. Pat. 112,365, Apr. 28, 1917. (Appl. No. 6008 of 1917.)

ONE or both of the side walls of a rectangular gas-fired furnace is provided with a regenerative chamber, A, in direct communication with the interior of the furnace and heated by the waste



gases. The gas burners, D, D, are placed inside the regenerative chambers, preferably in pairs at the top and bottom. The other gas and air fittings, E, F, are outside the chambers and are therefore kept cool. Access to any burner is readily obtained. The floor is constructed of blocks, C, C, C, to carry the saggars or goods to be heated; between these blocks are channels c, c, through which pass the flames from the bottom burners. Refractory blocks, b¹, may be fitted into the outlet passages, a², so as to regulate the flow of the waste gases.—A. B. S.

*Plastic composition [for retorts], and process of producing same.* J. D. Hollingsworth and J. Mitehem, Kusa, Okla. U.S. Pat. 1,249,960, Dec. 11, 1917. Date of appl., Jan. 4, 1917.

A PLASTIC composition for use in manufacturing retorts consists of fireclay 36 lb., fireclay cement 59 lb., graphite 14 lb., and common salt 1 lb. The salt is dissolved in water and the other ingredients are mixed together in a dry state and then added to the salt solution, the whole being mixed to form a mass of the consistency of dough. The paste is allowed to stand for "a substantial period" and is then made into retorts.—A. B. S.

*Enamels; Production of ground —.* P. Eyer, Halberstadt. Assignor to R. Koepf und Co., Oestrich, Germany. U.S. Pat. 1,219,937, Dec. 11, 1917. Date of appl., July 21, 1915.

SEE Eng. Pat. 107,392 of 1916; this J., 1917, 963.

## IX.—BUILDING MATERIALS.

*Plaster and cement; Mechanism of the setting process in —.* C. H. Desch. Faraday Soc., Jan. 14, 1918. [Advance proof.] 7 pages.

THE rival hypotheses of the setting processes are examined and compared. Le Chatelier's theory of the setting of plasters (this J., 1888, 675) is regarded as corresponding closely with the facts, but attention is drawn to the possibility that it is not quite complete, as indicated by the work of Cloez (this J., 1903, 366) and Davis (this J., 1907, 727). The oxychloride and certain other cements set by a similar process, i.e., crystallisation of an insoluble compound from a solution of the component bodies. The hardening of lime mortar



is a simple process of desiccation followed by carbonation. The setting of Portland cement is a more complex problem. Le Chatelier (this J., 1888, 847) holds that the mechanism is similar to that in the case of plaster. There is the alternative hypothesis, originally due to Michaelis (see this J., 1909, 836), that amorphous and gelatinous products are formed, the gradual desiccation of which leads to hardening of the cement. Further light has been thrown on the problem by the work of the U.S. Bureau of Standards (see this J., 1916, 1016) on the setting of the individual constituents, from which it appears that the physical nature of the hydrated product may depend on the concentration and so on the amount of water present. In practice the amount of water is restricted and favours the production of gelatinous substances, whereas in Le Chatelier's experiments water was in excess, whereby crystallisation was favoured.

—H. J. H.

*Portland cement; The setting and hardening of* — G. A. Rankin. Faraday Soc., Jan. 14, 1918. [Advance proof.] 6 pages.

THE author summarises the findings of the U.S. Geophysical Laboratory and the Bureau of Mines on the nature of the chief constituents of Portland cement and their influence on the setting process. The essential components of the clinker are  $\text{CaO}$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{SiO}_2$ , and a good cement can be made from the three pure oxides. In commercial cements other and minor constituents are present, but their influence on the setting is purely secondary. The study of the above mentioned ternary system shows that in a well-burned clinker three compounds are capable of a stable existence, viz.,  $2\text{CaO}.\text{SiO}_2$ ,  $3\text{CaO}.\text{SiO}_2$ , and  $3\text{CaO}.\text{Al}_2\text{O}_3$ . If the time and temperature of heating are insufficient, then free lime and  $5\text{CaO}.3\text{Al}_2\text{O}_3$  are also present. All these compounds have been identified in commercial clinkers and the first three form 90% of the whole. The hydration of the separate compounds was studied. Lime hydrates to an amorphous product which later crystallises.  $5\text{CaO}.3\text{Al}_2\text{O}_3$  with a little water sets rapidly, but disintegrates with excess, partly owing to dissociation and partly to its considerable solubility in water.  $3\text{CaO}.\text{Al}_2\text{O}_3$  with water, sets and hardens rapidly, but the product has little strength in water, being comparatively soluble. Probably there is first formed an amorphous hydrate which crystallises later.  $2\text{CaO}.\text{SiO}_2$  sets only after several months, but the product is not very soluble. Free lime is leached out by water, gelatinous silica being left. If sufficient water be taken, the decomposition can be carried to completion—a process which is accelerated by the presence of  $3\text{CaO}.\text{Al}_2\text{O}_3$ .  $3\text{CaO}.\text{SiO}_2$  with water, sets and hardens rapidly, giving a product of strength comparable with that of good Portland cement. This compound is the essential constituent of a good cement, which contains 30–35% of it. On hydration, free lime is extracted, leaving gelatinous hydrated silica, just as with the dicalcium silicate, but the decomposition is more rapid and can be carried to completion if washing is continued. From these observations it seems that the setting and hardening of cement involve the formation of gelatinous hydrated material which subsequently partly crystallises. The initial set is probably caused by hydration of the tricalcium aluminate. This and hydration of the tricalcium silicate are responsible for the hardness and cohesive strength at first. The gradual increase of strength is due to the continued hydration of these and of the dicalcium silicate. Gelatinous silica is probably the chief cementing material, just as it appears to have formed the original binder of sand grains in tough sandstones, which are ideal concretes.—H. J. H.

*Portland cement; Constitution and hydration of* — A. A. Klein. Faraday Soc., Jan. 14, 1918. [Advance proof.] 9 pages.

PROGRESS towards a solution of the problem of the constitution of cements dates from the application of the microscope and optical methods by Le Chatelier and Tornebohm. Three main theories of constitution have held the field, viz., that cement is (1) a mutual solid solution of various constituents of disputed composition, (2) a definite compound of lime, silica, and alumina, and (3) a mixture of separate silicates and aluminates, some or all of which are hydraulic. The probability of the third view has been established by the work of the United States Geophysical Laboratory (see this J., 1915, 139; 1916, 842) and Bureau of Standards (this J., 1916, 1016). The former studied the equilibrium relations of the ternary system,  $\text{CaO}$ ,  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ , and the latter applied the results to typical cement clinkers. Within the composition limits of good practice Portland cements are essentially a mixture of  $3\text{CaO}.\text{SiO}_2$ ,  $3\text{CaO}.\text{SiO}_2$ , and  $3\text{CaO}.\text{Al}_2\text{O}_3$ . The hydration of the individual constituents was studied so that conclusions could be drawn as to the mechanism of the setting of cement. The aluminate hydrates rapidly and probably causes the initial set. Compounds of aluminate and calcium sulphate may be formed, and free lime, if present, is hydrated. Next the tricalcium silicate is hydrated and this process continues for about one month, producing a mass of dense structure. The dicalcium silicate hydrates to a granular porous mass. Neither should predominate in good cement. It is possible by grinding together the previously burned constituents in suitable proportions to make a mixture having the properties of Portland cement.—H. J. H.

*Cements; Crystalloids against colloids in the theory of* — H. Le Chatelier. Faraday Soc., Jan. 14, 1918. [Advance proof.] 4 pages.

THE author holds that explanations of hardening which state that the cement is colloidal are meaningless. Colloids are only insoluble bodies in a state of extremely fine division, which imparts to them a relatively high surface energy, and this gives rise to a number of special properties associated with the colloidal state. Soluble bodies cannot persist in the colloidal condition in presence of a liquid capable of partially dissolving them, owing to progressive recrystallisation and growth of crystals. In the setting of hydraulic cements it is possible that the particles, in the initial stages of the set, are in the colloidal condition just as are many precipitates, e.g., barium sulphate, but there is no proof that this persists, even though the particles in the hardened cement are not recognisable as true crystals. Probably they are too small to be detected. The conditions of setting are such that the growth of the particles is limited, but it is possible to choose conditions such that the cements on hydration form visible crystals. The author does not see any reason to discard his original theory (this J., 1888, 847) that the anhydrous cement dissolves, to form an aqueous solution supersaturated with respect to the hydrated compounds. These crystallise out in a confused mass, thus giving to the product its mechanical strength.—H. J. H.

*Cement; Setting of* — J. G. A. Rhodin. Faraday Soc., Jan. 14, 1918. [Advance proof.] 2 pages.

IN experiments to render soluble the potash in felspar the latter was heated with lime and salt. The residue after leaching with water had setting properties, especially after heating it with more lime. Some cements of this and other origins can lose their setting properties completely on standing

in a stoppered bottle for 24 hours. They can be prevented from acquiring this inert form by the addition of 0.5% of aluminium sulphate. Cements also become inert after fusion. An active cement is in a state of "labile molecular equilibrium."

—H. J. H.

*Portland cement: Effect of addition of suitable slag on setting properties of —.* E. Deny and E. H. Lewis. *Faraday Soc.*, Jan. 14, 1918. [Advance proof.] 4 pages.

CEMENT clinker made from blast-furnace slag (hematite pig) and limestone, owing to the high alumina to silica ratio, usually gives a quick-setting cement. This can be corrected by the addition of a properly-ground slag, and at the same time, with up to 30% of slag, the tensile strength is increased. Experiments on the addition of slag ( $\text{SiO}_2$  30.5%,  $\text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  16.6%,  $\text{CaO}$  49.4%,  $\text{MgO}$  2.3%) to ordinary brands of Portland cement, gave similar results. Mixtures of 70% of cement and 30% of slag always showed increased tensile strength as compared with the neat cement at 7 days and 28 days, whilst the setting times—initial and final—and the expansion were generally diminished. By the addition of slag to a cement clinker made from blast-furnace slag, a cement has been produced with a strength of 1300 lb. at 28 days.—H. J. H.

*Cement: Setting of — in relation to engineering structures.* B. Blount. *Faraday Soc.*, Jan. 14, 1918. [Advance proof.] 3 pages.

THE absence of precise knowledge of the mechanism of setting of cements—plaster of Paris excepted—is emphasised. The setting of the commoner and more important Portland cements is a slow and gradual process and it is not possible practically to treat the time of setting in a rigid quantitative manner. Furthermore, tests are usually made on cements neat, whereas in practice the cement is generally used in concrete and the setting times in the two conditions are not quantitatively related. Further investigation is advocated.—H. J. H.

*Mortar: Ancient and modern —.* W. J. Dibdin. *Faraday Soc.*, Jan. 14, 1918. [Advance proof.] 5 pages.

IN work carried out for the Institute of British Architects, the author was able to compare the mortars used by builders in ancient and modern times. The former were not superior to properly compounded mixings of the present day. In modern practice 1 volume of lime is usually taken to 3 of aggregate, but 1 to 1 was the average proportion found in the old mortars examined. Whereas now clean washed sand is specified, in ancient mortars the aggregate was found to contain up to 19.5% of clay, the builders having used any convenient materials without any special care in selection. Experimental mixings showed that the ratio of 1 of lime to 2 of aggregate usually gives the strongest mortar and sometimes it is advantageous to incorporate a little clay. No evidence of the formation of soluble silicates to any appreciable extent could be found. The author believes that the mortar sets owing to a process of aggregation, without chemical combination; crystallisation also assists in the setting. Where the ratio 1 : 3 is adopted there is insufficient lime to fill the pores and mechanical weakness results.—H. J. H.

*Recovery of polish from greensand.* Charlton. See VII.

#### PATENTS.

*Plaster composition: Process of making a —.* H. M. Olson. Lompoc, Cal., Assignor to Celite Products Co., Los Angeles, Cal. U.S. Pat. 1,249,997, Dec. 11, 1917. Date of appl. Mar. 3, 1917.

FINELY divided amorphous silica, e.g., kieselguhr,

is ground with finely divided lime, and sufficient water is added to hydrate the lime and give a product in the form of a dry powder.—A. B. S.

*Process for increasing the solubility of polish in cement-kiln dust.* U.S. Pat. 1,249,708. See VII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Wrought iron: Some unusual features in the microstructure of —.* H. S. Rawdon. *Technol. Paper No. 37*, Bureau of Standards, U.S. Dept. of Commerce. Engineering. 1918. 105, 77—79.

A WROUGHT iron eyebar which had been in use for 30 years as a tension member of a railway bridge revealed unusual features upon microscopical examination. In addition to the usual structure of wrought iron, the ferrite crystals had a peculiar mottled appearance which was restricted to certain streaks throughout the metal, and particularly associated with unusually large crystals. Using Stead's method of etching with an alcoholic solution of cupric chloride acidulated with hydrochloric acid, a structure consisting of broad parallel bands was seen across the elongated crystals, bearing some resemblance to twinned crystals, and also, more often, presenting an indefinite mottled appearance of light and dark areas; such patterns are not found on etching ordinary wrought iron. Fracture occurred through the crystals and parallel to the markings constituting the mottled pattern at that point, leading to the conclusion that the non-homogeneity of the individual crystals, as indicated by the mottled etch pattern, is due to some impurity—in this case phosphorus—dissolved in the iron but not uniformly diffused. The ill effects of phosphorus may be much increased in this way. Examination of a series of wrought irons showed that such features are not common, or characteristic of all low-grade irons, which may be unsuitable for other reasons. A second type of markings—the well-known Neumann lines—are shown to bear no apparent relation to the other variations in structure described.—T. H. B.

*Carbon steels: Anomaly of elasticity of — and the reversible transformation of cementite.* P. Chevenard. *Comptes rend.*, 1918, 166, 73—76.

ON the analogy of the nickel-steels there should be an anomaly in the elasticity of the carbon steels corresponding to the magnetic transformation and to the anomaly in the dilatation of the cementite (see this J., 1917, 882). This was investigated by measuring the variation with temperature of the torsional rigidity of three steels containing up to 1.5% of carbon. A torsion pendulum was employed made of wire from the steel under test and at different temperatures maintained by an electrically heated furnace. At each temperature it was necessary to measure the time of oscillation of the pendulum and, by determining the elongation, the decrement of the torsion modulus. The wires had all been subjected to the same heat and mechanical treatment. It was found that the variation with temperature of the inverse squares of the times of oscillation only represents the apparent variation of the torsion modulus up to 300°C. Above 300°C. the decrements showed a rapid increase. The curves of the torsional rigidity showed a slight discontinuity at 210°C. indicating the Curie (magnetic transformation) point of cementite. The anomaly in the elasticity of carbon steels is directly proportional to the carbon content.—H. J. H.



*Manganese in steel; Determination of — in the presence of chromium and vanadium by electro-metric titration.* G. L. Kelley, M. G. Spencer, C. B. Hingworth, and T. Gray. *J. Ind. Eng. Chem.*, 1918, 10, 19—23.

MERCUROS nitrate was the only reducing agent found which would reduce permanganate quantitatively and rapidly at ordinary temperatures without reducing chromates and vanadates. A standard solution is made by dissolving 10.5 grms. of mercurous nitrate in 150 c.c. of water and 2 c.c. of nitric acid. The clear liquor is decanted off and made up to 1 litre. This solution is compared electrometrically with a solution of potassium permanganate containing 0.5 grm. of manganese per litre and standardised against sodium oxalate. The titration is made in a solution of 50 c.c. of sulphuric acid (sp. gr. 1.58) in 200 c.c. of water. The apparatus has been described previously (see this J., 1917, 1011). For the determination of manganese in steel, either sodium bismuthate or ammonium persulphate may be used as the oxidising agent. Using sodium bismuthate, the oxidation is carried out as described by Blair ("The Chemical Analysis of Iron," 7th Edn., p. 122). After filtering, a small piece of ice is added and 50 c.c. of sulphuric acid (sp. gr. 1.58). The volume should be 250 c.c. and the titration should not be performed above 40° C. Using ammonium persulphate, 0.5 grm. of the steel is dissolved in 65 c.c. of sulphuric acid (sp. gr. 1.58) and when solution is complete, nitric acid is added, drop by drop, to oxidise. After boiling a minute or two, the solution is diluted with hot water to a volume of 200 c.c., heated to boiling, and 10 c.c. of silver nitrate solution (2.5 grms. per litre) and 20 c.c. of ammonium persulphate solution (100 grms. per litre) added. Boiling is continued for about one minute, the solution cooled and titrated at about 20° C. after the addition of a little more sulphuric acid. Using this method, filtration is avoided and it is not necessary to make the titration at once as when using sodium bismuthate. Neither method requires special skill for its application. The authors discuss the reaction between permanganic acid and mercurous nitrate.—L. A. C.

*Steel; Electrolytic pickling of —.* M. De K. Thompson and F. W. Dodson. *Met. and Chem. Eng.*, 1917, 17, 713—714. (See also this J., 1917, 1097.)

COMPARATIVE experiments were made on the chemical and electrolytic pickling of black sheet iron and transformer sheet iron containing silicon. Electrolytic pickling gave a silvery white, clean surface much better than the spotted surface of the chemically pickled sheets. The amount of iron dissolved from the black sheet was 0.0032 grm. and 0.00386 grm. respectively per sq. cm. of surface. Another advantage of electrolytic pickling is the low temperature of working. The acid consumption is in proportion to the iron removed and the difference is not sufficient to be decisive. The presence of ferrous sulphate in the cell had no depolarising effect.—H. J. H.

*Bismuth [in copper]; Colorimetric determination of —.* H. A. B. Motherwell. *Eng. and Min. J.*, 1917, 104, 1091—1092.

The following method is suitable for the determination of small quantities of bismuth in copper. A suitable quantity of the copper is dissolved in nitric acid, the solution diluted and cooled, sodium carbonate is added to precipitate a small quantity of copper, and, after 6 hrs., this precipitate (which will also contain all the bismuth present) is collected on a filter. The precipitate is dissolved in hydrochloric acid, the copper and bismuth are precipitated as sulphides, which are collected,

washed until free from iron, and then dissolved in nitric acid. Five c.c. of lead nitrate solution (13.5 grms. per litre) is added to the solution, the mixture is nearly neutralised with ammonia, and an excess of ammonium carbonate is added. After boiling and cooling, the precipitate is collected, dissolved in nitric acid, and the precipitation repeated. If the colour due to copper is strong, dilute cyanide solution is added until the coloration has nearly disappeared and the precipitate then washed free from copper. The precipitate is dissolved in a very small quantity of nitric acid, the solution evaporated to a syrup, cooled, dissolved in 3 drops of nitric acid and 5 c.c. of water, the solution diluted to 25 c.c. and made up to 50 c.c. by the addition of 1.7% potassium iodide solution. The coloration obtained is compared at once with that given by a known quantity of bismuth in the presence of lead under similar conditions. The same quantity of lead nitrate solution must be present in the test and comparison solutions.—W. P. S.

*Manganese ores in Sweden; Utilisation of —.* J. Harden. *Met. and Chem. Eng.*, 1917, 17, 701—704.

THE Swedish consumption of manganese compounds is equivalent to about 6000 tons of manganese per annum, and this could be covered without importation if the internal resources were developed. Ferromanganese has been usually produced in blast furnaces by smelting manganese and iron ores with charcoal. The loss of manganese in the flue gas is great and a high carbon content of the alloy is almost inevitable. These can be obviated by the use of electric smelting where power costs permit. Three-phase types of furnace with the neutral point under the hearth seem preferable and the iron should be added as scrap, not as ore. The power consumption is 8000—8500 kilowatt hours per ton of ferromanganese (80% Mn, 12% Fe, 6% C). The addition of solid ferromanganese to the Bessemer converter or open-hearth furnace is to be deprecated on account of loss by volatilisation and in the slag. When solid ferromanganese is added to the metal in the ladle there is insufficient time for complete admixture and irregularity in the ingot may occur. To avoid these difficulties the alloy should be added after fusion and for this purpose an electrical melting furnace is well suited. The heat balance is given for a 3-ton arc furnace used for this purpose, showing a thermal efficiency of 40.4% and a cost of 21s. 10d. per ton of ferromanganese melted. Induction furnaces are still more economical of power. The manganese losses in melting are negligible. Comparative tests of the manganese losses in the addition of solid and liquid alloy to the steel show a saving of 44% in favour of the use of the molten alloy. Moreover, greater uniformity of the ingots produced is secured and the time of operation is reduced.—H. J. H.

*Manganese deposits; Utilisation of low-grade —.* J. E. Johnson, jun. *Engineers' Soc. W. Pa.*, Nov., 1917. *Eng. and Min. J.*, 1917, 104, 1027—1030.

FOR several years practically no manganese was produced in the U.S.A., the deposits, though considerable, being mostly low-grade. Spiegeleisen was mostly used in Bessemer converting, but the development of the open-hearth process brought about the use of ferromanganese, which is poor in carbon and hence produces low-carbon steel. The production of ferromanganese requires high-grade ores low in iron, phosphorus, and silica. Low-grade ores are as a rule refractory to concentration because the oxides are finely disseminated through the gangue. In the blast furnace, the silica combines much more readily with manganese than

with ferrous oxide; at higher temperatures the reduction is more complete but manganese is lost by volatilisation. A smelting loss of 15% is a minimum. The electric furnace is being introduced in the U.S.A. for the manufacture of ferromanganese. Less fuel is used, hence less silica is introduced, than in the blast-furnace; moreover, the silica is largely reduced to silicon, forming ferrosilicon-manganese, which takes up much less carbon than ferromanganese. Ferrosilicon cannot be used as a substitute, as it has a much weaker deoxidising power; calcium-silicon and calcium-aluminium-silicon are said to have been used in Europe. By altering existing blast-furnace practice so as to produce pig iron containing 1—2% of

destructive distillation of Canadian resinous wood waste, if twice the normal proportion (i.e., 20%) was used. Addition of 4 lb. of caustic soda per ton of slime gave a great improvement in extraction and grade of concentrate. Coal tar creosote was found to be indispensable as a collecting agent, but it could be replaced in part by hardwood oil. Attempts at utilising the whole crude oil distillates from the destructive distillation of Canadian resinous woods were unsuccessful. The successful hardwood oils were tried on a commercial scale, and extractions 20% higher than those found in the laboratory tests were obtained. The results tabulated below prove that the oils named can be used in practice instead of pine oil.

Frothing agent.	Collecting agent.		Oil mixture, lb. per ton ore.	Tons of ore treated.	Average extraction.
	Coal tar creosote.	Coal tar.			
Pine oil G.N.S. No. 5 .....	% 20	% 60	% 20		% 86.8
ditto .....	20	60	20	1994	88.3
Heavy hardwood creosote .....	40	50	10	2019	87.4
Hardwood acid creosote oil ....	40	50	10	807	89.1
Ketone residue .....	25	65	10	675	88.5
			106	739	

—W. R. S.

manganese from a mixture of iron and low-grade manganese ores, the present shortage of manganese could be partly overcome. This can also be attained by charging low-grade manganese ore into the open-hearth furnace, a substantial reduction to metal being caused by the metalloids in the bath.—W. R. S.

*Molybdenum in Norway.* E. R. Woakes. Inst. Min. and Met., Jan., 1918. [Advance copy.] 12 pages.

MOLYBDENUM is chiefly mined in the part of Norway lying south of a parallel passing through Christiania. The deposits are of three types: quartz lodes, fissures in granite, and impregnated granite. There are only a few mines having a regular output, and the total export in 1916 is said to have been 140 tons of concentrate. The ores are crushed in Blake breakers and ball mills, and concentrated by the Elmore vacuum process. Mica and copper pyrites constitute objectionable impurities. If the post-war price does not fall below 80s. per unit of  $\text{MoS}_2$ , the Norwegian output of molybdenum may be maintained at about 100 tons per annum. It is probable, also, that ferro-molybdenum will be made in Norway in the near future.—W. R. S.

*Flotation; Canadian wood oils for ore* —. R. E. Gilmore and C. S. Parsons. Canadian Chem. J., 1917, 1, 151—157, 180—187.

THE investigation was undertaken on account of the difficulty of securing an adequate supply of imported pine oil (longleaf southern pine) for the flotation plants at Cobalt, Ont., where it is used as a frothing agent, with coal tar creosote and coal tar as collecting agents. The standard oil mixture giving successful commercial results at Cobalt consists of pine oil 10, coal tar creosote 80, and coal tar 10%. Other oils were substituted for pine oil in the above mixture, and if 10% was unsatisfactory the percentage was raised. Similarly, substitutes were introduced for the coal tar creosote and coal tar. Successful frothing or collecting substitutes were then mixed and tested, a Janney testing machine being used. The results proved that the following were suitable frothing agents for Cobalt silver ore: Crude hardwood creosote oils; crude ketone residue (from the refining of grey acetate of lime); pine oil from the

*Flotation [of ores]; Effect of addition agents in—*. H. M. Thornberry and H. T. Mann. Met. and Chem. Eng., 1917, 17, 709—713.

THE influence of a number of common inorganic salts on the concentration of a Missouri lead ore was studied in a small laboratory flotation machine. The ore contained 4.64% of lead. Three oils were used—a hardwood creosote, "Naval Stores flotation oil 17," and cresylic acid. The action of sulphuric acid and alkaline-earth sulphates was to lower the extraction about 10% but without much effect on the grade of concentrate. The acid and normal alkali sulphates and the alums had little or no effect on the flotation. The effect of the sulphates of the heavy metals was usually small but was to lower the extraction. Cadmium sulphate exercised a powerful and injurious effect on both the extraction and grade of concentrate.—H. J. H.

*Commercial aspect of recovery of potash in pig iron manufacture.* Chance. See VII.

*Effect of addition of suitable slay on setting properties of Portland cement.* Deny and Lewis. See IX.

## PATENTS.

*Steel; Manufacture of* —. Sir. W. G. Armstrong, Whitworth and Co., Ltd., and W. C. Rowden, Newcastle-upon-Tyne. Eng. Pat. 111,916, Dec. 20, 1916. (Appl. No. 18,248 of 1916.)

STEEL scrap is melted together with carbonaceous matter in a basic open-hearth furnace. The steel is then poured into an open-hearth acid or basic furnace, pig iron, scrap steel, or carbonaceous matter being added.—W. R. S.

*Blast furnaces or steel furnaces; Processes or means for removing solid "metal bottoms" of* —. W. Riddle, Middlesbrough. Eng. Pat. 112,199, Mar. 5, 1917. (Appl. No. 3251 of 1917.)

IN removing the mass of metal known as a "metal bottom" which accumulates below the hearth of a blast furnace, the holes for the reception of blasting cartridges are formed by burning with an oxygen jet instead of by drilling. To obtain the initial "bite" on the metal, a pocket of clay or an open-ended metal tube containing small coal or other combustible material is applied to the



surface and ignited by a lamp with the aid of the oxygen blowpipe. The burning is then continued by the blowpipe.—W. F. F.

*Steel; Producing — in side-blown converters.* J. H. Hall, New York, Assignor to Taylor Wharton Iron and Steel Co., High Bridge, N.J. U.S. Pat. 1,249,075, Dec. 4, 1917. Date of appl. Mar. 17, 1914.

THE charge subjected to lateral air-blowing contains silicon and manganese in such proportions that a fluid slag is produced which contains approximately 60 parts MnO to 40 parts SiO<sub>2</sub>.  
—W. R. S.

*Coating iron with brass; Process for —.* T. A. Iyer, Seshadripuram, India. Eng. Pat. 112,045, Dec. 21, 1916. (Appl. No. 18,329 of 1916.)

BRASS is melted in a crucible with 2.5% of potassium ferrocyanide, the two are mixed together, and 2.5% of barium chloride is added. The iron article is polished, dipped into the molten brass, removed, and cooled. In a modification the potassium ferrocyanide and barium chloride are replaced by potassium ferricyanide and sal-ammoniac (ammonium chloride) in the same proportion.—W. F. F.

*Galvanising iron and steel plates, sheets, and the like; Mode of "hot process" —.* The White-inch Galvanizing Co., Ltd., and R. E. Ordidge, Partick. Eng. Pat. 112,220, Apr. 20, 1917. (Appl. No. 5550 of 1917.)

TO galvanise iron or steel plates on one side only by the dipping process, the side which is not to be galvanised is coated with a paint made by mixing lithopone 12 parts, Paris white 4 parts, and pulp size 1 part, with water, and boiling to the consistency of milk. This coating prevents the adherence of the molten zinc.—W. F. F.

*Iron and steel; Process of rust-proofing — by vapours containing phosphorus compounds.* W. H. Allen, Detroit, Mich. U.S. Pat. 1,248,053, Nov. 27, 1917. Date of appl. Feb. 1, 1917.

THE metal is placed in a closed chamber into which phosphorus pentoxide vapour and steam are passed simultaneously.—W. E. F. P.

*Puddling furnaces.* J. J. H. Mackinlay, Shifnal, Salop. Eng. Pat. 112,179. (Appl. Nos. 1249, Jan. 25, and 10,665, July 25, 1917.)

IN a puddling or reverberatory furnace, a pre-heating chamber is provided on the opposite side of the hearth to that of the grate, and the iron is introduced into it through an air-locked chamber having an outer door and a door communicating with the preheating chamber. The air-locked chamber is provided with a travelling floor which may be moved into the preheating chamber with its charge; the inner door is then closed and the floor withdrawn, leaving the charge in the preheating chamber. The charge is preheated by the hot gases passing to the flue and is then pushed on to the hearth by a ram moving longitudinally through the chamber. The walls of the preliminary heating chamber and the head of the ram may be cooled by internal water pipes. The combustion gases may be used finally for heating a steam boiler or for preheating the air or gas used for heating the furnace.—W. F. F.

*Heating-furnace; Metallurgical —.* E. C. Walker and A. S. Moses, Assignors to G. M. Ilges, St. Louis, Mo. U.S. Pat. 1,248,213, Nov. 27, 1917. Date of appl. Jan. 3, 1916.

IN a coal-fired furnace adapted to deliver dry heating gases under pressure to form a dry reducing atmosphere, the closed fuel chamber has a hopper casing at the top, from which fuel is supplied, and

an ash-pit at the bottom. Means are provided for maintaining air under pressure in the hopper in such manner as to form an air seal and also in the ash-pit; and for controlling the character of the dry reducing atmosphere by varying the amounts of air supplied to the hopper and the ash-pit.—W. E. F. P.

*Furnace for treating ores.* G. L. Tanzer, Seattle, Wash. U.S. Pat. 1,249,854, Dec. 11, 1917. Date of appl. July 24, 1917.

AN airtight metal cylinder, lined with refractory material, rotates on a horizontal axis. A perforated partition of refractory material is provided close to one end, forming a narrow gas compartment, and a grating is arranged parallel to the partition and nearer the centre of the chamber, forming a compartment for charcoal. Ore to be treated is placed in the remaining larger compartment and a fan is provided in a co-axial chamber within the lining of the opposite end of the casing, communicating by passages with the ore compartment. Gas is circulated by the fan through the gas compartment, charcoal compartment, and ore compartment to the fan chamber, and thence through a passage in the lining back to the gas compartment.—W. F. F.

*Metal pipes, rods and the like; Apparatus for covering — with lead. Apparatus for covering the internal surface of metal pipes and the like with lead.* J. A. Reavell, Beckenham, Kent, and W. J. May, London. Eng. Pats. (A) 112,321 and (B) 112,322, Jan. 9, 1917. (Appl. Nos. 393 and 394 of 1917.)

(A) THE external surface of a pipe or rod is "tinned" with an alloy of tin and lead, and the pipe or rod is mounted with its axis vertical in a concentric casing, and supported by guides and by a sliding collar at the lower end, so that it may be moved vertically through the casing. An annular polished die is arranged in the casing, surrounding the pipe or rod, and slightly spaced from it, so that when molten lead is poured into the upper part of the casing, it solidifies in the annular space between the die and the pipe. The lower part of the casing is surrounded by a water jacket so that the coating of lead solidifies as the pipe is drawn downwards, and the upper part of the casing is heated, preferably by gas rings, to keep the lead molten. (B) The apparatus is similar to that described in (A) except that the pipe is coated internally and itself forms the container for the lead. A polished internal cylindrical die is used, slightly smaller than the pipe, and the pipe is heated by an external gas-ring above the die and cooled by a water jacket at the lower end of the die.—W. F. F.

*Zinciferous ores; Treatment of —.* J. H. and P. M. Gillies, East Camberwell, Victoria, Australia. Eng. Pat. 112,336, Feb. 1, 1917. (Appl. No. 1633 of 1917.)

COMPLEX zinc ore containing also lead, copper, arsenic, antimony, gold, and silver, is coarsely crushed, and partly desulphurised by roasting, then mixed with carbonaceous material, preferably coke, and about 20% of water, and fed to a fuming furnace having a perforated grate through which a low pressure air blast is supplied. Zinc and lead are volatilised and drawn off mainly as zinc oxide and lead sulphate, and condensed together with arsenic and antimony impurities. The clinker remaining in the furnace, containing the non-volatile metals, is smelted in an electric furnace with the necessary flux to form a matte and any residue of zinc and lead which is driven off is also condensed. The condensed compounds are treated with sulphuric acid to dissolve the zinc oxide and arsenic and antimony compounds, and the lead

sulphate is then allowed to settle out. The solution is heated to about 80° C., and treated with hydrogen sulphide to precipitate the impurities as sulphides, which are then filtered off. If cadmium is present it is removed prior to the treatment with hydrogen sulphide by passing the solution over granulated zinc. The purified zinc sulphate solution is electrolysed with zinc cathodes and lead anodes, until the amount of liberated sulphuric acid reaches 10—12%, and the liquor may be used for treatment of a further charge of condensed fume. To secure an even deposition of zinc, about 0.5—1.0 gm. of gum arabic may be added per litre of solution.—W. F. F.

*Zinc-extracting furnace with vertical retorts.* R. von Zelewski, Engis, Belgium. U.S. Pat. 1,250,071, Dec. 11, 1917. Date of appl., Feb. 24, 1917.

A CASING is provided with a vertical cylindrical chamber for the retort, and contains gas and air ducts communicating with the chamber. The chamber is open at the bottom, and the retort is supported on a platform, movable vertically, which is counterbalanced by weights attached to cables running over pulleys, and which closes the bottom of the chamber.—W. F. F.

*Spelter; Process for producing* —. C. H. Fulton, St. Louis, Mo., Assignor to Metallurgical Laboratories, Inc., Chicago, Ill. U.S. Pat. 1,249,961, Dec. 4, 1917. Date of appl., Jan. 22, 1917.

FINELY divided spelter or zinc dross is mixed with coke and tar or pitch and compressed into briquettes that will retain their original form and volume when submitted to zinc distillation temperatures.—W. R. S.

*Amalgamating and other apparatus; Agitators for use in* —. S. L. Berry, San Francisco, Cal., U.S.A. Eng. Pat. 112,405, Sept. 28, 1917. (Appl. No. 14,059 of 1917.)

By an amalgamating apparatus, pulp is fed into a tank containing mercury at the bottom, and a turbine pump casing is mounted concentrically on a vertical, positively driven shaft, and submerged in the liquid in the tank. The pump casing contains an impeller rotated independently, and has a flared suction inlet at the top for the pulp. The pulp is discharged through slots in radial pipes projecting outwards from the pump casing close to the bottom of the tank. The slots discharge into the mercury and are inclined slightly upward in the direction of revolution of the radial pipes. The direction of rotation of the impeller may be reversed when desired by means of a suitable bevel wheel reversing gear, to clear the slots when clogged. The pump casing is preferably driven through a belt passing over cone pulleys, so that the relative speed of casing and impeller may be varied.—W. F. F.

*Case-hardening material; Process of making* —. A. O. Blach, Assignor to A. O. Blach Co., Chicago, Ill. U.S. Pat. 1,248,604, Dec. 4, 1917. Date of appl., Sept. 18, 1917.

THE process consists in shrinking and drying granular carbonaceous material at a temperature above 1000° F. (540° C.), then impregnating the granules with a "solution of an "energiser" infusible at the temperature employed, and mixing the impregnated granules with a pulverulent "energiser."—W. R. S.

*Alloy; Metal* — [for electrical contacts]. H. S. Cooper, Assignor to The Electro Metals Products Co., Cleveland, Ohio. U.S. Pat. 1,248,621, Dec. 4, 1917. Date of appl., Jan. 21, 1916. Renewed Oct. 8, 1917.

A HARD, ductile, malleable alloy for electrical contact devices consists of gold, silver, and approximately 4% of osmium.—W. R. S.

*[Zirconium] alloy and process of producing same* — J. B. Gronagle, Catonsville, Md. U.S. Pat. 1,248,648, Dec. 4, 1917. Date of appl., Dec. 7, 1915.

AN iron-zirconium alloy containing 60—90% of the latter is prepared from materials containing iron and zirconium by the aluminothermic method.—W. R. S.

*White metal alloy.* H. K. Sandell, Assignor to H. S. Mills, Chicago, Ill. U.S. Pat. 1,248,925, Dec. 4, 1917. Date of appl., June 18, 1917.

THE alloy is composed of mercury 10, bismuth 10, tin 10, zinc 20, and copper 50 parts by weight. A primary alloy is first made by melting the bismuth and tin together, cooling the mixture to just above its melting point, and adding the mercury. This is then added to the molten zinc, and the product to the molten copper, each of the last two metals being maintained at its melting temperature.—W. E. F. P.

*Metallic [arsenical] ores; Apparatus for treating* —. W. R. Smith, Buffalo, N.Y. U.S. Pat. 1,249,226, Dec. 4, 1917. Date of appl., Feb. 18, 1914.

THE apparatus consists of a blast furnace connected at the upper part with a long horizontal pipe into which the gases are drawn by means of a fan; and at the lower part with an enclosed bed upon which the molten metal is discharged. Means are provided for introducing water into the horizontal pipe and collecting the particles precipitated therein, and for supplying water to the bed enclosure for the purpose of condensing the arsenical fumes evolved from the molten metal.—W. E. F. P.

*Flotation separation [of ores]; Apparatus for* —. C. F. Spaulding, Salt Lake City, Utah. U.S. Pat. 1,249,684, Dec. 11, 1917. Date of appl., Feb. 11, 1916.

THE pulp container is provided with a vertical air-lift column to which compressed air and oil are fed at the lower end. A number of rotating vertical shafts are arranged in the column, rotating in the same direction, and each carrying a number of beater arms.—W. F. F.

*Electrodeposition of metals; Apparatus for* —. A. Leuchter, Brooklyn, N.Y. U.S. Pat. 1,249,787, Dec. 11, 1917. Date of appl., Oct. 5, 1916.

THE cathodes and anodes are suspended from suitable supports, provided with current connections, in a vertical position spaced from the inner walls of the electrolytic tank, and submerged in the electrolyte. A line of non-perforated pipe, arranged along the lower inner edges of the sides and ends of the tank, is connected to pipes supplying air, and to perforated pipes which are disposed closer to the vertical plane of the front face of the cathodes than to the adjacent faces of the anodes. Adequate quantities of air are thus discharged upwards through the spaces between the electrodes, and the electrolyte is kept in constant upward circulation, so that the impoverished electrolyte is moved from the cathode surfaces and caused to pass over to the back of the cathodes.—B. N.

*Blast furnaces.* W. J. Foster, Walsall, Eng. Pat. 112,513, Jan. 17, 1917. (Appl. No. 843 of 1917.)

*Blast furnaces.* G. H. Fisher, Stoke-on-Trent, and R. Fisher, Pontypool, Eng. Pat. 112,589, July 23, 1917. (Appl. No. 10,532 of 1917.)

*Baths or tanks for treatment of shell cases and metals by acid; System of* — J. E. Thornton, Cardiff, Eng. Pat. 112,531, Jan. 25, 1917. (Appl. No. 1257 of 1917.)



*Welding or fusion deposition of metals; Electric* ——. The Quasi-Arc Co., Ltd., and W. L. Cole, London. Eng. Pat. 112,353, Mar. 9, 1917. (Appl. No. 3486 of 1917.)

*Tin; Apparatus for recovering* — from waste material. E. A. Considère and C. H. Bethell, Coventry. Eng. Pat. 112,471, Mar. 26, 1917. (Appl. No. 13,996 of 1916.)

*Tin or other metal coated plates or sheets; Apparatus for the manufacture of* ——. W. Gilbertson and Co., Ltd., E. Griffiths, and G. Webb, Pontardawe, Glamorgan. Eng. Pat. 112,555, Apr. 2, 1917. (Appl. No. 4748 of 1917.)

*Alloys; Metal* ——. C. L. Jones, Oakland, N.J., U.S.A. Eng. Pat. 112,377, May 29, 1917. (Appl. No. 7644 of 1917.)

SEE U.S. Pat. 1,244,742 of 1917; this J., 1918, 13 A.

*Method and apparatus for separating gases and formation of products thereby. [Drying air for blast-furnaces.]* Eng. Pat. 112,153. See I.

*Manufacture of composition fuel.* Eng. Pat. 112,341. See 11A.

*Production of potassium chloride [in manufacture of pig iron].* Eng. Pat. 112,338. See VII.

*Electrolytic anodes.* Eng. Pat. 108,311. See XI.

*Commutator-section of cast alloy.* U.S. Pat. 1,268,924. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Electric furnace smelting of phosphate rock, and use of the Cottrell precipitator in collecting the volatilised phosphoric acid.* Carothers. See VII.

*Electrolytic pickling of steel.* Thompson and Dodson. See X.

### PATENTS.

*Accumulators; Electric* ——. I. Chkhar, Paris. Eng. Pat. 102,976, Dec. 29, 1916. (Appl. No. 18,611 of 1916.) Under Int. Conv., Dec. 29, 1915.

ELECTRODES for accumulators are made of a continuous band of metal formed from two sheets of rolled, drawn, or cast metal, with or without the interposition of a sheet of metal unattacked by acids, and thus forming a double surface electrode with a positive and negative surface. It is bent along its whole length so as to form on each of its surfaces alternate spaces or compartments, and the positive acting substance is placed in the compartments on one of the surfaces of the plate, and the negative substance in the alternating compartments on the opposite surface, the electrode being afterwards fixed in a suitable frame.—B. N.

*Furnaces; Electric* ——. G. B. Filmer and J. J. Denton, London. Eng. Pat. 112,177, Jan. 23, 1917. (Appl. No. 1146 of 1917.)

THE furnace is provided with a resistor and heater consisting of a normally non-conducting material, such as magnesia, sand, clay, or the like, which is ionised by the addition of a fusion mixture, such as sodium hydroxide and water, and removable electrodes are arranged within it. A muffle, tube, or plate, of fused zirconia or other suitable material, is partially or entirely enclosed in, or placed upon, the resistor material, and is utilised as a separate and independent heater when rendered conductive by the first heater.—B. N.

*Furnace; Electric* — for producing endothermic gas reactions. R. Pfähler, Charlotte, N.C., and I. Hechenbleikner, Great Falls, S.C., Assignors to Southern Electro-Chemical Co., Charlotte, N.C. U.S. Pat. 1,249,479, Dec. 11, 1917. Date of appl., Aug. 26, 1913.

THE opposite walls of the reaction chamber diverge from the base of the furnace upwards, each wall being provided with an opening, and a door mounted on a carriage and trolley. A pair of opposed electrodes are arranged in the chamber, one opposite each door, each electrode having a base member extending through and insulated from the furnace wall, and a removable diverging member attached to it, the diverging part conforming substantially to the contour of one of the diverging walls of the furnace chamber, and adapted to be withdrawn through the opening in the wall. Means are provided for circulating a cooling medium through the two members of each electrode. Two "kindling-blades" are associated with the electrodes, and separate nozzles are provided for delivering air into the furnace chamber, one of the nozzles being arranged to deliver a current of cooling air against the "kindling-blades," whilst a current of cooling air is also delivered through separate outlets directed laterally against the insulated portion of each electrode.

—B. N.

*Anodes; Electrolytic* ——. Electrolytic Zinc Co., Inc., New York, Assignees of H. M. Stuart, Baltimore, U.S.A. Eng. Pat. 108,311, July 12, 1917. (Appl. No. 10,072 of 1917.) Under Int. Conv., July 26, 1916.

THE anode, e.g., of zinc-bearing metal, is surrounded by an envelope of material pervious to the electrolyte, but through which the slimes, etc., resulting from the disintegration of the anode will not pass, and certain portions of the anode surface are protected from electrolytic action by non-conducting shields, such as a pair of non-conducting strips, one on each side of the envelope. The shields thus divide the anode surface into distinct areas, and bear against the anode surface, so as to maintain a continuous tight fit of the envelope as disintegration proceeds, thus preventing the slimes produced during electrolysis from passing from one area to another.—B. N.

*Battery plates; Storage* ——. A. E. White, London. From U.S. Light and Heat Corporation, Niagara Falls, N.Y., U.S.A. Eng. Pat. 112,133, Sept. 22, 1916. (Appl. No. 13,483 of 1916.)

THE paste is composed of a mixture of lead oxides, such as crystalline and amorphous lead monoxide, and water, one of the oxides being of a weight (per unit volume) which is relatively a small fraction of that of the other.—B. N.

*Commutator-section of cast alloy.* H. K. Sandell, Assignor to H. S. Mills, Chicago, Ill. U.S. Pat. 1,248,924, Dec. 1, 1917. Date of appl., June 18, 1917.

A CAST commutator-section for use in dynamo electric machinery is made of an alloy composed of copper 95 and nickel 5%.—W. E. F. P.

*Metal alloy [for electrical contacts].* U.S. Pat. 1,248,621. See X.

## XII.—FATS; OILS; WAXES.

*Copra and other coconut products; Study of* ——. A. J. Cox, Philippine J. Sci., 1917, A. 12, 49—53.

THERE are two large vegetable oil plants in the Philippine Islands, and several smaller ones are

in course of erection. In the tapahan (drying grill) method of drying copra, development of moulds and consequent deterioration of the copra is of frequent occurrence. Experiments have shown that the Californian method of treating deciduous fruits with the fumes of burning sulphur may be applied to the coconut. The nuts are opened and treated with the sulphur dioxide in a box to which only sufficient air to allow the sulphur to burn is admitted. They may then be kept for 2 weeks without moulds developing, and yield a very white copra, producing an oil free from rancidity and fully equal to the best Cebu oil. In the Manila markets, coconut oil is known as "Cebu sundried," "fair marketable Manila," and "Laguna," the first-named being worth about 2s. 6d. per 100 kilos. more than the Laguna grade. The distinction between these grades, however, is ill-defined, and it is suggested that the Government should subsidise good copra, and penalise smoked, dirty, mouldy, or imperfectly dried copra, in order to create a demand for the better grades. As the character of copra varies with the kind of nut and the district, it would be necessary to establish grades for different areas. These would be based not only on the proportion of oil, but also on the appearance, colour, and amount of moisture, the proportion of which may be roughly estimated by inspection by an expert.

—C. A. M.

*Copra and coconut oil.* H. C. Brill, H. O. Parker, and H. S. Yates. Philippine J. Sci., 1917, A, 12, 55—86.

The exports of copra and oil from the Philippine Islands in 1916 were 72,277,164 kilos. and 16,091,169 kilos. respectively, the copra representing about a third of the world's output. Owing to its poor quality, however, Philippine copra fetches a lower price than any other copra on the market. The oil usually obtained from it is discoloured and rancid and contains from 5 to 20% of free fatty acids. Unless the moisture is reduced to about 6% immediately after opening the nuts, the copra is attacked by various micro-organisms, which reduce the yield of oil and deteriorate its quality. Samples of copra from various localities in the Philippines contained from 10.4 to 29.1% of moisture, and laboratory experiments showed that under the most favourable conditions for the growth of moulds (10 to 20% of moisture) there was a loss of 25% or more of the total oil present. If copra contains sufficient moisture for the growth of brown mould (*Aspergillus flavus*), a loss of 30 to 40% of oil may be anticipated. This species is frequently associated with black mould (*A. niger*), which only appears on copra containing at least 12% of moisture. It causes a pronounced loss of oil (sometimes over 40%), and increases the acidity of the oil, although not to the same extent as brown or white moulds. Where green mould (*Penicillium glaucum*) alone is present, there is but little loss of oil, but its presence indicates a proportion of moisture favourable for the development of the other moulds. The white mould (*Rhizopus* sp.) requires an atmosphere saturated with moisture, and then only develops on fresh copra, and is killed as soon as the drying process begins. Hence, although it grows rapidly and destroys a large proportion of oil, it is the least important of the four moulds. The amount of water is unevenly distributed in copra. For example, the total percentage may be as low as 5%, sufficient to inhibit the growth of moulds, whereas the upper layer invariably contains more moisture than the lower layer (e.g., 10.5 to 24%), or sufficient to support mould growth. If, however, copra has once been properly dried to contain not more than about 6% of moisture, it will not subsequently absorb sufficient moisture for moulds to develop,

unless it is kept for prolonged periods in air saturated with moisture. Both the spores and the mycelia of these moulds will survive exposure to 50°C. for 9 to 12 hours. Sun-drying produces an excellent grade of copra, but owing to rainy periods is chiefly used only in the southern islands. In other parts the tapahan or grill method is used. In this process the nuts are freed from husks, divided, and placed on bamboo rods beneath which shells and husks are burned. When partially dry the copra is removed from the shells and the drying completed. The finished product contains not less than 20% of moisture, and not infrequently 28 to 29%. Mechanical drying apparatus has not been introduced commercially into the Philippine Islands, largely owing to the belief that the current of heated air causes loss of oil. Experiments are described, however, which show that copra can be dried in a current of air at 70° to 100°C. without loss of oil. Apart from mechanical drying, however, much better products than are now usually put on the market could be obtained by a combination of the sun-drying and kiln processes carefully applied. Good copra should contain from 4 to 6% of water, and 65% of oil, calculated on the anhydrous substance, with an acidity of less than 1% (as oleic acid), and should be free from dirt and smoke. The cost of refining commercial coconut oil is estimated at 8 pesos (1 peso = 50 cents U.S.A. currency) for each 1% of fatty acids per ton of oil.—C. A. M.

*Coconut oil; Methods for the production of pure* — H. O. Parker and H. C. Brill. Philippine J. Sci., 1917, A, 12, 87—94.

THE authors' experiments have shown that the oil cannot be readily expressed from freshly grated coconut, over 60% of the total oil remaining in the press-cake after being pressed at 1000 lb. per sq. in. By treating the material with water and live steam for 3 hours before expression, 80% of the total oil is removed by a single expression. The resulting emulsion when heated as in the native process always yields a coloured oil of burnt odour and taste, and no better results are obtained by heating the emulsion with salt, acids, or alkalis. The use of 10% sodium sulphite solution facilitates the separation, but the resulting oil is of inferior colour and taste. The treatment might be useful, however, for the manufacture of soap stock oils. The best method is to chill the emulsion for 24 hours at 15°C., which causes the water to separate, leaving an upper layer of solid fat and cellular tissue, and also checks the development of moulds. After removal of the water the solid layer is melted at a gentle heat, and passed through a filter-press, and the filtered oil sterilised for 30 mins. at 100°C. and stored in air-tight vessels. Oil thus prepared has a pleasant odour, is free from acidity or rancidity, and keeps much better than ordinary copra oil. Another method is to dry the shredded coconut in a current of air at 70°—85°C. until its moisture is reduced to 10%, which is sufficiently low to prevent the formation of emulsions. The material will then yield over 80% of its total oil in one expression. Commercial copra press-cake is used for stock foods and for fertilisers, but the product obtained by the method described is sufficiently pure for human consumption. Meal prepared from it had the following composition:—Water, 7.35; oil, 32.14; ash, 4.05; crude fibre, 37.12; and proteins, 20.34%. It might be used with advantage as a substitute for wheat flour and lard in the preparation of edible products.—C. A. M.

*Coconut oil; Rancidity of Philippine* — H. C. Brill and H. O. Parker. Philippine J. Sci., 1917, A, 12, 95—110.

CERTAIN oils which are unmistakably rancid do not



restore the colour to decolorised magenta, whereas other oils which are not rancid give a coloration with the reagent. Hence no reliance can be placed upon the test as a criterion of rancidity, and the same applies to the test with diazobenzene-sulphonic acid. A high proportion of free fatty acids does not necessarily accompany rancidity. Distillation with steam removes rancidity, but does not cause pronounced change in the acidity, whilst neutralisation with alkali and washing with water removes the acidity but not the rancidity. There appears to be no definite relationship between the Reichert-Meissl value or the iodine value and the rancidity of coconut oil, but the proportion of soluble fatty acids shows indications of a relationship. The acetyl value may afford evidence of rancidity, but does not measure the degree of rancidity. Although a few samples of rancid oils gave low oxidisability values (this J., 1916, 898), the test appears to afford confirmatory evidence of rancidity, and where the value was high the oil was invariably rancid. Copra which has been attacked by moulds or copra with coconut milk adhering to it contains oxidising enzymes, some of which might be separated with the oil. Such enzymes, if present, would probably have considerable influence on the development of rancidity (see also this J., 1906, 381; 1910, 704, 1330).—C. A. M.

*Castor oil used for lubricating aeroplane motors; Detection of foreign oils in —.* C. Frabot. Ann. Chim. Analyt., 1918, 23, 7—11.

FURTHER investigation of the method described previously (this J., 1917, 1279), which depends on the relatively small solubility of castor oil in petroleum spirit and on the solubility of the petroleum spirit in the oil, shows that the character of the petroleum spirit used is of importance. The solubility of castor oil increases with the boiling point of the spirit; for instance, when the spirit is fractionated, the first fraction, b.pt. 33.2° C., dissolves 6.51 grms. of the oil under the conditions given (*loc. cit.*), whilst the last fraction, b.pt. 76° C., dissolves 13.0 grms. A fraction, b.pt. 184° C., obtained from ordinary motor petrol, dissolves 25.3 grms. of castor oil. The increase in volume of the oil due to dissolved petroleum spirit does not depend so much on the kind of spirit used; it varies from 10.5 c.c. with very light spirit to 18 c.c. with certain high-boiling fractions. It is therefore suggested that only the portion of petroleum spirit boiling between 35° and 65° C. be used for the test; with this spirit pure castor oil shows an increase in volume of 11.5 c.c. and the weight of oil dissolved is 8.85 grms.—W. P. S.

*Sperm oils used for burning purposes; Characteristics of —.* A. G. Blakeley and E. A. Reilly. J. Ind. Eng. Chem., 1917, 9, 1099—1100.

EXAMINATION of a large number of samples of genuine sperm oil, intended for burning in miners' safety lamps of the Davy type, gave results agreeing, in the main, with recorded values. Some of the constants, however, showed a wider variation than commonly accepted, viz.:—Refractive index (15.6° C.), 1.4619 to 1.4669. Fatty acids:—Sp. gr. (15.6° C.), 0.893 to 0.899; neutralisation value, 186.8 to 216.2; "titer" test, 6.2° to 13.6° C. Alcohols:—Iodine value, 63.9 to 74.1; m.pt., 20.0° to 27.0° C.; and saponification value of acetate, 189.5 to 216.0. The "flake-test," i.e., the temperature at which solid matter separated from the oil, varied from 38° to 50° F. (3.3° to 10.0° C.). Sperm oil containing from 1 to 4% of oleic acid caused a slight crust to form upon the wick of the lamp but otherwise gave satisfactory results in burning, but the addition of 6% made

the oil unsatisfactory. These results show that the injurious effect of free acids upon a lamp oil has been somewhat overestimated in the case of sperm oil. In judging as to the suitability of sperm oil as a lamp oil the iodine value and refractive index should be taken into account as well as the acidity and the other physical values.—C. A. M.

*Wool-fat (lanoline) substitute and the preparation of cetyl alcohol.* S. Axelrad. J. Ind. Eng. Chem., 1917, 9, 1123—1125.

SPERMACETI (20 grms.) is saponified with alcoholic potassium hydroxide solution, the alcohol evaporated, the soap dissolved in water, and the soap solution treated with excess of 10% calcium chloride solution. The calcium soap is separated, dried at 100° C., and distilled at 340° C. A cheaper method, which also gives a larger yield of cetyl alcohol, is to mix 20 grms. of calcium oxide containing about 5% of water with 15 grms. of melted spermaceti, and to heat the mixture, with occasional stirring, for about 6 hours. The mass is then cooled, water expelled at 100° C., and the temperature then raised to 340° C. for the distillation of the cetyl alcohol. The yield is over 40% of the weight of spermaceti, as compared with the theoretical yield of about 45%. The distilled cetyl alcohol is perfectly white and melts above 49.5° C. A copper still is preferably used for the distillation. Lanoline substitutes and cold creams may be made by mixing suitable proportions of a petroleum base with cetyl alcohol and water, and preferably with about 5% of lanoline to give the "unctuous" property of wool fat. A preparation suitable for pharmaceutical purposes consists of:—"Petroleum" base, 70; paraffin wax (m.pt. about 60° C.), 20; cetyl alcohol, 10; anhydrous lanoline, 10; and water, 100 parts. This preparation has the same properties as wool fat, and has the advantage of not becoming rancid.—C. A. M.

*Basking-shark liver oil; Saturated hydrocarbons in —.* M. Tsujimoto. J. Ind. Eng. Chem., 1917, 9, 1098—1099.

THREE genuine samples of the liver oil from the basking-shark (*Cetorhinus maximus*), the largest of the shark tribe, were pale yellow to orange liquids of low sp.gr., and contained 41.92 to 55.51% of unsaponifiable matter, in which the unsaturated hydrocarbon squalene (this J., 1916, 609; compare Chapman, this J., 1917, 392) was identified. One of the samples also contained about 10% of a yellow liquid saturated hydrocarbon, probably  $C_{15}H_{32}$ , which was not present in the other two samples. This sample had the following characters:—Sp. gr. at 15°/4° C., 0.8839; acid value, 1.09; saponif. value, 102.45; iodine value (Wijs), 178.3; refractive index at 20° C., 1.4773; and unsaponifiable matter, 41.92%. The unsaturated hydrocarbon, squalene, forms hexahydro-halides of the general formula,  $C_{30}H_{50}6HX$ , which may be readily crystallised and serve for the identification of the hydrocarbon.—C. A. M.

*Preparation of an edible fat and meat-extract substitute from boncs.* Kraus. See XIXA.

#### PATENTS.

*Grating, "defibrating" or pulping machines [for coconuts and other oil-bearing products].* H. K. Reeves, London. Eng. Pat. 112,135, Sep. 27, 1916. (Appl. No. 13,744 of 1916.)

CELLULAR tissue of the coconut or other oil-bearing nuts is broken up and pulped by means of a rotating cylindrical drum, etc., the face of which

bears against the corresponding face of a curved grating plate, to which a reciprocating motion is communicated, e.g., by means of cam mechanism. The rear surface of the grating plate may also have prepared faces adapted to bear against prepared surfaces in the casing of the machine.

—C. A. M.

*Utilisation of certain nuts or seeds in the manufacture of artificial butter or the like.* Eng. Pat. 112,164. See XIXA.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Preparation, by the distillation and by the combustion of wood, of acid coagulants, and their effect on the quality of —.* B. J. Eaton and G. S. Whitby. Agric. Bull. Fed. Malay States, 1917, 5, 124—131.

PyroLIGNEOUS acid prepared by the dry distillation of wood from the mangrove and the rubber tree, when used for the coagulation of latex, yielded a darker sheet or slab rubber than that produced by acetic acid, although in the case of crêpe the presence of a small proportion of sodium bisulphite permitted the formation of a pale product. A coagulant was also obtained by passing smoke from rubber wood in slow combustion through water and so extracting the soluble constituents. When used at the same concentration of acid for the coagulation of latex, the filtered "smoke solution" gave products of similar appearance to those obtained with the use of pyroLigneous acid. In both cases, however, the rate of vulcanisation of the resulting slab or smoked sheet rubber was inferior to that of rubber obtained with simple acetic acid coagulation, the difference being more marked with the "smoke solution" product. The retardation of vulcanisation is attributed to the presence of phenolic substances and also of formaldehyde. Tests as to the effect of the addition of phenol or cresol to latex before coagulation with acetic acid showed a marked tendency to the formation of discoloured coagula, and the resulting smoked sheet rubber vulcanised more slowly than similar rubber produced in the absence of phenol or cresol; with slab rubber, however, the difference in rate of vulcanisation was inappreciable. The conclusion is drawn that although the smoke employed in the preparation of fine hard Para rubber may act as a preservative, it also has a bad effect on the rate of vulcanisation.—D. F. T.

*Coagulation of latex in the presence of sugars.* B. J. Eaton. Agric. Bull. Fed. Malay States, 1916, 5, 48—52.

IN reviewing the results of an investigation by Gorter and Swart of the West Java Rubber Testing Station and correlating them with the results of Eaton and Grantham, it is stated that the addition of sugar to latex aids coagulation by the bacterial formation of lactic acid, together with a smaller quantity of acetic acid and a trace of succinic acid. Lactic acid also plays an important part in the slow coagulation of latex with minimal quantities of acetic acid, the lactic acid in this case being produced by the action of micro-organisms on the sugars present in the latex. In order relatively to favour the growth of the lactic micro-organisms, it is necessary either to exclude air, or to add at least 0.2% of sugar, the former process checking the production of putrefactive and other bacteria, whilst the latter enables a sufficiently rapid production of lactic acid to inhibit the action of the other bacteria. The addition of 0.5 grm. of sodium bisulphite per litre

of latex does not interfere with the coagulation, using the additional 0.2% of sugar. Hydrofluoric acid, sulphuric acid, and alum are not recommended as coagulants, especially for making sheet rubber. Fermentation of the milk of young coconuts yielded a liquid containing a disappointingly low proportion of acetic acid, the action of the acetifying micro-organisms apparently having been inhibited by the formation of lactic acid.—D. F. T.

*Rubber; Preparation of raw — with special reference to "slab" rubber and variability.* B. J. Eaton. Agric. Bull. Fed. Malay States, 1917, 5, 183—188.

"SLAB" rubber as a substitute for sheet rubber possesses the advantage of greater rate of vulcanisation, increased uniformity in this respect, and superior tensile properties. There is no reason why the introduction of "slab" rubber should cause confusion or increase the difficulties of the manufacturer, and it will probably be advantageous to ship the rubber as "virgin slab."—D. F. T.

*Rubber allowed to become damp; Development of chromogenic organisms in dry raw —.* B. J. Eaton. Agric. Bull. Fed. Malay States, 1917, 5, 177—179. (See also this J., 1917, 1222.)

RUBBER despatched from the estate in a satisfactory condition is sometimes found to be affected with "spot disease" on arrival in London or other buying centres. The trouble is due to chromogenic organisms which develop in damp rubber, and it is now shown by experiment that the same development of coloured spots may occur in perfectly clean pate crêpe if this is wetted and rolled loosely, ten days being a sufficient period for a marked effect. Sheet rubber rolled into a cylinder direct from the sheeting machine and also "slab" rubber show no development of such pigment until they are cut, when the new surface becomes affected; thin crêpe which, whilst still wet, has been converted into thick or blanket crêpe is more liable to affection than original thick crêpe.—D. F. T.

*Rubber; Effect of copper on crude —.* C. P. Fox. J. Ind. Eng. Chem., 1917, 9, 1092—1033.

IN comparative tests in which sheet rubber was covered with discs of metal and exposed for several days at 150° F. (65.5° C.), Dewar found that copper had the greatest action owing to its promoting oxidation. His results were confirmed by Thompson and Lewis (this J., 1891, 717). In accordance with these observations a coating of copper for use in vulcanising rubber to iron has recently been superseded by a plating of other metals. In the author's experiments pieces of crude sheet rubber were treated with 1% aqueous copper acetate solution, copper acetate solutions containing 10% of acetic acid and 10% of ammonia respectively, and oil mixed with 1% of dry copper acetate. The duration of the experiments was about 3 months, and parallel tests were made in a bright light and in the dark. It was found that copper acetate will cause "tackiness" in crude rubber, the amount of action being proportional to the quantity of reagent and duration of contact, but not much affected by light or heat. The control samples of crude rubber ("pale plantation crêpe") were not changed in colour or texture when exposed to the light under the same conditions as the treated samples, and their behaviour affords an index of the value of this type of rubber. The presence of acetic acid had no material influence on the production of tackiness, but ammonia had a pronounced effect, producing a hard, cracked surface. The mixture of lubricating oil (containing



rancid vegetable oil) and copper acetate had a greater action on the rubber than the same oil by itself. This confirmed the observations of Weber ("Chemistry of Rubber") as to the action of cloth containing copper (used in dyeing) upon rubber used for waterproofing, especially when oil or grease is also present in the cloth.—C. A. M.

## XV.—LEATHER; BONE; HORN; GLUE.

*Tannin content of Pacific Coast trees.* H. K. Benson and F. M. Jones. *J. Ind. Eng. Chem.*, 1917, 9, 1096—1098.

THE following amounts of tannin were extracted:—Western larch (*Larix occidentalis*), wood, 6.7; bark, 10.6; western yellow pine (*Pinus ponderosa*), wood, 8.9; bark, 10.9; western hemlock (*Isuga heterophylla*), wood, 1.0; bark, 10.93; dogwood (*Cornus nuttallii*), wood, 1.3; bark, 1.7; cottonwood (*Populus trichocarpa*), wood, 1.8; bark, 4.7; and alder (*Alnus oregona*), wood, 0.7; bark 3.3%. The wood extract of the western larch gives a pleasant, light red colour to the tanned hide. As the wood of this species also gives a high yield of alcohol when hydrolysed, this tree is more suitable for chemical utilisation than for timber. The tannin content of the western hemlock is equal to that of the eastern species. The bark has been regularly used by tanners on the Pacific Coast. The western pine, which is chiefly used for making boxes, yields a satisfactory amount of a good extract and the saw-mill waste of this species might be utilised in the manufacture of tannin extract. Of the deciduous woods the only one suitable for the manufacture of extracts is the dogwood. The extract could not be used alone for tanning, but might be employed as a red dye in conjunction with other tanning extracts. It was found that Douglas fir wood in slabs may be advantageously seasoned for a year before being used for the preparation of extracts. The effect of the hydrolysis, and, possibly, oxidation taking place during seasoning is to increase the amount of tannin (e.g., from 5.92 to 7.5%), to increase the total solids (e.g., from 14.92 to 15.8%), and to reduce the non-tannins (7.10 to 6.2%).—C. A. M.

*Tanning [of fishing nets] and the study of tannins.* W. Tombrock. *Chem. Weekblad*, 1917, 14, 1143—1150.

AN account of the methods employed by the Dutch fishermen for preserving their nets. The usual procedure is to immerse the net in a hot solution of tannin for some time, and then to dry it. The author has found that if the net is then immersed for 15 minutes in a solution containing 1% of copper sulphate and 0.7% of ammonia, and again dried, the durability is much increased. After this treatment, it is important for the net to be used for fishing as soon as possible. The method is also adapted to the preservation of other forms of netting.—A. J. W.

## PATENT.

*Leather-like material; Method of manufacturing* —. A. G. Emery, New York, Assignor to Rubber Regenerating Co. U.S. Pat. 1,249,360. Dec. 11, 1917. Date of appl. July 14, 1916.

A MIXTURE of linseed oil and calcium rosinate is boiled until it is of a gelatinous consistency. It may be coloured if desired by incorporating American ochre and iron oxide, and the resulting product is used to impregnate fibrous material and is then oxidised. A solvent may be used to facilitate the impregnating process, and is then allowed to evaporate before the oxidation process. F. Sp.

## XVI.—SOILS; FERTILISERS.

*Soils; Movement of soluble salts through* —. M. M. McCool and L. C. Wheeting. *J. Agric. Res.*, 1917, 11, 531—547.

EXPERIMENTS were carried out on moist soils in tubular containers, the short middle sections of which were filled with the soil and the salt to be studied, and the longer end sections with the soil alone. The containers were then closed, or left open if evaporation was to be studied, and placed in a closed thermostat at 18° C. It was found that soluble salts do not long remain localised but pass at a moderately rapid rate from regions of high to regions of lower concentration. When moisture is being lost by evaporation an upward movement of the salt takes place much more rapidly than any downward movement; this is especially marked in sandy soils. As the salts move through the soil, the soil solution changes in character, as a result of reactions between the salt and the soil particles as well as of diffusion. These changes may spread much farther than the soil mass receiving the application.—J. H. J.

*Hawaiian soils; Reaction of — with calcium bicarbonate solution, its relation to the determination of lime requirements of soils, and a rapid approximate method for the determination of lime requirements of soils.* M. O. Johnson. *J. Ind. Eng. Chem.*, 1918, 10, 31—33.

WHETHER lime is applied to the soil as oxide, hydroxide, or carbonate, the main reaction of the limed soil appears to be with calcium bicarbonate in solution. Absorption by soils from calcium bicarbonate solutions increases with the period of contact, approaching an equilibrium. With a decrease in the concentration of the bicarbonate solution, there is a decrease in the actual absorption, but an increase in the percentage absorption of the calcium bicarbonate in solution, indicating that at a certain concentration, absorption would be complete under prolonged contact with the soil. The total amount of calcium carbonate (expressed as % of wt. of soil) in the solution at the concentration at which absorption by a given soil is complete, expresses a definite absorptive capacity for that soil, and has been named the "minimum absorption." For mainland soils, the following procedure is recommended as a method of determining lime requirements. 200 c.c. of N/100 calcium bicarbonate solution is measured into a 500 c.c. Erlenmeyer flask and 10 grms. of the soil is added. The flask is shaken vigorously for 10 secs. The solution is filtered and 100 c.c. titrated with N/10 acid, using methyl orange as indicator. The difference in titration between 100 c.c. of the filtrate and of the original solution is proportional to the amount of calcium carbonate required by the soil. Multiplying by 10 the percentage of calcium carbonate required gives directly the tons of calcium carbonate per 2,000,000 lb. of soil, i.e., per acre.—L. A. C.

*Acidity of soils; Determination of the — and its value in determining the quantity of lime to be applied in practice.* J. Grantham. *Agric. Bull. Fed. Malay States*, 1917, 5, 138—149.

TRUOG's method (this J., 1916, 699) by which he determines the "active" and "latent" soil acidity and the avidity of the active acids, is considered to be the most satisfactory measure of the lime requirements of soils.—W. G.

*[Soil] liming experiments.* B. Bunting. *Agric. Bull. Fed. Malay States*, 1916, 4, 373—380.

THE results obtained from the growth of one or

at the most two crops of six different kinds of plants on one particular type of soil show that a decided improvement in crop production may be obtained by the application of lime in quantities not sufficient entirely to neutralise the acidity of the soil. The improvement in crop production is not directly proportional to the reduction of soil acidity. The author considers that one ton of quicklime per acre applied every four years will give the best return.—W. G.

*Reverted phosphate.* C. C. James. J. Ind. Eng. Chem., 1918, 10, 33—35.

EXPERIMENTS both on the laboratory and factory scale showed that the water-soluble phosphoric acid in acid phosphate (superphosphate) mixed with lime or calcium carbonate decreases on standing, the change being accompanied by a rise in temperature, which is greater with lime than with calcium carbonate. The decrease is more rapid at first, becoming slower on further standing. On trying the effect of the reverted phosphates on crops, it was found that it is just as valuable as the water-soluble phosphate when applied to sugar-cane on upland, ferruginous, clayey soils. Experiments upon rice with the reverted phosphate also showed favourable results.—L. A. C.

*Bonedust : its adulteration with phosphate rock.* C. E. Chapman. Dept. Chem., S. Australia, Bull. 9, 1917. 11 pages.

PHOSPHATE rock can be detected in bonedust by shaking the sample with Sonstadt's solution (a solution of mercuric and potassium iodides, sp. gr. 2.8). The bonedust floats, but 80 % of the rock phosphate sinks. For proper separation the tube containing the solution and sample of dust should be centrifuged for four or five minutes. The amount of tricalcium phosphate present in the material which floats in Sonstadt's solution can be determined in order to see if it complies with the legal standard of the country concerned. Finally the rock phosphate settling in the liquid can be examined microscopically.—W. G.

*Calcium cyanamide : Determination of nitrogen in —.* B. Turkus. Ann. Chim. Analyt., 1918, 23, 3—5.

ONE gm. of the sample is heated for 30 mins. with a mixture of 40 c.c. of concentrated sulphuric acid and 10 c.c. of water; the ammonia formed is then determined by distillation. If the sulphuric acid used is weaker or stronger than the concentration mentioned, the digestion must be prolonged for more than 30 mins. in order to decompose the cyanamide completely.—W. P. S.

*Commercial aspect of recovery of polish in pig iron manufacture.* Chance. See VII.

*Estimation of phosphoric acid as magnesium pyrophosphate.* Balareff. See XXIII.

#### PATENTS.

*Phosphate rock ; Method of treating — [to produce a fertiliser composition].* H. H. Meyers, Pittsburgh, Pa., Assignor to Armour Fertilizer Works, Chicago, Ill. U.S. Pat. 1,246,636, Nov. 13, 1917. Date of appl., Jan. 15, 1917.

FINELY divided phosphate rock, mixed with water to form a sludge, is treated with a mixture of sulphurous and sulphuric anhydrides produced by subjecting a mixture of sulphurous gases and air to the action of a suitable catalyst.—W.E.F.P.

*Manufacture of composition fuel.* Eng. Pat. 112,341. See IIA.

*Process of recovering values from lime mud [from ammonia-soda process].* U.S. Pat. 1,249,739. See VII.

*Method of increasing the yield and reducing the production cost of sugar cane.* U.S. Pat. 1,249,355. See XVII.

### XVII.—SUGARS ; STARCHES ; GUMS.

*Sugars, starches, gums, proteins, and organic acids ; A precipitant for —.* W. Schulte. Chem.-Zeit., 1917, 41, 822.

SUCROSE, invert sugar, dextrose, lactose, dextrans, and starch are precipitated from their aqueous solutions by ammoniacal lead acetate solution; gum arabic, gum tragacanth, albumin, casein, pepsin, peptone, and gelatin are also precipitated by the reagent. Whilst citric, tartaric, malic, tannic, gallic, and succinic acids, and pyrogallol are precipitated by lead acetate alone, benzoic, salicylic, and picric acids, and phenol yield precipitates only with ammoniacal lead acetate. The ammoniacal lead acetate solution must be freshly prepared (it readily absorbs carbon dioxide from the atmosphere) and is made by mixing 100 c.c. of saturated lead acetate solution with 26 c.c. of water and 8 c.c. of ammonia (sp. gr. 0.91). One part of gum arabic or phenol in 20,000 parts of water gives a turbidity or precipitate with the reagent; with sugars the minimum limit is about 1 part in 10,000 of water. The precipitates formed may be collected, washed with dilute ammonia, dried at 100° C., and weighed, or the precipitate may be ignited and the residual lead oxide weighed. The following factors are given for obtaining the quantity of substance present either from the weight of the precipitate or of the lead oxide :—

	Sucrose.	Gum arabic.	Phenol.
From weight of precipitate	0.1727	0.2564	0.2636
From weight of lead oxide	0.2386	0.3703	0.3125

Since glycerol is not precipitated by the reagent, the latter may afford a means of separating sugars, etc., from glycerol in the determination of this substance in wines, etc. Attention is directed to the fact that sugars are, to a certain extent, precipitated by basic lead acetate from their neutral solutions. In the clarification of saccharine solutions with basic lead acetate it is necessary, therefore, to add a small quantity of acetic acid if the solution is not already acid in reaction.—W. P. S.

*Lime cake [calcium saccharate] analysis ; A quick method for —.* A. N. Clark. J. Ind. Eng. Chem., 1918, 10, 51.

THE required weight of lime cake is weighed into a counterbalanced, nickel-plated, copper beaker of about 300 c.c. capacity, the calculated volume of acetic acid added from a pipette, and the mass mixed with a small pestle. A charge of basic lead acetate solution is added from a Sachs-Le Docte or Kruger pipette, the mass again mixed with a pestle, and poured on to a filter. This method is found to be much more rapid and less liable to loss by foaming and spilling, than the usual procedure. When the "free" sugar is to be determined, water is added in place of acetic acid.—L. A. C.

#### PATENTS.

*Sugar cane ; Method of increasing the yield and reducing the production cost of —.* C. F. Eckart, Oloa, Hawaii. U.S. Pat. 1,249,355, Dec. 11, 1917. Date of appl., June 25, 1917.

COVERED nitrate beds are established between the rows of canes, e.g., by applying lime compounds



or a mixture of organic matter with lime or calcium carbonate, and covering the spaces so treated with a dark-coloured, opaque, waterproof paper or fabric in contact with the ground.—J. H. L.

*Molasses ; Manufacture of alcohol from* —. G. H. Hughes, London. Eng. Pat. 112,232, May 21, 1917. (Appl. No. 7324 of 1917.)

MEGASSE is mixed with molasses in the proportion of about 15 parts of fine, dry, screened megasse to 85 of concentrated hot molasses ; the solid mass is formed into blocks or bales suitable for transport and is subsequently "broken down" with water and fermented.—J. F. B.

## XVIII.—FERMENTATION INDUSTRIES.

*Brewing materials.* P. Petit. Brasserie et Malterie, 1917. 7, 129—132.

THE author discusses the use of potato-starch, manioc, chit-malt, and raw barley as malt adjuncts. The first is satisfactory (cp. Koudelka, this J., 1917, 399) but expensive. Manioc was employed in several French breweries about 12 or 15 years ago, until a very heavy import duty was placed upon it because it was stated to be cyanogenic. When digested with water it produces about 3 mgrms. of hydrogen cyanide per 100 grms., but this small amount is entirely eliminated in the course of brewing. Manioc requires cooking, like rice or maize, and owing to its slightly alkaline reaction it is advisable to add a small quantity of acid, e.g., 75—100 grms. of phosphoric acid per cwt., before the cooking process. Treated in this way it forms an excellent malt-adjunct. Experience of French brewers with the short-grown or chit-malt advocated by Windisch (see this J., 1917, 1188) has been unsatisfactory. It is difficult to grind properly without a special mill, and does not saccharify completely during the ordinary mashing process ; even when it is first cooked to gelatinise the starch, saccharification and wort-filtration are unsatisfactory. The author considers that raw barley might just as well be used, as all malting expenses are thus eliminated ; the raw grain is ground, preferably in a special mill, and then cooked, or it may simply be steeped as if for malting and cooked without grinding if it is well agitated during cooking. The raw barley may amount to 10—12 % or more of the malt employed.—J. H. L.

*Brewing ; Couch grass as malt-adjunct in* —. F. Koritschoner. Chem.-Zeit., 1917, 41, 797—798.

IT has been proposed in Germany to utilise the rhizome of couch grass (*Triticum repens*) as brewing adjunct, in view of the present scarcity of malt. The dried rhizome yields about 20 % of extract, derived chiefly from a reserve carbohydrate, trititin, which forms lævulose on hydrolysis. The practical difficulties attending its use in brewing are very great, for it is a very bulky material and requires 10—15 times its weight of water in mashing. Mashed alone, it was found to produce worts dirty red in colour and not altogether pleasant in odour and flavour. These worts developed a peculiar odour during fermentation and the corresponding beers became undrinkable after storage.—J. H. L.

*Worts ; Artificial cooling of* — in closed coolers, and the elimination of glutin. L. Heintz. Woch. Brau., 1917, 34, 221—224, 230—235.

H. T. BROWN has shown that the nitrogenous substances which separate from solution during the cooling of brewers' wort are formed by the combination of wort-proteins with hop-tannins, and that agitation of the wort during cooling

promotes their separation in a flocculent form (see this J., 1913, 442). The author found that with Continental worts, which are of lower gravity and less heavily hopped than those studied by Brown, agitation does not usually bring about flocculation of the precipitate. He suggests that the tendency to flocculate depends on the amount of tannin-protein compounds present. An important means of promoting flocculation is to maintain in suspension, during the cooling process, the proteins coagulated in the copper. A process of wort cooling is described in which this condition is realised. The hot wort, freed from hops but containing the coagulated proteins, is run into a large closed receptacle to a depth of 3—5 ft. From the cover of this receptacle a number of tubular cooling units, resembling ordinary vertical refrigerators in shape, are suspended, so as to be almost completely immersed in the wort. Cold water is circulated through the coolers and the wort is kept in constant agitation by a rotating paddle or by injection of sterile air. When cooled to the pitching temperature the wort may be allowed to stand for a time to allow the precipitate to subside, or it may be filtered. This process enables the whole of the heat of the wort to be recovered, and can be used to heat the water required for mashing. It also protects the wort against infection.—J. H. L.

*Wine ; Plastering of grapes and the increase of acidity and sulphates in the* —. A. Borntraeger. Giorn. Vinicolo Ital., 1917, 43, 412—414. Bull. Agric. Intell., 1917, 8, 1138—1149.

IT is well known that addition of calcium sulphate to grapes at the time they are put in the vat increases the acidity of the wine produced, and the reaction which takes place has been represented by Chancel as follows :— $2C_6H_5KO_6 + CaSO_4 = C_6H_5CaO_6 + C_6H_5O_6 + K_2SO_4$ . The author made comparative experiments in three successive years, with white "Catalanesca" grapes from Vesuvius, with and without addition of pure calcium sulphate. After vatting and filtering, the sulphates, expressed as potassium sulphate, and the total acidity, expressed as free tartaric acid, were determined in the wines. In all cases the "plastering" was found to have increased the acidity, but not to the extent calculated from the amount of sulphate present, according to the above equation. One reason for this discrepancy is that the sulphate in the wines consisted in part of unaltered calcium sulphate, and other contributory causes are suggested.—J. H. L.

*Wine-making experiments with sulphur dioxide.* C. Mensio. Staz. Sperim. Agrar. Ital., 1917, 50, 300—314. Bull. Agric. Intell., 1917, 8, 1140—1141.

COMPARATIVE experiments were made on the use of metabisulphite and certain other recognised preparations of sulphurous acid which contain also phosphoric acid and ammonia, viz., Ottavi mixture, Hubert's "sulphophosphate," and Jacquemin's "biosulphite" (cp. Dalmasso and Sutto, this J., 1916, 134). Black grapes in fairly bad condition and with abundant fungoid growth were employed. The wines made with use of sulphur compounds were more brilliant and possessed an intensity of colour about 30—40 % greater than in the case of the control. This is attributed to the influence of the sulphurous rather than the phosphoric acid, for the results with metabisulphite were slightly better than with the other preparations. About 80 % of the added sulphur remained in the wine, the greater part in combination with acetaldehyde. No trace of ammonium salts was found in any of the wines, and the glycerol-content was in all cases low, about 6.8 grms. per 100 grms. of alcohol. The

total acidity and the content of organic acids in wines made with use of sulphur preparations were higher than in the control, but the volatile acidity was lower. The results indicate that the use of sulphur dioxide in any form is advantageous in wine-making, and that metabisulphite gives somewhat better results than the other preparations used.—J. H. L.

*Wine-making; Substitution of calcium sulphite for potassium metabisulphite in —.* F. A. San-nino. *La Revista Viticult.*, 1917, 23, 330—331. *Bull. Agric. Intell.*, 1917, 8, 1142.

OWING to the present scarcity of potassium salts the author studied the suitability of various substitutes for potassium metabisulphite, and obtained good results with gaseous sulphur dioxide, and with solutions of sulphur dioxide in water or must. Pure calcium sulphite (free from carbonate and containing 40%  $\text{SO}_2$ ) also proved satisfactory, but it was found necessary to use about 50% more of it than of metabisulphite.—J. H. L.

*Alcohol; Determination of —.* C. J. Haines and J. W. Marden. *J. Ind. Eng. Chem.*, 1917, 9, 1126—1127.

ALCOHOL can be "salted out" from solutions containing not less than 1 to 2% thereof, by the addition of potassium fluoride. Ten c.c. of the liquid at 15-6° C. (or 5 c.c. diluted to 10 c.c. if the alcoholic strength exceeds 50%), is treated in a centrifugal tube (graduated in 0.01 c.c.) with potassium fluoride until the contents of the tube measure about 13 c.c. A small crystal of malachite green is added to colour the separated alcoholic layer, and the tube closed, shaken vigorously for 2 to 3 mins., allowed to cool, and centrifuged, and the volume of alcohol read, a correction of 0.001 c.c. being made for each 1° C. by which the temperature of the room differs from 15.6° C. To allow for incomplete precipitation and traces of alcohol adhering to the sides of the tube, an addition of 0.15 c.c. must be made to the reading. The method is applicable to beer, cider, spirits, tinctures, etc., and gives results agreeing within  $\pm 0.4\%$  of those obtained by determining the sp. gr. of the distillate. In the case of tincture of iodine the presence of the iodine has little effect upon the volume of separated alcohol.—C. A. M.

*Hops; Dried "spent" —.* J. L. Baker and H. F. E. Hulton. *J. Inst. Brew.*, 1917, 23, 455—457.

FOR breweries equipped with grains-drying plant it is at the present time a profitable method of disposing of spent hops to dry them, and then grind them finely so that they can be sold for incorporation in cattle foods. The average composition of the dried and ground material is as follows:—Moisture, 4.8%; ash, 7.4%; ether extract (chiefly resins, with traces of fat), 9.0%; proteins ( $\text{N} \times 6.25$ ), 21.9%; crude fibre, 21.6%; pentosans, 13.0%; assimilable carbohydrates, 35.3%. Calories per pound, 1062. Instances of the spontaneous heating and even firing of consignments of the material have occurred. This heating was not due to packing the hops whilst still warm, but was probably in some way connected with spontaneous oxidation of the hop resins, for the heated material acquired an odour like that of linseed oil and the iodine-value of the resins fell from 118 to 93. A preventive measure, which has proved successful up to the present, is to spread the dried and ground hops over a large surface in a fairly thin layer for 24 hours, before the material is packed into bags. During this exposure to the air the spent hops acquire the oily odour already mentioned, and it is possible that the changes which take place may enhance the feeding value of the material.—J. H. L.

*Yeast; Autolysis of — and the influence of its products of proteolysis on the development of yeast and lactic bacteria.* P. Vansteenberghe. *Ann. Inst. Pasteur*, 1917, 31, 601—630.

THE value of autolysed yeast water must be attributed to the presence of a mixture of proteolytic products in which peptone plays the principal part, and in which the small amounts of a series of substances, such as leucine, tyrosine, and asparagine, exert, independently of the peptone, a favourable influence on the development of yeast and of lactic bacteria also. The value of malt extract probably also lies in the presence of a series of proteolytic products analogous to those from autolysed yeast. (See also *J. Chem. Soc.*, Mar., 1918.)—W. G.

*Enzymes concerned in the decomposition of dextrose and mannitol by B. coli communis. II. Experiments of short duration with an emulsion of the organisms.* E. C. Grey. *Proc. Roy. Soc.*, 1918, [B], 90, 75—92.

AN emulsion of *B. coli communis* in saline solution containing an amount of bacteria which would weigh, when dry, 1 grm., is sufficient to ferment 40 grms. of dextrose completely in 48 hours in the presence of calcium carbonate. The actual amount of any of the products of fermentation, viz., alcohol, carbon dioxide, formic, acetic, lactic, and succinic acids, obtained is dependent on the concentration of salts and the temperature of fermentation. By comparison of the various amounts produced in different experiments, the author draws the conclusion that alcohol, acetic acid, and succinic acid are all formed from a common intermediate substance by the action of the same enzyme. The formation of lactic acid appears to be quite independent of that of the other products of fermentation, and its production is therefore due to a separate enzyme. The addition of peptone to the culture medium containing dextrose leads to the production of a greater proportion of lactic acid, and a lesser proportion of alcohol, acetic acid, and succinic acid. The products of the bacterial decomposition of mannitol closely resemble in amounts those obtained from dextrose, which result is in harmony with the author's view that the fermentation of various carbohydrates and allied substances by bacteria is effected by a single set of enzymes, the action of which is common to all such cases of fermentation. (See also *J. Chem. Soc.*, Mar., 1918.)—H. W. B.

*Enzymes concerned in the decomposition of dextrose and mannitol by B. coli communis. III. Various phases in the decomposition of dextrose by an emulsion of the organisms.* E. C. Grey. *Proc. Roy. Soc.*, 1918 [B], 90, 92—106.

BY examining portions of a fermenting solution containing dextrose and bacteria at the end of 12, 24, 48, and 72 hours respectively, it was found that during the first 12-hour period the number of living bacteria diminishes almost to nothing, whilst during the second period a rapid growth of bacteria sets in, so that at its close the number present greatly exceeds that at the commencement of the experiment. Corresponding with these changes there is a cessation in the production of lactic acid in the first period, the chief products being carbon dioxide, alcohol, acetic and succinic acids, whilst subsequently in the third period, when the number of bacteria is at a maximum, the production of lactic acid is so much accelerated that it forms 70% of the fermented sugar. The period of exalted bacterial growth is associated with the transformation of a large proportion of the dextrose into a complex carbohydrate which only reduces Fehling's solution after hydrolysis. A synthesis of fat was also observed on one



occasion in circumstances which could not be again realised. The extent of the synthesis during one period is quantitatively of the same order as the degradation which ensues in the following period. The author draws the conclusion that the fermentation is brought about, not by the cells as such, but by enzymes in the cells. Different conditions result in variations in the amounts of the products of fermentation due to the varied influence of the altered conditions on the operation of the different enzymes present.

—H. W. B.

*A precipitant for sugars, starches, gums, proteins, and organic acids.* Schulte. See XVII.

*Value of the yeast-vitamine fraction as a supplement to a rice diet.* Emmett and McKim. See XIXA.

#### PATENTS.

*Alcohol; Removing — from liquors.* R. H. Pflugfelder, Chicago, Ill. U.S. Pat. 1,249,480, Dec. 11, 1917. Date of appl., May 29, 1917.

A DEEP rectangular tank is provided with a series of downwardly-sloping brackets, one above another, fixed alternately to one or the other of two opposite walls and extending nearly across the tank. Each bracket supports a set of heating pipes on which rests a shallow tray, so that liquor delivered to the uppermost tray flows over each of the lower ones in turn, in a zig-zag path, to the bottom of the tank.—J. H. L.

*Lactic acid; Manufacture of —.* S. Saxe, New York. U.S. Pat. 1,249,511, Dec. 11, 1917. Date of appl., Apr. 20, 1917.

AN organism capable of producing lactic acid is grown in an extract of vegetable ivory, which may be prepared, for example, by hydrolysing the material with sulphuric or other acid and neutralising with calcium carbonate. Sufficient calcium carbonate may also be added to neutralise the lactic acid formed, the latter being afterwards liberated by addition of an equivalent quantity of sulphuric acid.—J. H. L.

*Manufacture of alcohol from molasses.* Eng. Pat. 112,232. See XVII.

*Process for the purification of crude alcohols.* Eng. Pat. 112,166. See XX.

#### XIXA.—FOODS.

*Milk; Determination of fat and casein in —. Ratio of casein to fat as a criterion of skimming.* F. Repiton. Ann. Chim. Anal., 1918, 23, 11—15.

To determine fat in milk, the author evaporates 10 c.c. of the milk with 5 to 10 grms. of sand, transfers the dry powder to a filter-tube, and extracts the fat by percolation with ether; the ethereal extract is then evaporated and the residue of fat dried and weighed. Another portion of the sample is then treated with dilute alcohol slightly acidified with acetic acid and the precipitate formed, consisting of the casein and fat, is collected, washed with very dilute alcohol, dried at 100° C. for 4 hours, and weighed. After deducting the quantity of fat found previously, the resulting weight is multiplied by 0.942 (allowance for ash) to obtain the weight of the casein. In the case of genuine milk the ratio of casein to fat is 0.82; when this ratio reaches or passes unity, skimming is indicated.—W. P. S.

*Fat in condensed milk and milk powders; Determination of —.* C. H. Biesterfeld and O. L. Evenson. J. Ind. Eng. Chem., 1917, 9, 1111—1114.

HARDING and Parkin (J. Ind. Eng. Chem., 1913, 5, 131) found that the Röse-Gottlieb method of extracting fat (Landw. Vers. Stat., 1892, 40, 1) did not remove the whole of the fat from evaporated milk, and obtained higher results by a method in which the protein is dissolved in acetic acid and the fat in a mixture of alcohol and carbon tetrachloride prior to extraction with petroleum spirit. The authors' experiments, however, show that these higher results must be attributed to the action of the solvents on the rubber stoppers, and that the average error in the Röse-Gottlieb process when applied to condensed milk does not exceed about 0.04%. The method may also give low results with milk powder or cream, but this error may be corrected by extracting the fat in the presence of acetic acid. From 4 to 4.5 grms. of evaporated milk, or 7 to 7.5 grms. of sweetened condensed milk, is diluted to 9 c.c., and treated first with 1.5 c.c. of strong ammonia solution and then with 15 c.c. of 95% alcohol, and the mixture vigorously shaken for 2 mins. with 50 c.c. of a mixture of 400 c.c. of petroleum spirit, 200 c.c. of ether, and 20 c.c. of 95% alcohol, in a large modified Röhrig tube with side tubulure. After standing for 10 mins. the ethereal layer is drawn off, filtered, and evaporated in a weighed flask. The residual liquid in the tube is treated with 3 c.c. of glacial acetic acid, and the tube immersed to the depth of the liquid in water which is heated from about 60° to 80° C. in about 10 mins. After cooling the tube in running water, 50 c.c. of a mixture of 350 c.c. of petroleum spirit, 280 c.c. of ether, and 63 c.c. of 95% alcohol is introduced, and the tube again shaken for 2 mins. and allowed to stand. The ethereal layer is drawn off and filtered into the same flask as before, and the extraction repeated with an additional 50 c.c. of the same mixture. The united residues of fat recovered from the extracts are dried at 100° C. until constant in weight.

—C. A. M.

*Mares' milk; Analysis of —.* A. Hildebrandt. Milchwirtsch. Zentr., 1917, 46, 273—278, 289—293, 305—310, 317—319.

THE milk from three mares was examined at intervals during the period April 4 to June 22, 1916; twenty-one analyses were made. The following are the minimum and maximum results obtained: Sp. gr., 1.0296—1.0390; total solids, 8.84—12.03; fat, 0.10—3.35; non-fatty solids, 8.03—9.84; ash, 0.32—0.74; nitrogen, 0.31—0.49; lactose, 4.32—7.56%. Generally, the milks did not give a peroxydase reaction, but in one or two cases a feeble reaction was obtained.—W. P. S.

*Pepsin; Rennetic properties of —.* H. T. Graber. J. Ind. Eng. Chem., 1917, 9, 1125—1126.

EXPERIMENTS have been made to ascertain the suitability of pepsin from the hog's stomach as a substitute for calf rennet in the manufacture of cheese. Sweet unpasteurised milk which was rapidly curdled by the rennet was only occasionally curdled by the dilute pepsin solution, but by increasing the acidity of the fresh milk to 0.185% (as lactic acid) by the addition of lactic acid, coagulation readily took place. This difference in behaviour points to the rennetic activity being due to a different substance in the two preparations. (See also J. Chem. Soc., Mar., 1918.)

—C. A. M.

*Bones; Preparation of edible fat and meat-extract substitute from —.* A. Kraus. Preuss. Verband beamt. Nahrungsmittel Chem., July, 1917. Chem.-Zeit., 1917, 41, 835—836.

FRESH bones when heated with water or steam

in an autoclave under 4—6 atmos. pressure yield a considerable quantity of fat which, after washing and clarification, is suitable for human consumption. The average yield of fat is about 15%, and bones from joints which have been boiled in the household will, generally, yield over 8% of fat. The aqueous extract from the bones contains much gelatinous substance and, with the addition of salt and spices, forms an edible product. Old or decomposed bones yield a fat which may be used for technical purposes, whilst the aqueous extract is useful as a manure.—W. P. S.

*Cottonseed meal injury; Iron as an antidote to*—W. A. Withers and F. E. Carruth. *J. Biol. Chem.*, 1917, 32, 245—257.

THE injurious effects of cottonseed meal may be lessened or prevented by the addition of iron salts to the feed. It is suggested that the iron salts combine with, or facilitate, the oxidation of the harmful substances in the meal.—W. P. S.

*Soya bean as food; Use of*—T. B. Osborne and L. B. Mendel. *J. Biol. Chem.*, 1917, 32, 369—387.

SOYA bean meal when cooked with water to make it palatable is capable of promoting the growth of young white rats and of maintaining the life of the adult animals. It contains, therefore, both the "water-soluble" and the "fat-soluble" accessory substances, but it is deficient in mineral salts. This latter deficiency may be rectified by the addition to the diet of an artificial mixture of salts.—H. W. B.

*Seed proteins; Values of some*—for maintenance. *Biological analysis of pellagra-producing diets.* III. E. V. McCollum and N. Simmonds. *J. Biol. Chem.*, 1917, 32, 347—368.

DATA are given which show the minimum amounts of protein which must be taken by rats in order to maintain health and body-weight when the protein is derived from the seeds of wheat, maize, oats, rice (polished), millet, flax, beans, or peas, or, for comparison, from lucerne leaves. Oats are distinctly more efficient for maintenance purposes than either wheat or maize, whilst rice and flax seed are less satisfactory. Millet seed approaches oats in dietetic value in this respect. The protein of lucerne leaves has no advantage regarding maintenance over that obtained from any of the above seeds. All these deductions are based on the supposition that all other accessory dietary factors are present or administered in adequate proportions.—H. W. B.

*Yeast-vitamine fraction; Value of the*—as a supplement to a rice diet. A. D. Emmett and L. H. McKim. *J. Biol. Chem.*, 1917, 32, 409—419.

THE authors describe the preparation of a vitamine extract which rapidly cures polyneuritis in pigeons. The vitamins in an autolysed yeast filtrate are adsorbed by Lloyd's reagent (a special form of fullers' earth) or by ordinary fullers' earth ground to a definite fineness. Infusorial earth or other commercial kieselguhrs do not adsorb the yeast vitamine. After polyneuritic pigeons have been cured by the vitamine preparation, further attacks of polyneuritis are prevented if doses of fullers' earth containing adsorbed vitamine are administered with the polished rice diet, but the pigeons do not attain their former weights and only a certain amount of growth is observed. If, however, brown rice (unpolished), barley, maize, or similar cereal is fed instead of the polished rice, the pigeons recover or surpass their former healthy condition, and recovery is greatly assisted if the

vitamine preparation is also included in the diet. It appears, therefore, that the outer layers of the rice grain contain two vitamins, one of which cures or prevents polyneuritis and the other produces growth, and of these two, the fullers' earth yeast vitamine preparation contains chiefly the curative fraction. Hence to ensure complete recovery from polyneuritis not only should the adsorbed vitamine be given, but the diet should include foods containing vitamins.—H. W. B.

*Orange and lemon peels and pips.* F. Mach and P. Lederle. *Chem.-Zeit.*, 1917, 41, 830.

THE composition of these substances was found to be as follows:—

	Water.	Crude pro- tein.	Crude fat.	Nitrogen- free extrac- tives.	Crude fibre.	Ash.
	%	%	%	%	%	%
Orange peel ..	19.30	4.66	1.92	62.67	8.12	3.33
Lemon peel ..	14.03	7.01	1.56	61.16	11.80	4.44
Lemon peel ..	15.32	6.56	2.17	56.66	14.00	5.29
Orange pips ..	6.82	13.72	33.37	31.22	11.30	3.57
Lemon pips....	8.23	18.25	34.30	22.21	14.35	2.66

—W. P. S.

*Methods for the production of pure coconut oil.* Parker and Brill. See XII.

*A precipitant for sugars, starches, gums, proteins, and organic acids.* Schulte. See XVII.

*Dried "spent" hops.* Baker and Hulton. See XVIII.

#### PATENTS.

*Nuts or seeds; Utilisation of certain*—in the manufacture of artificial butter or the like. De Bruyn, Ltd., and J. de Bruyn, London, Eng. Pat. 112,164, Dec. 27, 1916. (Appl. No. 18,500 of 1916.)

COCONUT or copra, the kernels of the babassu nut (*Attalea funifera*), or of cohune nuts or of other species of *Attalea*, and, in general, of any species of palm, except *Elæis guineensis*, or the meals or cakes made from these kernels, are used in the manufacture of artificial butter. The washed and cleaned material is finely divided, and agitated with a solution of common salt or of an alkaline salt or salts with similar solvent properties, at a suitable temperature (130°—140° F.), whereby solution and emulsification of part of the material are effected. The aqueous emulsion is separated from the undissolved residue and concentrated *in vacuo*, or it may be treated with acid, and the resulting precipitate redissolved and emulsified in alkaline solution. The original solution or emulsion or that made from the concentrated products is inoculated at a suitable temperature with an active culture of lactic acid bacteria, an addition of lactose or other sugars being made if desired, and the final ripened product is incorporated with a mixture of edible vegetable or animal oils or fats, and converted into artificial butter.—C. A. M.

*Flour; Process of bleaching and maturing*—J. N. Alsop, Owensboro, Ky. U.S. Pat. 1,250,072, Dec. 11, 1917. Date of appl., Mar. 15, 1915.

FLOUR is bleached and matured by the action of a mixture of chlorine and air, the air having been previously saturated with steam and subjected to a disruptive electric discharge.—W. F. F.



## XIXB.—WATER PURIFICATION; SANITATION

*Bacilli of Eberth and paratyphoid B bacilli in water; Search for* — F. Diénert, A. Guillard, and A. Leguen. *Comptes rend.*, 1918, 166, 84—85.

PARIS water is examined daily as follows:—Several litres are filtered through collodion, and the deposit is mixed with 50 c.c. of sterile physiological saline solution which is added to 50 c.c. of peptone water containing sterile bile and coloured with malachite green solution. The mixture is incubated at 37° C. for 1—2 days. A trace of this culture is inoculated successively into 5 tubes of melted agar containing lead acetate, and plates are made in Petri dishes. When the medium has set, a tube full of ordinary agar is poured over each, so as to make the cultures anaërobic. The plates are incubated at 37° C., and the colonies examined. Typhoid and paratyphoid B colonies are brown and surrounded with a pale halo; *B. pyocyaneus* gives similar colonies, while *B. coli* colonies are only slightly brown and without a halo. For confirmatory tests typical colonies are subcultured into peptone-bouillon for indole formation and agglutination; into neutral-red glucose agar; into lactose agar; into milk; and on to an agar slant for staining by Gram's method.—J. H. J.

*Activated sludge process; Experiments on sewage purification by the* — A. Cronin. *Austral. Pharm. Notes*, Oct. 1, 1917, 18—20.

THE first trial of this process in Australia was made at Sydney in 1916. The plant consisted of three wooden tanks of 500 galls. each, air being supplied through perforated pipes covered with broken stone. Two of the tanks were started by seeding with sludge from contact beds, but this method proved a failure. In the third tank raw sewage was treated in the usual way, three days' aeration being followed by 15 hours' settling. Analyses made after 2, 3, and 4 months' working gave practically constant results, the percentage purification being about 99 in suspended solids, 92 in organic nitrogen, and 84 in oxygen absorption. All the effluents were stable by the methylene blue test, and no smell was given off during treatment. A larger tank of 10,000 galls. was then constructed in concrete, and has been in continuous operation since November, 1916, on the fill and draw principle. The aeration period at starting was 4 days, then 3 days, then 20, 10, 7, 6, and 5 hrs. respectively. At the end of 8 weeks, the accumulated sludge occupied 20—25% of the capacity of the tank. The results were not so good after 5 hours' aeration, and after six months the period was fixed at 6 hours with 2 hours' settling, which gave as good results as a longer period. The percentage purification obtained was: suspended solids 96, organic nitrogen 87.7, and oxygen absorption 86.3. The effluent was odourless, of a faint "peaty colour," and was stable after the first 3 weeks' working. The air used was 4.8 cub. ft. per gall. with a ten hours' aeration period. The sludge contained about 66% of moisture and dried readily on sand. An analysis, calculated to a basis of 10% of moisture, showed that it contained 3.2% of total nitrogen, 2.7% of phosphoric acid, and 0.35% of potash.

—J. H. J.

*Copper in sewage at the New Haven sewage experiment station.* F. W. Mohlman. *J. Ind. Eng. Chem.*, 1917, 9, 1093—1096.

THE sewage after receiving the residual acid waste from a large munitions factory where the scale is removed from brass shells, after annealing, contained from 5.6 to 8.8 parts per million of

copper. The effect of this copper was to reduce the number of bacteria from 990,000 to 3000 per 20 c.c.—C. A. M.

## PATENTS.

*Water; Method of sterilising* — P. C. Rushen. London. From Quarzlampen Ges. m. b. H., Hanau, Germany. Eng. Pat. 112,149, Dec. 6, 1916. (Appl. No. 17,538 of 1916.)

WATER containing humic substances, etc., is sterilised by means of chemically active rays or light radiations (e.g., from a mercury quartz lamp), the action of which is assisted by adding to the water a very dilute solution of a photo-dynamically active substance, such as eosin.

—C. A. M.

*Distilling apparatus [for sea-water]*. J. Andrews. Kirkintilloch. Eng. Pat. 112,223, Apr. 27, 1917. (Appl. No. 5972 of 1917.)

A STEAM-HEATED still, a feed-water heater, a tubular combined water-heater and condenser, and a receiver, are arranged so that all or a portion of the distillate may be sent direct to the hot well, to a hot water tank for domestic or other purposes, or through a combined cooler and filter to a cold drinking water tank.—W. H. C.

*Sterilising animal and vegetable products; Process of* — H. Bart, Landau, Germany. U.S. Pat. 1,250,079, Dec. 11, 1917. Date of appl., June 29, 1917.

ANIMAL and vegetable substances are treated with nitrogen protoxide at pressures between 5 and 25 atmospheres and at a temperature below normal. The process is applicable to vaccines and similar preparations. The substance to be treated may be first rendered completely or partially germ-free, and then preserved by the process. The process may be carried out also above 25 atmospheres' pressure.—J. H. J.

*Water; Process of softening* — H. Heller, Cologne, Germany. U.S. Pat. 1,247,833, Nov. 27, 1917. Date of appl., Mar. 27, 1916.

SEE Eng. Pat. 103,698 of 1916; this J., 1917, 401.

*Permatites; Process for regenerating* — W. C. Foster, Rutherford, N.J., Assignor to Staten Island Chemical Corporation, New York. U.S. Pat. 1,249,368, Dec. 11, 1917. Date of appl., Aug. 19, 1915.

SEE Eng. Pat. 101,207 of 1916; this J., 1917, 977.

*Sterilising and preserving animal and vegetable products; Process for* — and for preparing inoculating substances or vaccines. H. Bart, Bad Dürkheim, Germany. Eng. Pat. 112,498, Jan. 9, 1917. (Appl. No. 404 of 1917.)

SEE U.S. Pat. 1,250,079 of 1917; preceding.

*Sterilising liquids contained in hermetically-closed vessels; Apparatus for* — L. Giommi, Milan, Italy. U.S. Pat. 1,249,740, Dec. 11, 1917. Date of appl., Apr. 9, 1913. Renewed Dec. 22, 1916.

SEE Eng. Pats. 9912 of 1912 and 30,048 of 1913; this J., 1913, 622; 1915, 197.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Asparagus sprengeri; Oil from [flowers of]* — F. Elze. *Chem.-Zeit.*, 1917, 41, 842.

THE flowers of *Asparagus sprengeri*, a variety of asparagus usually grown for decorative purposes, when extracted with a solvent yield an oil having

a strong aldehydic odour; this oil yields a semicarbazone. If the oil is obtained by subjecting the flowers to steam-distillation, the odour is less intense and the steam has evidently a decomposing action on the oil.—W. P. S.

*Cyanamide; Preparation of* —. A. E. Osterberg and E. C. Kendall. J. Biol. Chem., 1917, 32, 297—298. (See J. Chem. Soc., Mar., 1918.)

AN aqueous suspension of calcium cyanamide is decomposed by carbon dioxide at a temperature below 40° C. The calcium carbonate is filtered off and the cyanamide recovered from the filtrate by concentrating *in vacuo* and subsequently extracting with ether. The yield of pure crystallised cyanamide is about 92% of the theoretical, reckoned on the amount of calcium salt employed.

—H. W. B.

*Bark of the Hymenodactylon excelsum; Constituents of the* —. C. S. Gibson and J. L. Simonsen. J. Proc. Asiatic Soc. Bengal, 1916, 12, 161. (Reprint.)

THE extract of this bark when subcutaneously injected into two frogs gave no indication of any physiological action. The glucoside, aesculin, and its product of hydrolysis, scopoletin, were isolated from the bark.—W. G.

*Sero; Influence of phenol and of cresylic acid [cresol] on the concentration of antitoxin* — by the Banzhaf (1913) process. A. Homer. Biochem. J., 1917, 11, 277—282. (See also this J., 1917, 734.)

THE Banzhaf process may be improved by treating the original diluted plasma with 2% of sodium chloride and 0.3—0.35% of cresol before heating with ammonium sulphate. Between these limits of concentration cresol was found to give better results than phenol. The method of operating was as follows:—Oxalated plasma, diluted with one-fifth of its volume of water, was treated with 2% of sodium chloride and 0.3—0.35% of cresol, followed by sufficient saturated ammonium sulphate solution to bring the concentration of this salt to 30% of saturation. The liquid was heated gradually just to 60° C. and then cooled to 45° C. and filtered. The precipitate ("first fraction precipitate" consisting of protein practically free from anti-toxin) was washed with ammonium sulphate solution 30% saturated, and the mixed filtrate and washings treated with more ammonium sulphate to raise the concentration of this salt to 50% of saturation. The ensuing "second fraction precipitate," containing practically all the antitoxin, was filtered off, pressed, and dialysed as usual. Operating in this way on 50 litres of oxalated antitetanic serum, a final product was obtained occupying only 6.5 litres, containing 18.6% of protein (compared with 8.26% in the original plasma) and possessing a potency 8 times that of the original plasma, whilst Banzhaf's process only produces a 4- or 5-fold increase in potency and removes much less protein. The product was moreover clear and limpid. On dilution with saline it showed no trace of the presence of a colloidal suspension of euglobulin or of protein denatured by heat. It could be readily filtered through filter-candles and the filtered product did not become cloudy on standing. No undue loss of antitoxin occurs in this modified process provided the addition of 2% of salt is made and the mixture is neither heated beyond 60° C. nor allowed to remain at this temperature for more than 2—3 mins.—J. H. L.

*Nucleic acids; Preparation of plant* —. G. Clarke and S. B. Schryver. Biochem. J., 1917, 11, 319—324.

KNOWN methods of preparing nucleic acid from plant materials yield products containing protein,

which has afterwards to be removed by peptic digestion. To avoid this the authors devised a method in which the protein is first rendered unextractable, by heating with alcohol. The method was applied to yeast as follows:—Freshly pressed yeast was allowed to stand for 24 hours with a large excess of 95% alcohol, and afterwards dried in the air. The air-dried material was boiled for 2 hours with 95% alcohol, filtered, pressed, dried in a current of air at 37° C., and ground to a fine powder. One kilo. of the powder was extracted with 10 litres of 10% sodium chloride solution for 4—5 days, with frequent stirring and intermittent heating to 60°—80° C. The extract, filtered first through muslin and then through paper, was treated with 90 c.c. of hydrochloric acid (1:1) and vigorously stirred. After standing for 2 hours the liquid was siphoned off from the deposited cake of crude nucleic acid, and the latter was left overnight in 95% alcohol and then washed with absolute alcohol and ether. The yield was 1.4—1.6% of the dried yeast and the product did not give the biuret reaction. It was purified by dissolving 20 grms. in 500 c.c. of 10% sodium acetate solution in a water-bath, filtering, and adding 100 c.c. of alcohol and excess of hydrochloric acid. The precipitate, washed with 50% alcohol, 95% alcohol, and ether and dried *in vacuo*, formed a pure white powder. In applying this method of preparation to materials containing starch, it is best to saccharify the latter. For instance, wheat embryos were boiled with alcohol, dried in the air, heated with water to gelatinise the starch, saccharified by means of taka-diastrase, and then extracted with 10% salt solution.—J. H. L.

*Formaldehyde and hexamethylenetetramine; Iodine as a microchemical reagent for* —. C. van Zijp. Pharm. Weekblad, 1918, 55, 45—47.

IODINE gives with hexamethylenetetramine a compound of characteristic crystalline form which is not insoluble in alcohol as stated by Schmidt. The hexamethylenetetramine dissolved in a small drop of water is treated with a drop of iodine-potassium iodide solution (1:1:100), and protected from evaporation of the iodine (resulting in the disappearance of the crystals) by a cover-glass. 0.3 µgm. of hexamethylenetetramine may be detected in this manner; formaldehyde is detected after being converted into hexamethylenetetramine by means of ammonia solution.—F. W. A.

*Wool-fat (lanoline) substitute and the preparation of cetyl alcohol*. Axelrad. See XII.

*A precipitant for sugars, starches, gums, proteins, and organic acids*. Schulte. See XVII.

*Determination of alcohol*. Haines and Marden. See XVIII.

*Value of the yeast-vitamine fraction as a supplement to a rice diet*. Emmett and McKim. See XIXA.

## PATENTS.

*Smelling salts and solidified inhalants; Solidified* —. C. A. Cofman-Nicoresiti, London. Eng. Pat. 112,061, Jan. 29, 1917. (Appl. No. 1415 of 1917.)

ALCOHOL, liquid paraffin, or an essential or volatile oil is solidified by the addition of 5—10% of sodium stearate, and the mixture, just before it sets, is caused to absorb formaldehyde, ammonia, chlorine, or any other gas to produce a solidified inhalant or disinfectant.—J. F. B.

*Alcohols; Process for the purification of crude* —. W. J. Crawford, Llanelly. Eng. Pat. 112,166, Dec. 28, 1916. (Appl. No. 18,559 of 1916.)

CRUDE alcohols are treated with a concentrated



solution of sodium hypochlorite (sp. gr. 1.20), c.g., 1 part of crude wood naphtha with 2 of the hypochlorite or 2 parts of ethyl alcohol with 1 of hypochlorite, the quantity being such that no free chlorine is produced on distillation. The mixture is distilled and the chloro-compounds are separated from the distillate after dilution with water. If necessary, the treatment is repeated and the alcohol is finally purified by distillation with a dilute solution of alkali carbonate or calcium hydroxide.—J. F. B.

#### PATENTS.

*Antigene for tubercle bacilli; Preparation of* —. K. Momose, Osaka-Fu, Japan. Eng. Pat. 103,298, Jan. 6, 1917. (Appl. No. 283 of 1917.) Under Int. Conv., Jan. 7, 1916.

BOUILLON cultures of tubercle bacilli are washed with salt solution and water, pressed between sterilised filter-paper, and shaken and digested for about 48 hours in the dark, at 17° to 22° C., with 10% soda lye. They are then washed successively with salt solution and water, and the sediment repeatedly shaken, for about 2 hours each time, with chloroform to extract the waxy substance; the chloroform is separated by centrifuging. The residual stroma is dried at about 60° C., and made into an emulsion with salt solution and some carboic acid.—C. A. M.

*Acridine series; Manufacture of therapeutically valuable compounds of the* —. O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 112,809, Jan. 23, 1917. (Appl. No. 1166 of 1917.)

SEE U.S. Pats. 1,127,624 and 1,128,926 of 1917; this J., 1917, 867, 905. In addition to cadmium and silver salts, other heavy metal compounds, except zinc chloride, may be used in the preparation of the products.

*Alkaloidal compound.* J. U. Lloyd, Cincinnati, Ohio. U.S. Pat. 1,250,331, Dec. 18, 1917. Date of appl., July 6, 1915.

A COMPOUND consisting of an alkaloidal substance with an insoluble compound containing magnesium, e.g., hydrated magnesium silicate, is claimed.

—F. W. A.

*Desoxycholic acid; Process of producing addition compounds of* —. H. Wieland, Munich, Germany. U.S. Pat. 1,252,212, Jan. 1, 1918. Date of appl., May 16, 1917.

ADDITION compounds of desoxycholic acid with aromatic compounds are produced by dissolving one component in a hot solvent and adding the other component. The production of phenolcholeic acid (m. pt. about 165° C.), by the addition of desoxycholic acid to liquefied phenol and removal of the excess phenol by means of a solvent, is specially claimed.—F. W. A.

*Manufacture of lactic acid.* U.S. Pat. 1,249,511. See XVIII.

*Process of sterilising animal and vegetable products [e.g., vaccines].* U.S. Pat. 1,250,079. See XIXB.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photo-chemical processes; True* —. F. Weigert, Z. Elektrochem., 1917, 23, 357—368. (See also J. Chem. Soc., March, 1918.)

A THEORETICAL paper, in which after real and ideal photochemical processes have been differ-

entiated and a general survey has been made of a number of real processes, the author propounds a theory of the mechanism involved in the reactions. The author includes in the general discussion the processes operative in photo-electric actions, fluorescence, luminescence, and Röntgen-ray effects.—J. F. S.

## XXIII.—ANALYSIS.

*Collodion membranes; Preparation of uniform* — for dialysis. C. J. Farmer. J. Biol. Chem., 1917, 32, 447—453. (See also J. Chem. Soc., March, 1918.)

THE membrane is prepared by filling a glass tube with collodion solution (3 grms. of dried gun cotton to 50 c.c. of a mixture of equal parts of absolute alcohol and ether), inverting, and allowing to drain for one minute. The tube is then dried for one minute in a current of air and afterwards filled with cold water. After a few minutes, the thin membrane can be removed from the walls of the glass tube with the aid of a pair of forceps. The permeability of the membrane may be changed by varying the period of drying. Convenient apparatus for performing these operations is described in detail.—H. W. B.

*Fluorine; Method for the determination of* — with special application to the analysis of phosphates. C. R. Wagner and W. H. Ross. J. Ind. Eng. Chem., 1917, 9, 1116—1123.

A WEIGHED quantity of the dried material containing from 0.001 to 0.1 grm. of fluorine is mixed with 0.1 to 1 grm. of silica and 5 grms. of anhydrous copper sulphate in a 250 c.c. flask, one outlet of which is connected with a cylinder of compressed carbon dioxide or nitrogen fitted with a reducing valve, whilst the other outlet is connected with two traps containing strong sulphuric acid. Following these are a Schmitz tube containing a 10% solution of silver sulphate in 98.5% sulphuric acid, a Bowen tube containing a saturated solution of chromium trioxide in 98.5% sulphuric acid, a straight piece of tubing packed with glass wool, and, finally, a large test-tube containing about 50 c.c. of water. After the introduction of 100 c.c. of 98.5% sulphuric acid into the generating flask, a slow current of the inert gas, which passes through two wash-bottles containing sulphuric acid before entering the flask, is passed through the apparatus at the rate of 2 to 3 bubbles per minute, and this is continued throughout the determination. The flask is then gently heated, the final traces of silicon fluoride being expelled by means of the current of gas. Sulphur dioxide and trioxide and any hydrochloric or nitric acid are retained by the reagents in the Schmitz and Bowen tubes, so that the solution of hydrofluosilicic acid in the final tube is quite free from other acid substances. It is made up to 200—250 c.c., gently boiled to expel dissolved gases, cooled slightly, and titrated with *N*/10 sodium hydroxide solution, with phenolphthalein as indicator. Organic matter is removed by a preliminary ignition of the sample. The method is specially applicable to the determination of fluorine in phosphates, and was devised in connection with the so-called volatilisation method of recovering phosphoric acid from mineral phosphates (see this J., 1916, 1154; 1917, 134).—C. A. M.

*Sulphuric acid and barium; Gravimetric determination of* — as barium sulphate. Z. Karaglanow. Z. anal. Chem., 1917, 56, 417—439. (See also J. Chem. Soc., 1918, ii., 47.)

IN dilute solution, the precipitation of sulphuric

acid as barium sulphate depends on the concentration of the acid and on the quantity of barium chloride added; the quantity of barium sulphate formed decreases with an increase of barium chloride. This decrease is not due to solubility since barium sulphate is practically insoluble in the presence of barium chloride. When the precipitation is made from a solution containing a relatively large amount of hydrochloric acid or nitric acid, the results are too high. The presence of potassium chloride makes the results low, whilst with potassium nitrate they are too high, the extent of the influence of these two salts depending on the concentration of the solution. Potassium sulphate also causes low results but when hydrochloric acid is present in addition the results are too high. In the case of a solution containing both potassium chloride and potassium sulphate, the quantity of barium sulphate recovered is too low whether or not hydrochloric acid is present. If, however, the solution is very dilute the conversion of potassium sulphate into barium sulphate is quantitative even in the presence of nitric acid. The presence of sodium, ammonium, zinc, and magnesium chlorides does not affect the results; ferric chloride causes the figures for barium sulphate to be very low (about 3%), but in very dilute solutions containing hydrochloric acid the influence of iron salts is very small. Aluminium chloride in the presence of hydrochloric acid does not interfere, but chromium chloride causes the results for barium sulphate to be much too low.

—W. P. S.

*Strontium chloride and strontium bromide; Transition temperatures of — as fixed points in thermometry.* T. W. Richards and V. Yngve. J. Amer. Chem. Soc., 1918, 40, 89—95.

THE temperature at which the hexahydrate of strontium chloride is converted into the dihydrate is 61.341° on the international hydrogen scale. The corresponding change in strontium bromide takes place at about 88.62°. These temperatures may be used as fixed points for thermometric purposes. It has been found that the recrystallisation of strontium chloride in the form of the hexahydrate rapidly removes any barium present. Calcium is not nearly so rapidly eliminated, but if the salt is recrystallised to give the dihydrate, the calcium is quickly removed. By combining the two processes, strontium may be freed from both calcium and barium. The separation of small quantities of calcium from strontium may also be effected by crystallising the nitrates at a sufficiently high temperature to produce the anhydrous salt. (See also J. Chem. Soc., March, 1918.)

—H. M. D.

*Sodium and potassium; Estimation of —.* F. H. McCrudden and C. S. Sargent. J. Biol. Chem., 1918, 33, 235—241.

SODIUM and potassium are separated from other compounds as the combined chlorides and the amount of each is calculated from the chlorine content of a known weight of the mixture. The authors show that when the McLean-Van Slyke method (this J., 1915, 608) is employed for estimating the chlorine, the probable error in the estimation of the sodium increases as the ratio of sodium to potassium decreases and amounts usually to about 1%. (See also J. Chem. Soc. March, 1918.)—H. W. B.

*Phosphoric acid; Estimation of — as magnesium pyrophosphate.* D. Balareff. Z. anorg. Chem., 1917, 101, 229—234.

THE different methods of precipitating magnesium ammonium phosphate, namely, the older cold precipitation method of Neubauer and the hot methods of Schmitz, Jorgensen, and Järvinen have been compared, and it is found that, provided

precipitation takes place from pure aqueous solution, the results obtained are equally satisfactory, whichever method be used. In the presence of alkali cations, K<sup>+</sup> or Na<sup>+</sup>, however, high results are obtained, the effect being smaller at higher temperatures. If the concentration of alkali is great, even a second precipitation does not completely purify the precipitate. Sulphate ions have the same effect as alkali ions. Slow precipitation leads to low results, and the author concludes that rapid precipitation at a high temperature is best for accurate work. It appears probable from the author's experiments that the high results obtained when precipitation takes place in presence of alkali salts or of sulphates are not due to adsorption but to the formation of mixed alkali-magnesium phosphates.—E. H. R.

*Absorption of hydrogen by sodium oleate. Reagents for use in gas analysis.* VI. Anderson and Katz. See IIa.

*Determination of benzene vapour. Reagents for use in gas analysis.* VII. Anderson. See IIa.

*Effects of certain solvents on the determination of "free carbon" in tars.* Monroe and Brodersen. See III.

*Estimation of phenol in the presence of the three cresols.* Knight and others. See III.

*A constant temperature and humidity room for testing of paper, textiles, etc.* Veitch and Reed. See V.

*Method for determining the absorbency of paper.* Reed. See V.

*Detection and determination of small quantities of free hydrochloric acid in presence of chlorides and other mineral or organic acids.* Entat. See VII.

*Volumetric determination of formic acid or formates in presence of hydroxides, carbonates, oxalates, and acetates.* Tsirupinas. See VII.

*Comparative tests of chemical glassware.* Walker and Smither. See VIII.

*Determination of manganese in steel in the presence of chromium and vanadium by electrometric titration.* Kelley and others. See X.

*Colorimetric determination of bismuth [in copper].* Motherwell. See X.

*Detection of foreign oils in castor oil used for lubricating aeroplane motors.* Frabot. See XII.

*Reaction of Hawaiian soils with calcium bicarbonate solution, its relation to the determination of time requirements of soils and a rapid approximate method for the determination of time requirements of soils.* Johnson. See XVI.

*Determination of the acidity of soils and its value in determining the quantity of lime to be applied in practice.* Grantham. See XVI.

*Bonedust: its adulteration with phosphate rock.* Chapman. See XVI.

*Determination of nitrogen in calcium cyanamide.* Turkus. See XVI.

*A precipitant for sugars, starches, gums, proteins, and organic acids.* Schulte. See XVII.

*A quick method for lime-cake analysis.* Clark. See XVII.



*Determination of alcohol.* Haines and Marden. See XVIII.

*Determination of fat and casein in milk. Ratio of casein to fat as a criterion of skimming.* Repiton. See XIXA.

*Determination of fat in condensed milk and milk powders.* Biesterfeld and Evenson. See XIXA.

*Search for bacilli of Eberth and paratyphoid B bacilli in water.* Diénert and others. See XIXB.

*Iodine as a microchemical reagent for formaldehyde and hexamethylenetetramine.* Van Zijp. See XX.

## Patent List

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Barnes, and United Alkali Co. Apparatus for raising liquids. 2033. Feb. 4.

Björnstad, and Pulsometer Engineering Co. Filters. 2524. Feb. 12.

British and Foreign Supply Association, and Mitchell. Hydro-extractors and centrifugal separators. 2565. Feb. 13.

Brownlow. Centrifugal separators. 2564. Feb. 13.

Carlson. Furnaces. 2247. Feb. 7.

Croll. 2477. See XXII.

Fraser, and Fraser and Co. Machines for mixing granulated or triturated materials. 2246. Feb. 7.

Hayashi and others. 2245. See II.

Huhn. Drying granular materials. 2242. Feb. 7.

Hutchins. Apparatus for facilitating obtaining crystals from solutions. 2171. Feb. 6.

Leitch and Warburton. Apparatus for heating or cooling liquid or semi-liquid materials on a revolving cylinder. 2677. Feb. 14.

Perry. Apparatus for distilling. 2662. Feb. 14.

Sturgeon. Centrifugal machines or separators. 2438. Feb. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,282 (1916). Wallace and Tiernan. Treatment of liquids with gases. (113,106.) Feb. 20.

18,632 (1916). Wade (Buhl). Process and apparatus for desiccation. (112,965.) Feb. 13.

18,671 (1916). Adam. Fractional distillation. (112,966.) Feb. 13.

1156 (1917). Barbet et Fils et Cie. Apparatus for producing and maintaining a variable determined level of liquid in evaporating plant. (105,543.) Feb. 13.

2506 (1917). Tedesco. See II.

4813 (1917). Ostrejko. See II.

11,541 (1917). Wooster. See XVII.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Bramson. 2258. See V.

Chown. Carbonising. 2100, 2101. Feb. 5.

Clark. Water-gas manufacture. 2259. Feb. 7.

Crowe. Gas manufacture. 2489. Feb. 12.

Deakin (Roth and Venturino). Transforming heavy petroleum products into lighter products. 2521. Feb. 12.

Freeman. Carbonising or distilling apparatus. 2462. Feb. 12.

Furniss. Oil fuel. 2688. Feb. 15.

Hannay. Apparatus for obtaining mixtures of constant composition by evaporating volatile combustibles. 2624. Feb. 14.

Hayashi, and Kwanto Sanso Kabushiki Kaisha. Manufacture of decolorising-carbon. 2546. Feb. 12.

Lord. Manufacture of fuel oil from coal-tar products. 2655. Feb. 14.

Marks (Soc. Anon. Italiano G. Ansaldo & Co.). Gas-generating apparatus. 2736. Feb. 15.

Meredith and Pearson. Manufactured fuel. 2059. Feb. 5.

Nomi. Utilisation of petroleum pitch. 2432. Feb. 11. (Japan, Apr. 4, 1917.)

Robertson. Retorts for distillation of coal, shale, etc. 2776. Feb. 16.

Somerville, and South Metropolitan Gas Co. Manufacture of a material for removing carbon monoxide from gases. 2239. Feb. 7.

Southcombe. Lubricating-oils. 2055. Feb. 5.

Stewart. Gas-producers, and method of working them. 2132. Feb. 6.

Westwood. Treatment of heavy fuel oils. 2372. Feb. 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

629 (1917). Campbell and Goodyear. Coking-ovens, and methods of working them. (112,967.) Feb. 20.

1864 (1917). Nelson. Refining hydrocarbon oils and spirits. (113,131.) Feb. 20.

1865 (1917). Heyl. Distillation of oil shales. (112,997.) Feb. 13.

2506 (1917). Tedesco. Apparatus for purifying or refining liquids, particularly mineral spirits. (111,277.) Feb. 20.

2630 (1917.) Heyl. Distillation of coal. (113,012.) Feb. 13.

3964 (1917). Wollaston. Gas-producers. (113,025.) Feb. 13.

4813 (1917). Ostrejko. Production of decolorising-charcoal. (106,089.) Feb. 13.

4982 (1917). Stewart. Gas-producers. (113,182.) Feb. 20.

15,728 (1917). Blythe. Destructive distillation of bituminous coal. (113,079.) Feb. 13.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Blakeley. Stills or preheaters for dehydration of tar, etc. 2646. Feb. 14.

Clayton and others. See XXII.

Jack. Method of handling solid pitch, etc. 2554. Feb. 13.

Lord. 2655. See II.

Morris and Turnbull. 2039. See VII.

Robertson and Steele. Method of conducting alkaline fusions. 2366. Feb. 9.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Hart. Production of dyes, etc. 2562. Feb. 14.

Layland. Dry stains for wood. 2560. Feb. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

1950 (1917). Inray (Soc. Chem. Industry in Basle). Manufacture of copper compounds of substantive azo dyestuffs. (113,141.) Feb. 20.

4939 (1917). Ehrhardt and Ehrhardt. Manufacture of a colouring matter. (113,181.) Feb. 20.

6117 (1917). Ehrhardt and Ehrhardt. Manufacture of a mixed disazo colouring matter. (113,195.) Feb. 20.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Bramson. Utilising dead leaves as stock for making paper pulp and fuel. 2258. Feb. 7.

Cellon, Ltd., Tyrer, and Tyrer and Co. Production of dopes. 2754. Feb. 15.

Fort, Lumsden, MacKenzie, and Robinson. Treatment of scrap aeroplane fabrics, etc., and recovery and utilisation of products. 2365. Feb. 9.

Maxwell. 2158. *See* XIX.

St. Armande. Treatment of aeroplane fabrics. 2141. Feb. 6.

Smethurst. Conditioning yarn, etc. 1997. Feb. 4.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1564 (1917). Marks (International Cellulose Co.). Process of dissolving cellulose. (104,173.) Feb. 13.

2438 (1917). Elsaesser. Spinning copper oxide ammonia cellulose solutions. (113,010.) Feb. 13.

7869 (1917). Herbein. Treatment of wood and other cellulose-containing materials. (113,209.) Feb. 20.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATION.

Dudley. Dyeing machine. 2053. Feb. 5. (U.S., Feb. 17, 1917.)

##### COMPLETE SPECIFICATION ACCEPTED.

1323 (1917). Taylor. Means for circulating bleaching and other liquors in bleaching and like treatment of cotton, wool, etc. (112,909.) Feb. 13.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

Carlson. Manufacture of nitrogen compounds from carbides. 2386. Feb. 9.

Croll. 2477. *See* XXII.

Goldschmidt. Production of aluminium compounds. 2019. Feb. 4. (Norway, Feb. 2, 1917.)

Jenkins and Woolner. Manufacture of permanganates. 2744. Feb. 15.

Morris and Turnbull. Chlorination of water, alkaline, or other solutions, or organic liquids. 2039. Feb. 5.

Norsk Aktieselskab for Elcktrokemisk Industri. Production of aluminium compounds poor in iron. 2078. Feb. 5. (Norway, Feb. 6, 1917.)

Norton Co. 2253 and 2255. *See* VIII.

Pinkus and Somers. Stable compound of sodium permanganate. 2801. Feb. 16.

Pinkus and Somers. Manufacture of potassium permanganate. 2802. Feb. 16.

Pope. Manufacture of sulphuryl chloride. 2611. Feb. 13.

Rollason. 2475. *See* VIII.

Thompson. Manufacture of nitrides for use as fertilisers or for production of ammonia. 2469. Feb. 12.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1440 (1917). Imperial Trust for Encouragement of Scientific and Industrial Research, Glazebrook, Rosenhain, and Rodd. Manufacture of a new compound of zirconium, and its use in the production of pure zirconia. (112,973.) Feb. 13.

2003 (1917). Boehringer. Manufacture of borosilicates. (104,008.) Feb. 20.

3054 (1917). Jackson (Bombrini Parodi-Delfino). Apparatus for the catalytic preparation of sulphuric anhydride. (113,017.) Feb. 13.

3324 (1917). Dreyfus. Manufacture of acetic acid. (105,065.) Feb. 20.

8010 (1917). Ashcroft. Manufacture of potassium chloride from natural silicates, rocks, minerals, residues, or wastes containing potash. (113,211.) Feb. 20.

10,554 (1917). Standard Oil Co. Production of aluminium chloride. (109,790.) Feb. 20.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Dupont. Process and furnace for enamelling metal articles, etc. 2667. Feb. 14. (Fr., Mar. 30, 1917.)

Edwards, Edwards, and Mollart. Glaze for pottery, etc. 2579. Feb. 13.

Marlow. Ovens or kilns for manufacture of tiles, pottery, etc. 2773. Feb. 16.

Norton Co. Aluminous abrasives. 2252, 2251, 2256, 2257. Feb. 7. (U.S., Feb. 12 and Aug. 2, 1917.)

Norton Co. Composition containing alumina and zirconia. 2253. Feb. 7. (U.S., Feb. 12, 1917.)

Norton Co. Products containing  $\beta$ -alumina and process for preparing same. 2255. Feb. 7. (U.S., Mar. 2, 1917.)

Peeters. Glass furnaces. 2439, 2440. Feb. 11.

Rollason. Manufacture of hard, dead-shrunk, inert dolomite, and magnesite, etc. 2475. Feb. 12.

##### COMPLETE SPECIFICATION ACCEPTED.

1440 (1917). Imperial Trust and others. *See* VII.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Adam and Braid. Coating iron and steel, etc., with lead. 2450. Feb. 12.

Bosworth, and Elkington and Co. Muffles, or metal-heating furnaces. 2135. Feb. 6.

Bowen, and Steel, Peach, and Tozer. Steel-melting, etc. furnaces. 2273. Feb. 8.

British Carbonizing Co., and Fennell. Carbonisation of iron and steel articles. 2007. Feb. 4.

Coley and Wilbraham. Manufacture of metallic alloys. 2092. Feb. 5.

Donald. Soldering aluminium or its alloys. 2747. Feb. 15.

Dupont. 2667. *See* VIII.

Glässer. Tinning sheet metal. 2534. Feb. 12.

Hacking. Apparatus for extracting zinc from zinc ores. 1971. Feb. 4.

Kievits, and Kynoch, Ltd. Three-phase salt-bath electric hardening furnace. 2762. Feb. 16.

Marks (Soc. Anon. Italiano G. Ansaldo & Co.). Heating-furnaces. 2799. Feb. 16.

Naish. Aluminium alloys. 2186. Feb. 6.

Slingsby. Manufacture of malleable cast iron. 2466. Feb. 12.

Stock. Manufacture of wrought iron. 2396. Feb. 11.

Thermalloy, Ltd., and Vautin. Ignitable metallic mixtures. 2587. Feb. 13.

Wade (Verein. Hüttenwerke Burbach-Eich-Düdelingen A.-G.). Determination and regulation of blast in blast furnaces. 2532. Feb. 12.

White. Recovery of brass from foundry ash, etc. 2653. Feb. 14.

White. Recovery of brass and zinc from foundry ash, etc. 2654. Feb. 14.

##### COMPLETE SPECIFICATIONS ACCEPTED.

1393 and 1394 (1917). Martin. Ore concentration. (103,990 and 103,991.) Feb. 13.

1655 (1917). Schaffer. Heat treatment of manganese steel articles. (112,986.) Feb. 13.

5376 (1917). Whitham. Crucible furnaces. (113,036.) Feb. 13.

5463 (1917). Tissier. Calcining and roasting certain ores. (105,915.) Feb. 20.



## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Electric furnaces. 2368. Feb. 9.  
Féry. Electric cells. 2384. Feb. 9.  
Kievits, and Kynoch, Ltd. 2762. *See* X.

## COMPLETE SPECIFICATIONS ACCEPTED.

15,366 (1916). Leitner and Exley. Electric accumulators. (112,956.) Feb. 13.  
6046 (1917). Major and Pybus. Composite electric insulating substances. (113,194.) Feb. 20.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Barry, and Barry, Ostlere, and Shepherd. Treatment of linseed-oil fatty acids. 2057. Feb. 5.  
Boehm, Ltd., and Reihl. 2618. *See* XIII.  
Brizell, Martin, and C.W.S., Ltd. Process for deodorising cocoa butter. 2388. Feb. 9.  
Carmichael and Dixon. Apparatus for extracting grease, resins, fats, etc., from seeds, sewage sludge, etc. 2360. Feb. 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

11,541 (1917). Wooster. *See* XVII.  
12,512 (1917). Kimura. Apparatus for manufacturing hard fats. (113,232.) Feb. 20.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATIONS.

Barry and others. 2057. *See* XII.  
Boehm, Ltd., and Reihl. Process for turning linseed oil, etc., fatty acids into boiled oils. 2618. Feb. 13.  
Carmichael and Dixon. 2360. *See* XII.  
Cellon, Ltd., and others. 2754. *See* V.  
Cleghorn. Materials for coating substances to protect them or render them non-porous. 2105. Feb. 5.  
Corticine Floor Covering Co., and Thompson. Manufacture of cement for use in making linoleum, etc. 2009, 2021. Feb. 4.  
Dior. Manufacture of synthetic resins, etc. 2272. Feb. 8.  
McIndoe. Paints, etc., and solidifying compositions. 2093. Feb. 5.  
Singer. Manufacture of lacquers, varnishes, etc. 2598. Feb. 13.  
Suzukawa. Paint for coating bottoms of vessels. 2377. Feb. 9. (Japan, Feb. 22, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

5250 (1917). Tamari. Acid-proof coating compositions. (107,370.) Feb. 20.  
10,775 (1917). Wright. Paint compositions. (109,255.) Feb. 13.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

Bolton, and Haley and Co. Machines for treating hides, skins, and leather. 1990. Feb. 4.  
Tiltson. Apparatus for treatment of hides, etc. 2054. Feb. 5.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

Basset. Manufacture of superphosphates of lime. 2425. Feb. 11. (Fr., Feb. 9, 1917.)  
Conder. Apparatus for excavating superphosphates, etc. 2083. Feb. 5.  
England. Fertiliser. 2167. Feb. 6.  
Gaillard. Process of enriching material for the manufacture of fertilisers. 2130. Feb. 6.  
Thompson. 2469. *See* VII.

## XVII.—SUGARS; STARCHES; GUMS.

## COMPLETE SPECIFICATION ACCEPTED.

11,541 (1917). Wooster. Process for cleaning filters in connection with the purification of sugar, oils, and chemicals. (109,795.) Feb. 20.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Bellwood, Bolton, Downs, and Revis. Tanks or containers for ripening or souring milk or cream for manufacture of butter, margarine, etc. 2123. Feb. 6.  
Brizell and others. 2388. *See* XII.  
Carmichael and Dixon. 2360. *See* XII.  
Henderson. Method of making butter. 2143. Feb. 6.  
Hewett. Drying fruit, vegetables, etc. 2076. Feb. 5.  
Hewett. Drying-apparatus for potatoes, fruit, seeds, etc. 2805. Feb. 16.  
Maxwell. Dehydrating vegetable, textile, and like substances. 2178. Feb. 6.  
Straus. Non-alcoholic beverage, and process of making same. 2316. Feb. 8. (U.S., Feb. 23, 1917.)

## COMPLETE SPECIFICATION ACCEPTED.

16,281 (1916). Wallace and Tiernan. Apparatus and processes relating to purifying or treating water or sewage or other liquids. (113,105.) Feb. 20.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Boake, Roberts and Co., and Durrans. Manufacture of organic acid anhydrides and chlorides. 2733. Feb. 15.  
Commercial Research Co., Making halogenated products. 2001. Feb. 4. (U.S., Feb. 8, 1917.)  
Commercial Research Co. Processes of making chlorhydrins. 2002. Feb. 4. (U.S., Feb. 8, 1917.)  
Morris and Turnbull. 2039. *See* VII.

## COMPLETE SPECIFICATIONS ACCEPTED.

1441 (1917). Imray (Soc. Chem. Industry in Basle). Manufacture of therapeutically valuable compounds of the quinine group. (112,974.) Feb. 13.  
1650 (1917). Mouneyrat. Preparation of aqueous solutions of arsenobenzene derivatives. (112,984.) Feb. 13.  
1905 (1917). Dehn (Chemical Works Rohner and Co.). Manufacture of a difficultly soluble complex compound from dimethylaniline, benzyl chloride, and zinc chloride. (104,676.) Feb. 13.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## COMPLETE SPECIFICATION ACCEPTED.

5732 (1917). De Brayer. Preparation of chemical substances for use in photography. (105,920.) Feb. 20.

## XXII.—EXPLOSIVES; MATCHES.

## APPLICATIONS.

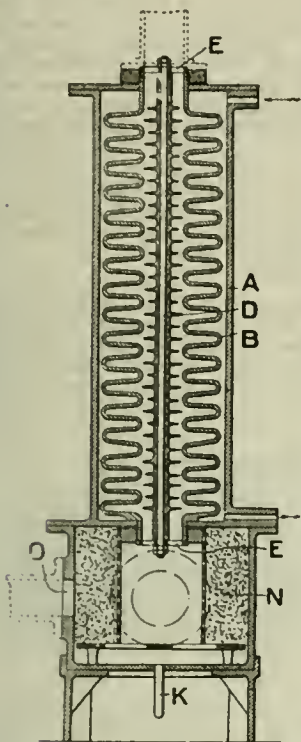
Clayton, Holliday, and Holliday and Co. Production of picric acid. 2200. Feb. 7.  
Croll. Containers or liners for explosives, acids, etc. 2477. Feb. 12.

## COMPLETE SPECIFICATION ACCEPTED.

1964 (1917). Commercial Research Co. Production of explosive compositions. (106,086.) Feb. 20.

**I.—GENERAL; PLANT; MACHINERY.****PATENTS.**

*Oil extracting, cooling and like apparatus for use with compressed air.* H. Smethurst, Hollinwood. Eng. Pat. 112,362, Apr. 23, 1917. (Appl. No. 5664 of 1917.)



The compressed air enters through the opening, O, passes through the wood-wool filter, N, and after being partly purified it passes up the corrugated tube, B, contained in the casing, A, and escapes at E. Cooling water is passed through the casing, A, and the compressed air, directed into the corrugations of the tube by the flanges on the central rod, D, is cooled and deposits the oil, which flows down into the bottom chamber from which it is from time to time discharged through the pipe, K.

—W. H. C.

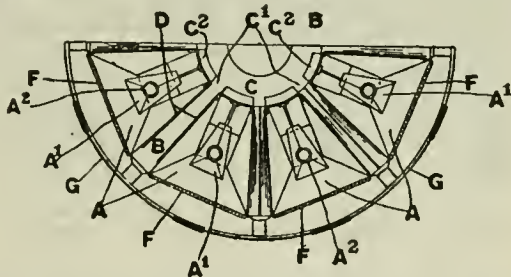
*Separation and recovery of liquids of different densities, and the separation of light solid particles from a heavy liquid; Process and means for the —.* E. Sepulchre, Woluwe-St. Pierre, Belgium. Eng. Pat. 103,670, Jan. 24, 1917. (Appl. No. 1192 of 1917.) Under Int. Conv., Oct. 20, 1915.

THE two immiscible liquids to be separated, e.g., petroleum and water, are contained in the tank, 1, shown in plan, and the heavier portion is withdrawn through the sluice, 3, to the discharge channel, 4, 5. The lighter mixture passes into the channel, 6, which diverges horizontally and

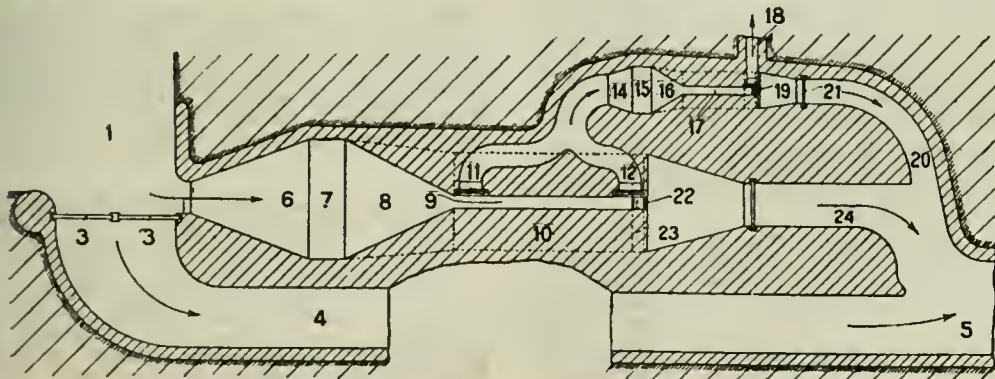
has its floor sloping upwards towards the flat-topped weir, 7, where the liquids separate by gravity. The lighter liquid flows on through a channel, 8, of decreasing section, and the heavier liquid through the lower part of the channel which deepens and widens to a large channel, 10. The lighter liquid is drawn off from the narrow upper part, 9, of the channel through the passages, 11, 12, to a second similar separating device, 14, 15, 16, 17. The lighter portion is finally withdrawn through the channel, 18, and the heavier portion passes under sluices, 19 and 21, to the channels, 20 and 5. The heavier liquid from the channel, 10, passes under a vertical plate, 22, having a lip, 23, at the bottom, curved backwards, so that a flat sheet of the liquid is formed and the lighter portions are separated and diverted upwards. In a modification, the mixture is delivered into a vertical column, having a flat horizontal outlet at the bottom, into a horizontal chamber having a triangular section which gradually increases in size. The lighter liquid is separated in the upper narrow part of the chamber, and rises into a vertical discharge column. The heavier liquid passes along the floor of the chamber, and over a lip projecting upwards just below the column for the lighter liquid, so that it is spread out into a shallow stream and any lighter particles separated upwards. The heavier liquid then passes on to another discharge column of less height than that for the lighter liquid.—W. F. F.

*Cooling and drying compressed materials and substances, seeds and the like; Apparatus for — and for removing dust therefrom.* W. Marshall, Kingston-upon-Hull. Eng. Pat. 112,512, Jan. 17, 1917. (Appl. No. 827 of 1917.)

In an apparatus for cooling, drying, and eliminating dust from compressed blocks or pieces of material such as cattle food, a hopper provided



with a delivery spout projecting from the side is mounted centrally above the apparatus shown in the figure in sectional half plan, and rotated slowly by bevel gearing so as to deliver the material into a number of sector-shaped cooling and





drying chambers, A. Each chamber is provided with a number of superposed spreaders, each comprising a hollow cone, A<sup>1</sup>, with a short central tube, A<sup>2</sup>, projecting upwards nearly to the base of the next cone. Each drying chamber is provided with perforated grids or plates, F and D, the latter forming the walls of radial air passages, B, leading from a central air chamber, C. The inner walls of the chamber, A, are provided with cooling ribs, C<sup>2</sup>, projecting into the chamber, C. Air is supplied from a pump through a central supporting standard to the chamber, C, and passes thence through the passages, B, which are covered at the top, to the chambers, A. The air passes into the annular space enclosed by the outer casing, G, where dust is allowed to settle, and finally escapes through a dust-collecting fabric at the top. The dried material is delivered through hoppers at the base of the chambers, A. In a modification, the cooling chambers are arranged side by side in line, the air being delivered to a common air space at the back and passing into the cooling chambers through the perforated walls.—W. F. F.

*Cooling, humidifying and filtering air or the like; Apparatus for* —. H. F. Brown, Sheffield. Eng. Pat. 112,653, Jan. 16, 1917. (Appl. No. 769 of 1917.)

In apparatus of the type in which the air to be treated is passed between wet plates, the plates are arranged radially and vertically in a cylindrical casing. Water is supplied upwards through a central rotating pipe carrying horizontal perforated branches which move over the top of the plates. The horizontal branches are shaped similarly to the horizontal section of the plates, which are preferably curved to an involute form. The perforations are on the lower side of the pipes, which carry horizontal flanges to overlap several of the wet plates, the flange on the following side being wider than that on the leading side. Air is thus prevented from passing upwards over those plates to which water has just been supplied and where there is thus an excess of water. The water drips off the plates on to a conical plate having a filter arranged around its periphery, and thence into the bottom of the casing from which it is supplied again to the sprinkler by a pump. A constant level of water is maintained in the casing by means of a float valve.—W. F. F.

*Furnaces; Oil-fired* —. R. Halkett, Glasgow. Eng. Pat. 112,700, Mar. 24, 1917. (Appl. No. 4278 of 1917.)

In an air control device for an oil-fired boiler furnace, a cylindrical casing is fixed to the front of the furnace and provided with openings in its periphery controlled by hinged louvres. Tangential guide-vanes are attached to the inner cylindrical wall of the casing to give the air a rotary motion, and the air passes inwards through two similar concentric sets of guide-vanes to the centre of the cylinder, where it meets the oil spray from a central spray burner. The burner is provided with tangential passages to give the spray a whirling motion, and the delivery is regulated by a needle-valve.—W. F. F.

*Baking-furnace [for carbons, etc.]*. R. K. Wehner, Assignor to Isbell-Porter Co., Newark, N.J. U.S. Pat. 1,251,309, Dec. 25, 1917. Date of appl., Aug. 26, 1916.

In a gas-fired furnace of the ring type for baking carbons and the like, a battery of ovens is arranged in two parallel groups. The heating flues in the side walls of the ovens do not communicate with the interior of the ovens. Gas-supply mains extend along the outer sides of the ovens and are connected to a gas burner in each flue, and an air-supply main and exhaust main are arranged

between the groups of ovens and connected to each flue. The flues are arranged with alternate up and down passages connected in series, and passages are provided under the floors of the ovens to connect the down flues of one side wall with the up flues of the next wall. In operation, gas under pressure is admitted to the oven being fired, and air under pressure to the series of connected flues of other ovens previously fired. The combustion products are exhausted through the flues of all the ovens to be fired, and the pre-heated air is supplied by the same means to the oven being fired. The flow of gas, air, and combustion products is thus kept uniform, and a minimum pressure difference is maintained between the flue pressure and the atmosphere so as to minimise leakage of cold air into the heating flues.—W. F. F.

*Oxidising gases; Process and apparatus for* —. I. Hechenbleikner, Charlotte, N.C., Assignor to Southern Electro Chemical Co., New York. U.S. Pat. 1,249,392, Dec. 11, 1917. Date of appl., Feb. 5, 1914.

A CURRENT of gas or vapour is passed through a chamber and is oxidised by oxygen supplied in a number of fine streams, some of which move in the direction opposite to the current of gas or vapour and others at right angles thereto, so as to produce a swirling motion in the current of gas.—W. H. C.

*Calandria for vacuum drying-pans and the like*. H. S. Truscott, Makaweli, Hawaii. U.S. Pat. 1,249,557, Dec. 11, 1917. Date of appl., Sep. 5, 1916.

THE upper and lower tube-plates of the calandria have the form of an inverted truncated cone with a peripheral horizontal portion near the casing. The calandria has a large vertical central tube, surrounded by groups of smaller, inclined tubes set at right angles to the inclined portion of the tube-plates, and these are again surrounded by a ring of larger vertical tubes.—W. H. C.

*Filter frame or leaf*. O. J. Salisbury, Salt Lake City, Utah. U.S. Pat. 1,249,835, Dec. 11, 1917. Date of appl., Mar. 18, 1916. Renewed Aug. 4, 1917.

A FILTER frame is built up of sections of segmental shape, each of which consists of parallel, spaced, perforated plates covered with filter cloth and enclosing the filtrate space between them. The radial edges of the sections are tongued and grooved so that several may be united, substantially in one plane, into a complete circular frame or leaf.—W. H. C.

*Steam-heated drying-cylinders; Testing attachment for* —. G. H. Barrus, Brookline, Mass., Assignor to International Paper Co. U.S. Pat. 1,249,886, Dec. 11, 1917. Date of appl., Sep. 30, 1915.

THE pipe by which the condensed water from a rotary steam-heated drying cylinder is discharged, is provided with means for stopping the discharge at will, and also with a vent discharging into the air so that the condensed water can be inspected. The discharge pipe can be turned about its axis so as to vary the height of the inlet opening.—W. H. C.

*Separation of suspended particles from gases; Process and apparatus for* —. H. A. Burns, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,250,088, Dec. 11, 1917. Date of appl., Apr. 18, 1914.

THE gas is passed through the space between a discharge electrode having a relatively small surface and a liquid receiving electrode having a relatively large surface. A continuous electrical

discharge, maintained from the discharge to the collecting electrode, charges the suspended particles and causes them to move across the path of the stream of gas to the surface of the liquid collecting electrode. The latter flows in a continuous stream and carries away the collected particles.—W. H. C.

*Evaporator.* D. Cozzolino, Los Angeles, Cal. U.S. Pat. 1,250,094, Dec. 11, 1917. Date of appl., Sep. 20, 1916.

THE liquid to be evaporated is sprayed into the top of a vertical cylinder, and falls through a number of spaced, superposed screens of smaller diameter than the cylinder, the concentrated liquid collecting in the dome-shaped lower end of the cylinder. The cylinder has an upper vapour exit connected with a vacuum pump and is provided with a cooling coil just above the surface of the liquid to break up any foam that is formed.—W. H. C.

Initial temperature, °C. ....	20	100	150	200	250	300	350	400	500	600	700	750	800
Methane %, lower limit .....	6.00	5.45	5.20	5.05	4.60	4.40	4.15	4.00	3.65	3.35	3.25	—	—
Methane %, upper limit .....	13.40	13.50	13.60	13.85	14.00	14.25	—	14.70	15.35	16.40	18.75	23.60	29.00

*Evaporator.* G. C. Westby, Ludwig, Nev., Assignor to Western Process Co. U.S. Pat. 1,250,258, Dec. 18, 1917. Date of appl., Aug. 17, 1915.

A SERIES of layers of slats are arranged in vertical tiers in a framework, with an open space extending vertically through the centre of the layers, in which a pipe coil is arranged. A heating fluid is circulated through the pipe coil and the liquid to be evaporated is distributed from a superposed tank having its bottom perforated above the slats but not above the central space.—W. F. F.

*Liquids; Method of treating—*to remove dissolved salts. H. L. Doherty, New York. U.S. Pat. 1,251,486, Jan. 1, 1918. Date of appl., July 16, 1912. Renewed June 7, 1917.

THE liquid is subjected to comparatively quick heating in steps at temperatures increasing successively, by contact with a gaseous heating medium which has been partly preheated by contact with a previous charge of the liquid. The liquid is withdrawn at each step before any substantial amount of solid has crystallised, and caused to flow in a thin film over surfaces

Initial pressure, mm. mercury .....	760	1250	2100	2900	3350	3750	4650
Methane %, lower limit .....	6.00	6.05	—	6.20	6.25	—	6.40
Methane %, upper limit .....	13.00	13.15	13.35	13.60	—	13.80	14.05

of a material which promotes crystallisation. The area of the surfaces and the rate of flow of the hot liquid are so regulated as to permit the crystallisation of substantially the whole amount possible at each heating stage.—W. F. F.

*Separating materials; Apparatus for—*. E. Kardos, Chrome, N.J., Assignor to Th. Goldschmidt A.-G., Essen, Germany. U.S. Pat. 1,250,590, Dec. 18, 1917. Date of appl., Dec. 22, 1915.

SEE Eng. Pat. 3339 of 1915; this J., 1916, 459.

*Apparatus for the fractional uninterrupted distillation of liquid mixtures.* Eng. Pat. 106,088. See III.

*Process of making decolorising agents and the products thereof.* U.S. Pat. 1,251,546. See XVII.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Methane and air; Effect of temperature and pressure on the limits of inflammability of mixtures of—*. W. Mason and R. V. Wheeler. Chem. Soc. Trans., 1918, 113, 45—57.

MIXTURES of methane and air were saturated with water vapour at 20° C. and ignited by means of an electric spark in a U-tube of transparent quartz, one limb of which was closed and was longer than the other. Since the limits for upward or horizontal propagation of flame are wider than for downward propagation (this J., 1914, 1194), a flame just sufficient to reach the bottom of the shorter limb was carried up into the longer limb, where it could be observed in the space projecting above the furnace which heated the U-tube. The effect of temperature upon the inflammability at pressures lower than atmospheric pressure was thus found to be as follows:—

These results agree closely with those obtained by Taffanel and Floch (this J., 1913, 646, 939, 999). Under the conditions described, combustion without flame takes place to an appreciable extent above 600° C., as is shown by the fact that when a mixture containing 18.7% of methane was left in the tube for 5 seconds at 700° C. before sparking, no ignition occurred, whilst at 750° C. a mixture containing 23.5% of methane became non-inflammable after standing for 1.5 secs. before sparking. This may be attributed to the consumption of oxygen by the methane, and hence the results above 600° C. are not the true values for the upper limits of the auto-propagation of flame in mixtures of unaltered methane and air. For measuring the limits of inflammability at pressures above atmospheric pressure, the mixtures of methane and air were forced through a condensing syringe into a strong vertical glass tube the lower part of which was covered with black paper in which was cut a horizontal slit at a point 15 cm. below the point of ignition. Flame seen to pass this slit was considered to have traversed the tube. The following results were obtained:—

These lower limits agree closely with the results of Terres and Plenz (this J., 1916, 461), but the upper limits are higher than theirs, probably because in their experiments the source of ignition was not strong enough. Mixtures containing more than 11% of methane require a secondary discharge of considerable intensity for their ignition, and the intensity necessary increases rapidly with the rise in the proportion of methane. (See also this J., 1916, 345, 682; 1917, 283, 378, 702, 955.)—C. A. M.

*Hydrocarbon gases and vapours; "Spark-lengths" in—*. R. Wright. Chem. Soc. Trans., 1918, 113, 79—80.

THE following average results were obtained from three or more measurements, which usually differed by as much as 10%:—



## Spark-lengths.

	Hydrogen.	Methane.	Ethane.	Propane.	n-Butane.	n-Pentane.	Isopentane.	Hexane.	Heptane.	Benzene.	Toluene.
Air-gap=30 mm.	80	31	25	20	16	14	10	12	9	13	12
Air-gap=20 mm.	55	22	18	15	13	11	8	10	6	9	9

With the exception of isopentane an increase in the insulating power accompanies a rise in the molecular weight in the series.—C. A. M.

[Petroleum] oil prospects of the British Isles. W. H. Dalton. J. Inst. Petroleum Tech., 1917, 4, 37—50.

THE author reviews the known instances of the occurrence of petroleum in the British Isles and shows that oil in small quantity has been found over the whole country and in many of the geological formations from the Old Red Sandstone upwards. He concludes that oil-forming conditions have frequently recurred, but to a very limited extent, and that although conditions favourable to the accumulation of oil, and tectonic structures capable of conserving it from escape, are also of frequent occurrence, the conjunction of the latter essentials with original formation has generally failed. Whether from defect of original formation, of space accessible for accumulation, or of adequate seal from escape, the total result is, from a practical commercial point of view, almost entirely valueless.—T. St.

Motor fuel; Testing and standardisation of —. E. L. Lomax. J. Inst. Petroleum Tech., 1917, 4, 6—25.

THE author discusses the methods which have been proposed for determining the constituents and character of petroleum spirits, with the object of calling attention to the need for the adoption of British standard tests and specifications for motor fuel. *Specific gravity.* Owing to the varying amounts of the several classes of hydrocarbons which are present in spirits from different sources, the specific gravity is no criterion of the volatility, or, consequently, of the value as a motor fuel, of any given sample, and serves only as the basis for calculating the weight per given volume. It would be desirable, however, to have a generally accepted standard temperature for its determination. *Distillation tests.* Of the two best known methods of carrying out the distillation test, the Redwood method is shorter and less liable to personal error than the Engler method, but tends to mask the presence of low-boiling hydrocarbons, particularly when these are present in small quantities. The author has designed a special form of apparatus, of which a detailed description is given, to obtain the completeness of fractionation of the Engler method with the rate of distillation and ease of manipulation of the Redwood method. The special feature of this apparatus is a 4-pair Young dephlegmator column protected from draughts, and air-lagged, by an enclosing wide glass tube. *Sulphur.* The sulphur-content of petroleum spirits may be determined either by burning the spirit in a current of air and absorbing the products of combustion, or by combustion with oxygen in a calorimetric bomb. Owing, however, to the small weights which can be taken for test in the latter method, the accurate estimation of small percentages of sulphur becomes very difficult. The author gives a brief description of various modifications of the air-combustion method, and describes in detail one in which the petrol is mixed with alcohol and the mixture burnt in a good current of air, the products of combustion being absorbed by a definite quantity of a standard solution of sodium carbonate,

which is afterwards titrated against a standard solution of sulphuric acid, methyl orange being used as indicator. The combustion of all the petrol is assured by burning the petrol-alcohol mixture to dryness, and afterwards burning two or three successive small quantities of alcohol alone in the lamp. The method is stated to be quick and to give reliable results. *Calorific value* is determined in the bomb calorimeter. The pressure should be at least 25—30 atmospheres to ensure complete combustion. The spirit may be introduced into the platinum capsule of the bomb by means of a Lunge and Rey pipette, or may be sealed in a thin glass bulb which collapses on charging the bomb with oxygen. *Olefines* are usually estimated by shaking the spirit with an equal volume of strong sulphuric acid in a graduated tube and noting the decrease in volume of the spirit; by the bromine or iodine absorption method; or by shaking the spirit frequently for about 15 minutes with a standard solution of potassium permanganate acidified with sulphuric acid, and then titrating the excess of permanganate with ferrous ammonium sulphate. All three methods are affected by secondary reactions and give only approximate results. *Aromatic hydrocarbons* may be determined by sulphonation and nitration methods, or by extraction at low temperatures with liquid sulphur dioxide and separation by fractional distillation. All these methods are easy of manipulation and give fairly reliable results. *The spontaneous ignition temperature* (this J., 1917, 109) is the lowest temperature to which a platinum crucible must be heated in order that when a drop of the petrol is allowed to fall into the crucible an explosion will occur. This temperature is of great interest to automobile engineers, with relation to the compression limits, but further experiments are required before the method can be employed as a standard test.—T. St.

Toluol by cracking solvent naphtha in the presence of blue gas. G. Egloff. J. Ind. Eng. Chem., 1918, 10, 8—9.

THE author suggests the use of water-gas plants for the production of toluol by cracking solvent (coal-tar) naphtha in the presence of blue gas. From fifteen million galls. of solvent naphtha, the calculated produce of 1917, two million gallons of toluol could be produced, a more than sufficient number of carburetted water-gas plants for this purpose being already in operation in the United States. A test run on a Lowe 6 ft. carburetted water-gas plant over a period of 48 hrs. gave 57% of light oil containing benzol, 14.5% toluol, 23.7%; solvent naphtha, 19.8%; heavy naphtha, 9.7%; "dead" oil, 14.0%. The plant had a capacity of 500,000 cub. ft. per 24 hrs., and the amount of solvent naphtha used was 6000 galls., or 12 galls. per 1000 cub. ft. of gas produced.—L. A. C.

Acetylene; Storage of dissolved —. Home Office Report.

THERE are practically only three porous materials in use for absorption of dissolved acetylene in the United Kingdom: (A) a porous agglomerate of asbestos, kieselguhr, and charcoal, and a suitable cement; (B) charcoal filling; (C) kapok filling. These are satisfactory, but only the most suitable form of charcoal should be used. A sample of



each porous material allowed should be deposited at the Home Office. The porosity should not exceed 80%. Acetone is the most suitable commercially procurable solvent; substitutes should be allowed, but mixtures of various solvents are inadvisable. The solvent must not completely fill the porous material under any rise of temperature likely to be experienced. With the present type of cylinder, the pressure at present allowed, i.e., 150 lb. per sq. in., should not be increased; for solid drawn cylinders and for those of 100 cub. ft. capacity and over in which the acetylene-welding at the top and bottom is strengthened, the pressure allowed might safely be increased to 225 lb. per sq. in., provided a solvent is used in addition to the porous material. The pressure allowed should be stated at 60° F. (15.5° C.). The best material for the construction of cylinders is a mild steel of high ductility, of which the carbon content does not exceed 0.25%, and the phosphorus and sulphur each do not exceed 0.05%. The test pressure should be four times the working pressure and should be maintained for not less than fifteen minutes; re-testing, beyond visual examination, is not necessary. The use of acetylene-welded cylinders should be discontinued as soon as possible; those in use at present should be strengthened at the joints by spinning them over. Fusible plugs should not be used. Filling stations should be approved by, and subject to inspection by, the Home Office. Imported cylinders should be certified by the importer that they comply with regulations. One company recharging cylinders manufactured by another company should first obtain information as regards porous material and solvent. Records of cylinders, all of which should be marked, should be kept, and the use of cylinders for any other purpose should be prohibited. The charging of cylinders by the application of low temperature should be allowed.—L. A. C.

## PATENTS.

*Fuel briquettes from combustible waste products; Production of*—O. A. Tollefsen, Christiania. Eng. Pat. 112,514, Jan. 17, 1917. (Appl. No. 862 of 1917.)

FUEL briquettes are composed of paper waste 60%, sawdust 20%, tanning bark, fir and pine cones and splinters 15%, and a binder such as resin, flour, or glue 5%. The binder, mixed with water at 60° C., is first added to the paper waste softened in water, the other ingredients are added in succession, and the mixture is briquetted and dried.

—W. F. F.

*Gas-producer*. C. W. Lummis and G. H. Isley, Assignors to Morgan Construction Co., Worcester, Mass. U.S. Pat. 1,251,064, Dec. 25, 1917. Date of appl., May 21, 1914.

A U-shaped arm is pivoted at one end about a horizontal axis fixed in the top of the producer, the other end resting on the fuel, and the whole is arranged so that the arm can be moved radially so as to follow changes in the height of the fuel bed, and also horizontally to level the surface of the fuel.—A. B. S.

*Oils; Process and apparatus for treating*—O. C. Swan, Assignor to The Swan Process Oil Co., Denver, Colo. U.S. Pats. (A) 1,250,526 and (B) 1,250,527, Dec. 18, 1917. Dates of appl., June 11, 1917, and Feb. 21, 1916.

(A) IMPURE oil is initially heated with a suitable "defecating agent" and passed into a flotation separator in which it is mixed with a charge of an inert absorbent powder. The mixture is then filtered and the oil thus purified is relieved of its

naphtha content by distillation, the still residue being treated as before. (B) Impure oil is filtered and passed into a treating tank in which it is mixed by agitation with a highly buoyant and absorptive material; it is then passed into a flotation separator tank in which the heavier impurities are precipitated. The top layer of oil flows into a pressure filter of the type having replaceable bag-covered frames, whereby remaining colloidal and other suspended impurities are removed.—L. A. C.

*[Petroleum] hydrocarbons; Distilling*—J. W. Coast, jun., Tulsa, Okla., Assignor to The Process Co. U.S. Pat. 1,250,798, Dec. 18, 1917. Date of appl., Oct. 23, 1916.

HIGH-BOILING petroleum hydrocarbons are vaporised and cracked under a pressure greater than 50 lb. Steam, at a temperature lower than that of the vapour, is introduced into the retort above the liquid level, so that the hydrocarbons of relatively high boiling point condense and return for further cracking treatment. The remaining vapour is removed and condensed.—L. A. C.

*Petroleum hydrocarbons; Art of and apparatus for cracking*—J. W. Coast, jun., Tulsa, Okla., Assignor to The Process Co. U.S. Pats. (A) 1,250,799 and (B) 1,250,800, Dec. 18, 1917. Dates of appl., Dec. 11 and 18, 1916.

HYDROCARBON vapours are led off from a retort under a pressure greater than 50 lb. per sq. in., through an air-cooled reflux condenser or series of condensers, and then through a steam chamber supplied with superheated steam. The high-boiling hydrocarbons are condensed and returned to the retort through the condenser. The steam and low-boiling hydrocarbons are conducted to a main condenser under atmospheric pressure. A pressure-regulating valve is situated between the steam chamber and main condenser.—L. A. C.

*Petroleum hydrocarbons; Cracking*—J. W. Coast, jun., Tulsa, Okla., Assignor to The Process Co. U.S. Pat. 1,250,801, Dec. 18, 1917. Date of appl., May 5, 1917.

RELATIVELY cool steam is forced into hydrocarbon vapours (produced by heating hydrocarbons in a retort) confined at a pressure greater than two atmospheres; the condensed portions are returned to the still and the remaining mixture of steam and vapour is cooled (under pressure) to condense an intermediate fraction. Relatively cool steam is then forced into the remaining mixture of steam and vapour, also under pressure, and the steam and hydrocarbon vapour are condensed.—L. A. C.

*Hydrocarbons; Method of and apparatus for fractionating*—L. E. Hirt, Los Angeles, Cal. U.S. Pat. 1,250,879, Dec. 18, 1917. Date of appl., Oct. 21, 1916.

PREHEATED oil vapour is subjected to the action of an electric arc within a closed chamber or retort under regulated pressure, chlorine gas also being admitted to the chamber. The vapours are withdrawn and serve to preheat the oil supply.—L. A. C.

*Coal and the like; Process for producing liquid or soluble organic combinations from hard*—F. Bergius and J. Billwiller, Hanover, Germany. U.S. Pat. 1,251,954, Jan. 1, 1918. Date of appl., Aug. 3, 1914.

SEE Eng. Pat. 18,232 of 1914; this J., 1916, 167. A temperature of less than 600° C. and a pressure exceeding 20 atmospheres are specified.



*Peat; Treatment of* —. T. Rigby, Dumfries, Assignor to Wetcarbonizing, Ltd., London. U.S. Pats. 1,251,285 and 1,251,422, Dec. 25, 1917. Dates of appl., May 7 and July 14, 1914. SEE Eng. Pat. 11,133 of 1913; this J., 1914, 912.

*Gas-producer*. H. J. Green, Bolton. U.S. Pat. 1,252,000, Jan. 1, 1918. Date of appl., July 25, 1915.

SEE Eng. Pat. 17,687 of 1914; this J., 1915, 166.

*Process and means for the separation and recovery of liquids of different densities and the separation of light solid particles from a heavy liquid*. Eng. Pat. 103,670. See I.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Wood distillation*. H. O. Chute, New York, Assignor to K. P. McElroy, Washington. U.S. Pat. 1,250,282. Dec. 18, 1917. Date of appl., May 23, 1910. Renewed May 19, 1917.

THE vapours generated during the distillation of wood are passed into a bell reservoir at a rate proportional to their speed of production. They are cooled in the reservoir to a temperature above the boiling point of pyroligneous acid and then passed through a scrubber against a counter-current of tar oils to remove tar. The flow of vapours through the scrubbers is effected by suction controlled by the bell reservoir. The tar is fractionated and the oil fraction used for scrubbing the vapours.—L. A. C.

*Charcoal; Preparation of vegetable* —. T. E. Rule and J. Nicol, Liverpool, Assignors to A. H. Bonnard, London. U.S. Pat. 1,250,228, Dec. 18, 1917. Date of appl., July 11, 1916.

SEE Eng. Pat. 10,622 of 1915; this J., 1916, 1212.

*Baking-furnace [for carbons, etc.]*. U.S. Pat. 1,251,309. See I.

## III.—TAR AND TAR PRODUCTS.

*Toluol by cracking solvent naphtha*. Egloff. See IIA.

### PATENTS.

*Liquid mixtures [tar]; Apparatus for the fractional uninterrupted distillation of* —. W. Rothenbach, Florence, Italy. Eng. Pat. 106,088, Mar. 29, 1917. (Appl. No. 4562 of 1917.) Under Int. Conv., Apr. 15, 1916.

LIQUID to be distilled, e.g., tar, passes from a reservoir first through the outer parts of the condensers of the system to preheat it, thence to the top of a distilling column or retort, and then to a second similar column. The heating fluid, which may be superheated steam or combustion gases initially at 300°–400° C., is passed through the columns in the opposite direction to the liquid, and the liquid is distilled in a partial vacuum which gradually diminishes to atmospheric pressure in the direction traversed by the liquid, i.e., in the direction of increasing density. The distillation products are withdrawn as separate fractions at four points of the system, and passed into the condensers mentioned above.—W. F. F.

*Pitch; Cooling, solidification and delivery of* —. J. L. Major, Hull. Eng. Pat. 112,370, May 3, 1917. (Appl. No. 6314 of 1917.)

THE hot pitch is run into moulds carried on the band of a conveyor or by a drum or in any other

suitable manner, and after cooling and solidification, the blocks of pitch are discharged from the moulds by their own weight.—W. H. C.

*Toluol and like hydrocarbons; Manufacture of* —. F. Thuman, London. From O. B. Evans, Philadelphia, U.S.A. Eng. Pat. 112,878, Apr. 20, 1917. (Appl. No. 5568 of 1917.)

SEE U.S. Pat. 1,230,087 of 1917; this J., 1917, 866.

*Method and apparatus for fractionating hydrocarbons*. U.S. Pat. 1,250,879. See IIA.

## IV.—COLOURING MATTERS AND DYES.

*Coupling reaction; Mechanism of the* —. O. Dimroth, H. Leichtlin, and O. Friedemann. Ber., 1917, 50, 1534–1548.

DIMROTH supposes that in the first stage of the coupling of a diazonium salt with a phenol a diazo-ether is formed, which would subsequently change into the ordinary azo-compound under suitable conditions. Thus, in the case of *p*-nitrophenol and *p*-bromobenzenediazonium chloride, the intermediate, primary product has actually been isolated:  $\text{NO}_2\cdot\text{C}_6\text{H}_3\cdot\text{O}\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\text{Br} \rightarrow \text{NO}_2\cdot\text{C}_6\text{H}_3(\text{OH})\cdot\text{N}_2\cdot\text{C}_6\text{H}_4\text{Br}$ . Auwers has raised the question, however, whether the primary products are ethers,  $\text{R}\cdot\text{O}\cdot\text{N}$ :  $\text{N}\cdot\text{R}'$ , or diazonium salts,  $\text{R}\cdot\text{O}\cdot\text{NR}'$ :  $\text{N}$ , and a reply is now given. Morgan and Wootton's stable diazonium salts, 4-benzoylamino-naphthalene-1-diazonium and *p*-acetylaminobenzenediazonium chlorides, have been treated with phenols ranging in strengths from picric acid to pentamethylphenol. The molecular conductivities of the products in 80% acetone have been measured, and the results show that the compounds with picric acid and dinitrophenol are true diazonium salts, with the same conductivities as the chlorides, or trichloroacetates, whereas the weaker phenols and certain enols give true ethers, with conductivities as low as that of *p*-nitrobenzeneazo-methyl ether. —J. C. W.

*$\beta$ -Naphthylamine; Studies on the sulphonation of* —. A. G. Green and K. H. Vakil. Chem. Soc. Trans., 1918, 113, 35–44.

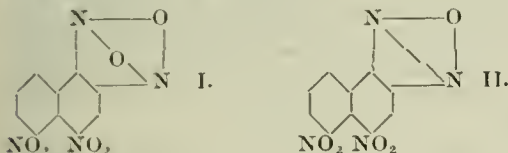
ON sulphonation of  $\beta$ -naphthylamine at temperatures from 20° to 80° C. there was only a slight variation in the proportion of the 2.8- to the 2.5-monosulphonic acids, which together constituted 97–99.5% of the sulphonation product; similar results were obtained for variations of the time from 1 to 10 hrs. Between these limits of temperature and time, the strength of the sulphuric acid appeared to exert little influence, except at 80° C., when an increase in the concentration of sulphur trioxide gave a slightly increased amount of the 2.6- and 2.7-isomerides. The lowest temperature and shortest time of reaction appeared to favour the production of the 2.5-acid. If the temperature of sulphonation is above 80° C., or the time of reaction at 80° C. more than 10 hrs., the percentage of 2.8-acid decreases, whilst that of the 2.5-isomeride increases; this is accompanied by an increased production of the 2.6- and 2.7-acids, the quantity of which reaches 7.5% with 96% sulphuric acid at 120° C. The proportion of the 2.8-acid depends on the proportion formed initially and the subsequent conversion of the 2.8- into the 2.5-acid, the latter factor only becoming prominent above 80° C., or on heating at 80° C. for more than 10 hrs., or by the use of a higher concentration of sulphur trioxide. The presence of the 2.1-acid in the sulphonation product could not be detected, and its behaviour on heating with sulphuric acid indicates that its isomerisation is due to hydrolysis and subsequent

resulphonation; the sulphonic group must therefore enter the second nucleus directly, and not through intermediate formation of the homo-nuclear isomeride. An equilibrium is not established between the 2.8- and 2.5- acids in sulphuric acid solution, as the isomerisation only occurs from the 2.8- to the 2.5-acid. The relative facility with which the isomeric acids undergo hydrolysis to 3-n phthylamine is expressed as follows:  $2.1 > 2.8 > 2.5 > 2.6$  and 2.7. It is concluded that the gradual conversion of the 2.8- into the 2.5-acid at  $80^{\circ}$ — $120^{\circ}$  C. is due to repeated hydrolysis of the 2.8-acid and resulphonation of the  $\beta$ -naphthylamine produced, which gives about 60% of the 2.5-acid. This is accompanied to a small extent by isomerisation to the 2.6- and 2.7-acids, especially when sulphuric acid of high concentration is employed at above  $120^{\circ}$  C., until at  $150^{\circ}$ — $160^{\circ}$  C. they constitute the main sulphonation product; their formation is probably due to concurrent disulphonation and hydrolysis, in which case the 2.6-acid would originate from the 2.8-acid, and the 2.7-acid from the 2.5-acid. The reactions of the sodium salts of the four isomerides with copper sulphate and silver nitrate are described.

—F. W. A.

*Iso-oxadiazole oxides and iso-oxadiazoles; Nitro-derivatives of—* A. G. Green and F. M. Rowe. Chem. Soc. Trans., 1918, 113, 67—74.

REDUCTION of the dinitro-derivative of benziso-oxadiazole oxide with titanous chloride has proved that the compound does not contain a hydroxyl group, as was suspected from its acidic function; the compound is therefore a true dinitrobenziso-oxadiazole oxide in which a hydrogen in the benzene ring possesses acidic properties. The dinitro-deriv. tive of naphthiso-oxadiazole oxide (I.) and two mono- and the di- (II.) derivatives of naphthiso-oxadiazole have been prepared, and probably have the following constitution:—



—F. W. A.

*Phenazononium and its simplest derivatives.* F. Kehrman and A. Boubis. Ber., 1917, 50, 1662—1667. (Compare this J., 1914, 687; 1915, 654, 792.)

It was proved a year or two ago that phenazononium forms both *meri-* and *holo-*quinonoid salts, and consequently a revision of other theories and spectrographic data in the case of phenazononium became necessary. The present chemical and optical investigations (see also following abstract) show that phenazononium is also normal in its behaviour towards acids. The reddish-violet salts obtained by dissolving phenazoxine in sulphuric acid, whether dilute or concentrated, are *meri-*quinonoid, mono-acid salts. If hydrogen peroxide is added to the solutions in concentrated sulphuric or perchloric acids, yellow, *holo-*quinonoid, di-acid salts are formed. These are unstable but can be preserved for a short time at  $0^{\circ}$ — $10^{\circ}$  C. If glacial acetic acid is added to these yellow solutions, the colour changes to wine-red, *holo-*quinonoid, mono-acid salts being

formed. The perchlorate,  $C_8H_4 \begin{array}{c} \diagup N \\ \diagdown O \end{array} C_2H_4$ ,  $ClO_4$

may even be isolated, as an explosive, brown powder. Similar behaviour is shown by 3.9-dimethyl- and 5- and 3-amino-phenazononiums.

—J. C. W.

*Quinoneimide dyes.* VII. Spectra of the simplest azoxine dyes. VIII. Supplementary observations on the absorption spectra of the simplest azothionium compounds. IX. Absorption spectra of the mono-acid salts of 3.9-diaminophenazononium. F. Kehrman and M. Sandoz. Ber., 1917, 50, 1667—1673, 1673—1682, and 1682—1683. (Compare preceding abstract.)

AN account, with tables and curves, of the absorption spectra of the salts of phenazononium, 3.9-dimethyl- and 3- and 5-amino-phenazononiums; phenazothionium, 6-methyl- and 5-amino-phenazothioniums; 3.9-diaminophenazononium monohydrochloride, 3-amino-9-anilinophenazononium 9-hydrochloride, 3.9-dianilinophenazononium monohydrochloride, 3.9-tetramethyldiaminophenazononium nitrite, and 3-dimethylamino-9-anilinophenazononium 9-monohydrochloride. In many cases the data correct earlier notices. All the phenazothioniums behave alike in the ultra-violet, exhibiting a band at  $\lambda$  290  $\mu\mu$ , whilst the phenazononiums are all alike in giving two bands, at  $\lambda$  265 and 285  $\mu\mu$ .—J. C. W.

*$\alpha$ -Naphtholphthalein; Preparation of —.* E. A. Werner. Chem. Soc. Trans., 1918, 113, 20—21.

IN place of the phthalyl chloride used by Grabowski (Ber., 1871, 4, 725; 1873, 6, 1065), phthalic anhydride was used. A mixture of 7 grms. of  $\alpha$ -naphthol and 3.8 grms. of phthalic anhydride was treated with 0.75 c.c. of concentrated sulphuric acid for 4 hrs. below  $65^{\circ}$  C. After extraction with water to remove sulphuric acid, the semi-solid mass was digested with 0.5% caustic soda at  $70^{\circ}$  C.; 0.8 grm. of Grabowski's "anhydride" (Ber., 1871, 4, 661) was left undissolved. The deep blue solution was treated with dilute hydrochloric acid sufficient to neutralise half the caustic soda, and the phthalein precipitated by means of carbon dioxide. The yield after reprecipitation was 2.3 grms.—F. W. A.

*Phthalic acid derivatives; Constitution and colour of —.* V., VI., VII. D. S. Pratt and G. A. Perkins. J. Amer. Chem. Soc., 1918, 40, 198—236. (See also J. Chem. Soc., March, 1918.)

*Some derivatives of tetrachloro- and tetra-iodophthalimides.*—In order to study the absorption spectra of different members of the same family with a view to obtaining a clearer insight into the relation between constitution and colour, the authors have prepared a large number of phthalic acid derivatives. A number of derivatives of tetrachloro- and tetra-iodophthalimides have been prepared and a new type of phthalic acid derivative in which chlorine in the nucleus is replaced by anilino groups. *Action of amines upon dichlorophthalic anhydrides.*—The authors describe the chlorination of phthalic anhydride and the separation of the isomers; the preparation of 3.4-, 3.6-, and 4.5-dichlorophthalanils and the aniline derivatives of the first two, the last giving nothing but tarry products. *Di-iodo-, tri-iodo-, and dichloro di-iodophthalic acids.*—The authors describe the iodation of phthalic acid, of phthalic anhydride, and of the three dichlorophthalic acids, and the separation of the resulting compounds; also the preparation of hydroxyl derivatives and anils of these compounds.—L. A. C.

*Phthalic acid derivatives; Constitution and colour of —.* VIII., IX., X. D. S. Pratt and A. B. Coleman. J. Amer. Chem. Soc., 1918, 40, 236—251. (See also J. Chem. Soc., March, 1918.)

*Tetraiodofluorescein and some of its deriva-*



*tives*.—The authors describe the preparation of tetraiodofluorescein and certain of its salts and derivatives. The compound exists at ordinary temperatures as an unstable mixture of the benzenoid and quinonoid modifications. The hydrate losses water readily to form a yellow hydrous derivative containing one molecule of water of crystallisation; this on heating gives the reddish-brown anhydride. *Tetraiodococsin* and some of its derivatives. *Tetraiodococsin* was prepared by the action of bromine on tetraiodofluorescein suspended in boiling glacial acetic acid. The compound forms canary-yellow crystals of the benzenoid modification. *Tetraiodoerythrosin* (*octoiodofluorescein*) and some of its derivatives.—Iodine substitution products of fluorescein have been made commercially by methods of electrolysis for use as dyes; these, however, are mixtures and contain no iodine in the phthalic residue. Pure tetraiodoerythrosin was prepared by treating tetraiodofluorescein with iodine and iodic acid in absolute alcohol and heating under a reflux condenser. A lower halogenated product was obtained as a by-product.—L. A. C.

*Phthalic acid derivatives; Constitution and colour of*—. XI. *Phenoltetraiodophthalein and some of its derivatives*. D. S. Pratt and A. F. Shupp. J. Amer. Chem. Soc., 1918, 40, 254—264. (See also J. Chem. Soc., March, 1918.)

THE authors describe the preparation of pure phenoltetraiodophthalein, its dinitro-, tetra-nitro-, tetrabromo-, and tetraiodo-derivatives, and their diacetates, dibenzoates, and dimethyl ethers. Tetraiodofluoran was obtained as a by-product.—L. A. C.

*Colloidal nature of colophony*. Paul. See XIII.

*Utilisation of the adsorptive power of fullers' earth for chemical separations. [Adsorption of quinine and Methylene Blue.]* Seidell. See XX.

#### PATENTS.

*Dyestuffs [from algae]*. N. Malcolmson, London. Eng. Pat. 112,474, Oct. 10, 1916. (Appl. No. 14,390 of 1916.)

A DYE paste or pulp is obtained from marine algae by injecting steam into the raw untreated weed, with or without subsequent evaporation. Dye liquors are obtained from olive, brown, or red algae by boiling the weed with an acid solution and then treating with a hot alkaline solution. In the case of olive and brown algae, the solution produced by treatment with acid may be evaporated to one-half to one-eighth its bulk, when the dyestuff obtained gives a shade different to that obtained if this step is omitted.—F. W. A.

*Sulphur dyes; Manufacture of*—. H. Heimann, Dessau, Assignor to Act.-Ges. f. Anilin-Fabr., Berlin, Germany. U.S. Pat. (A) 1,251,368 and (B) 1,251,369, Dec. 25, 1917. Date of appl., Jan. 21, 1915.

SULPHURISED dyes giving olive to brown shades are obtained by the action of sulphur on a mixture of (A) an amino or (B) an aminonitro derivative of the benzene-azo-naphthalene series with an aromatic C-alkylated diamine. The dyestuffs from (A) benzene-azo- $\alpha$ -naphthylamine and (B) nitrobenzene-azo- $\alpha$ -naphthylamine with *m*-toluylendiamine are specially claimed; they produce brown shades on cotton, fast to washing and to boiling dilute acids.—F. W. A.

*Aniline dyestuffs; Manufacture of*—. D. de Nagy, Assignor to A. E. King and G. H. Odell, London. U.S. Pat. 1,250,289, Dec. 18, 1917. Date of appl., June 28, 1917.

SEE Eng. Pat. 16,203 of 1915; this J., 1917, 78.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Velveteens; Ignition and combustion of* — in hermetically sealed tin cases. W. Thomson. J. Soc. Dyers and Col., 1918, 34, 6—7.

A CASE of ignition of velveteens packed in a hermetically sealed tinned iron case for shipment has been investigated. It was found that samples of black velveteen in a tin case could be ignited by the soldering iron used in sealing the case. Whereas the smouldering of velveteens dyed in colours gradually becomes extinguished, the iron oxide present in black velveteen, dyed with Logwood Black and topped on the pile with Prussian Blue, has the effect of continuing the combustion until only ash remains.—F. W. A.

*Wood; Constituents of* — which give colour reactions. II. H. Wichelhaus and M. Langc. Ber., 1917, 50, 1683—1685. (Compare this J., 1916, 1151.)

By the passage of steam at 180°—200° C. through pine or fir wood, a distillate is obtained which contains substances that give colour reactions with phloroglucinol and hydrochloric acid. The less volatile third of the liquid gives a typical cherry-red precipitate, and also a brick-red substance,  $C_{13}H_{15}O_5N_3$ , on adding *p*-nitrophenylhydrazine. This is apparently a derivative of a keto-furfuraldehyde,  $C_6H_5O_5$ , which would be related to a hexose thus:  $C_6H_{12}O_6 - H_2O + O = C_6H_5O_5 + H_2O$ .—J. C. W.

#### PATENTS.

*Fabric for aeroplanes, kites and the like; Method of manufacturing and after-treating*—. R. Wheatley, Edinburgh. Eng. Pat. 112,483, June 1, 1917. (Appl. No. 17,503 of 1916.)

AEROPLANE fabric and the like is treated with solutions of acetylcellulose or nitrocellulose or both and of a suitable protective colouring substance or substances soluble in the solution and in the dried dope, to protect the dope against deleterious incident radiation. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 105,137; this J., 1917, 544.)—F. W. A.

*Cotton-stalks; Process and apparatus for reducing* — to pulp suitable for the manufacture of paper. J. M. Irwin, Dallas, Tex. U.S. Pat. 1,250,106, Dec. 18, 1917. Date of appl., Sep. 14, 1916.

COTTON stalks are placed in a container in which hydration and chemical treatment are effected and the mass is alternately compressed, first from one end of the container and then from the other, and during the alternate compression periods the individual stalks and fibre body of the mass are caused to pass through and between "moving interstices of the container," setting up such a frictional engagement between the stalk or fibre body and the machine element as to cause the tentacles and fibre to be drawn and abstracted from the body of the stalk. In the container a shaft is arranged axially, carrying two attrition wheels provided with oppositely turned blades, adapted to travel along the shaft contiguously with each other and revolving in opposite directions.—J. F. B.

*Sulphite-cellulose liquors; Purifying exhausted* —. A. G. Bloxam, London. From Zellstoff-fabrik Waldhof, Mannheim-Waldhof, Germany. Eng. Pat. 112,395, Aug. 10, 1917. (Appl. No. 11,537 of 1917.)

WASTE sulphite liquor is freed from calcium salts by treatment with sodium carbonate, then concentrated to a density of about 30° B. (sp. gr.



1-246) and stirred with sufficient sulphuric acid to convert all the salts into sulphate. The mass is cooled while stirring, and the sodium sulphate is allowed to crystallise out from the liquor, which is left at rest at a low temperature for a period ranging from a few hours to several days.—J. F. B.

[Hydr]oxylignin and [hydr]oxylignon from lignin and lignon sulphonates [in sulphite cellulose waste liquor]; Method of preparing —. E. Oman, Stockholm, Sweden. Eng. Pats. (A) 103,649 and (B) 103,650, Jan. 4, 1917. (Appl. Nos. 181 and 182 of 1917.) Under Int. Conv., Jan. 12, 1916.

SULPHITE-CELLULOSE waste liquor is saturated with sodium chloride and heated to 40°–50° C.; a portion of the sulphonates, amounting to 33–50% of the total sulphonates present, is salted out in the form of a grey flocculent precipitate. The precipitated sulphonates are called ligninsulphonates and the soluble sulphonates lignonsulphonates. (A) Fifty parts of the precipitate containing about 60% of ligninsulphonate and 40% of sodium chloride is mixed with a 50% solution of sodium hydroxide, 300 parts, and 15 parts of potassium chloride, and the mixture is fused at 140°–160° C. for 2–3 hours; the hydroxylignin is precipitated by acidification after dissolving the melt in water. (B) The salt solution containing the lignonsulphonate is concentrated to about one-sixth of its volume and the sodium chloride is removed; 1 litre of the solution is treated with 750 grms. of solid sodium hydroxide and 60 grms. of potassium chlorate, and the sulphonic groups are eliminated by fusion as above.—J. F. B.

Lignin and lignon sulphonic acids or their salts; Method of preparing nitro compounds from —. E. Oman, Stockholm. Eng. Pats. (A) 103,651 and (B) 103,652, Jan. 4, 1917. (Appl. Nos. 202 and 203 of 1917.) Under Int. Conv., (A) Jan. 15 and (B) Jan. 20, 1916.

(A) 100 GRMS. of sodium ligninsulphonate (see preceding abstract) of about 60% concentration is added gradually, with stirring and cooling, to 200 grms. of 50% nitric acid, at 30°–40° C. The nitrated compound is partly deposited, and the remainder is extracted by neutralising the solution and precipitating with an equal volume of alcohol. (B) 200 c.c. of the filtrate from the waste sulphite liquor after separation of the ligninsulphonate, concentrated to  $\frac{1}{2}$  of its volume, is added gradually, with stirring and cooling, to 500 grms. of a nitrating acid prepared from 1.5 parts of sulphuric acid of sp. gr. 1.84, and 1 part of nitric acid of sp. gr. 1.4, at 30° C. The nitro-compound is precipitated directly, or if nitric acid alone has been used, the product may be precipitated by alcohol after neutralisation.—J. F. B.

Lignin sulphonates prepared from sulphite-cellulose waste lye; Method of treating — so that they may be dried without decomposition. E. Oman, Stockholm. Eng. Pat. 103,655, Jan. 4, 1917. (Appl. No. 206 of 1917.) Under Int. Conv., Jan. 15, 1910.

THE ligninsulphonates precipitated by salt from sulphite-cellulose waste liquor tend to blacken during drying, owing to the presence of free acid which cannot be removed by washing the precipitate with sodium chloride solution. This defect may be overcome by washing the precipitate with saturated sodium chloride solution or alcohol containing free alkali, such as sodium hydroxide or carbonate.—J. F. B.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Combustibility of cotton; Influence of certain processes of dyeing on the —. G. E. Holden. J. Soc. Dyers and Col., 1918, 34, 7–9.

THE rate of burning of dyed cotton was not accelerated by the presence of tannates of aluminium, antimony, and tin, but was considerably increased by the tannates of iron, manganese, lead, and copper. Probably owing to the relatively small amounts of copper or chromium precipitated on the fibre, no appreciable effect was produced by the after-treatment of substantive and sulphide dyeings with copper sulphate and bichromate respectively. Details of possible sources of certain metals in dyed cotton material are given. In determining the suitability of the various dyeing processes for certain materials, the ultimate destination should be taken into account, and the "burning test" should receive more attention.—F. W. A.

Cotton-fibre; Case of mechanical powdering [during beetling] of the —. M. Fort. J. Soc. Dyers and Col., 1918, 34, 9–10.

IF, owing to the breaking of a traverse during beetling, the hammers fall continuously on the same sector of the cloth beam, the temperature rises, and conditioning moisture is lost; the subsequent yellowing takes place more rapidly with linen than with cotton. The formation of "chalky" patches on the cloth by beetling is accounted for by the breaking down of fibres, and is assisted by tendering during the bleaching process. In the case described, an extraordinary degree of mechanical destruction of full-bleached, unmercerised cotton damasks, sound and free from chlorine and acid, was obtained, due to the breaking of a traverse during beetling. In less than half an hour, a considerable amount of cloth was ruined, and a large deposit resembling lumps of starch was obtained, which was easily crushed to a fine smooth powder with a slight tendency to break in layers. The product contained no undestroyed fibres, but an occasional small shred of cuticle was present; it consisted of particles quite as uniform but much less in size than starch, arranged in clusters, and often approximating to a cylindrical shape. The powder had the chemical properties of cellulose, being dyed by Methylene Blue and Congo Red similarly to ordinary bleached cotton; it was unchanged by boiling with water, and gave negative results to all tests for oxy-cellulose and hydrocellulose; on treatment with cold caustic soda (52° Tw.) the particles swelled but remained undissolved. The product gave no reaction with iodine solution, but after treatment with hydrochloric acid, the yellowish-brown coloration due to dextrin was obtained.—F. W. A.

Analysis of commercial dextrins. Lamb and Harvey. See XVII.

### PATENTS.

Bleaching process. H. R. Anders, Perth Amboy, N.J., Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,251,614, Jan. 1, 1918. Date of appl., Mar. 24, 1917.

INDUSTRIAL fibres and fibrous materials are wetted out with lime water at an elevated temperature, and then bleached in a bath containing "a substance carrying active oxygen," made alkaline by means of calcium hydroxide.—F. W. A.

Dyeing machines. J. Farrar, Halifax, and A.R. Whitehead, Leeds. Eng. Pat. 112,482, Dec. 6, 1916. (Appl. No. 17,497 of 1916.)

In dyeing machines in which the dye liquor circulates through the material and is heated by



steam from perforated pipes along the bottom of the vat, direct contact of the steam with the material and undue agitation of the liquor are avoided by providing hoods over the steam pipes, or by the use of an additional steam pipe in the circulating chamber to maintain the temperature of the liquor. In the second case the liquor is heated rapidly to the desired temperature by steam from the pipes in the dye-vat before the material is introduced, after which the temperature is maintained by means of the steam pipes, in the circulating chamber.—F. W. A.

*Dyestuffs [from algæ].* Eng. Pat. 112,474. See IV.

## VII.—ACIDS ; ALKALIS ; SALTS ; NON-METALLIC ELEMENTS.

*Sulphuric acid ; De-arsenication of*——. H. E. J. Cory. Chem. Trade J., 1918, 62, 89—90.

THE old type of de-arsenicating plant, consisting of a tower in which an ascending stream of hydrogen sulphide meets a descending stream of sulphuric acid, is effective with weak acid, but costly, sometimes wasteful of hydrogen sulphide, and limited in output. The modern continuous type, in which the acid is agitated by paddles, giving intimate contact with the gas, is very effective for acid up to 110°—120° Tw. (sp. gr. 1.55 to 1.60), and has the advantages of low initial cost, complete removal of arsenic, continuous output, and low running expenses. A third type of plant, in which hydrogen sulphide is pumped into the acid under pressure, is advantageous when a cheap, abundant supply of the gas is available, and it can be employed with a stronger acid, thus saving fuel in the concentration. It is particularly useful for acid low in arsenic. The minimum storage capacity should be one day's supply of arsenical and purified acid, and the settlers should be capable of holding half a day's output. Settlers, filters, and communicating troughs may all be made of wood lined with lead. Acid made from light arsenical ores may be filtered through porous tiles cemented together by water-glass thickened with lava or volcanic stone. When heavy arsenical ores are handled, pebble filters, composed of graded layers of quartz, supported on thin flat bricks, with "soap bricks" spaced well apart beneath them, are used. When practicable, the treated acid should be filtered warm. Nitrous compounds in the acid are objectionable, as they react with hydrogen sulphide, and interfere with de-arsenication. The strength of the treated acid must not be too high. The process may be controlled by a qualitative Marsh test of the acid leaving the de-arsenicator, and by the colour of the effluent acid and mud. After settling, a quantitative Marsh or modified Gutzeit test should be applied, and if the result is unsatisfactory, the acids should be sent back to the de-arsenicator, or to the mud plant. The closed (pressure type) process needs more careful control. Hydrogen sulphide at about 10 lb. pressure should be blown in until a quantitative Marsh test gives a satisfactory result. Acids low in arsenic can sometimes be conveniently filtered without settling. When high in arsenic, a considerable quantity of arsenical mud is obtained in the settlers and filters. A recent development is the addition of an oil of low specific gravity, e.g., paraffin, to the acid under treatment, which brings part of the sulphide to the top on standing. The arsenical residues are preferably heated in long leaden pans provided with mechanical scrapers, the acid being run off and returned to the de-arsenicator, while the dried residue is either thrown away or sold. Filter-press treatment of the residue is more or less ineffective.—F. Sp.

*Sulphuric acid ; Action of dilute*——upon a common glass carboy. A. D. Olle. Austral. Pharm. Notes, Sep. 1, 1917, 18.

A SAMPLE of sulphuric acid (sp. gr. 1.18) originally free from iron gave a deep red coloration when oxidised and tested with potassium thiocyanate, and was therefore useless for storage batteries, after it had stood for six years in a green glass carboy. The acid was found to contain 0.00127% iron, 0.0003% of aluminium, 0.0044% of calcium oxide, and 0.0739% of alkalis as  $\text{Na}_2\text{O}$ . Many samples of acid sold as chemically pure were found to contain iron, and iron could be detected in distilled water which had stood for some time in glass vessels.—C. A. M.

*Hydrofluosilicic acid ; Case of poisoning by*——and detection of this acid in animal organs. E. Spaeth. Pharm. Zentralh., 1917, 58, 599—601.

A BOY died soon after drinking a quantity of a disinfectant solution sold under the name of "Montanin," and consisting of a 27% solution of hydrofluosilicic acid. From the stomach and intestines of the boy a quantity of silica corresponding with 1.33 grms. of hydrofluosilicic acid or 4.8 grms. of the original solution was separated. The presence of the acid was detected by extracting the organs with 50% alcohol, filtering the solution, extracting the fat with petroleum spirit, and testing the solution with dilute potassium chloride solution; potassium silicofluoride separated after some time as a clear gelatinous precipitate. Barium chloride gave a white precipitate of barium silicofluoride, whilst ammonia yielded a precipitate of hydrated silica. The filtrate from the latter was treated with milk-of-lime, and evaporated to dryness in a platinum basin; the residue, when treated with sulphuric acid, yielded vapours of hydrofluoric acid. Determination of the silica was found to be the best means of ascertaining the quantity of the acid present in the organs.

—W. P. S.

*Hydrogen cyanide ; Detection of small amounts of*——. I. M. Kolthoff. Pharm. Weekblad, 1917, 54, 1157—1171. (Compare Anderson, this J., 1916, 1083.)

A DETAILED investigation of methods for the detection of hydrocyanic acid has been carried out in order to ascertain the relative delicacy of the various tests. The Prussian blue test is characteristic for hydrocyanic acid. Identification as ferric thiocyanate is also characteristic, but in presence of thiocyanates (e.g., in saliva, etc.), the hydrocyanic acid must be redistilled from borax solution. The picric acid test is not specific for hydrocyanic acid, but is given by many reducing agents, such as sulphurous acid, aldehydes, acetone, and hydrogen sulphide, of which the last-named may be removed by a cadmium solution; a negative result is hence of more value than a positive reaction, and a blank test must be made on the reagents used. The reaction with guaiacum tincture is not typical for hydrocyanic acid, being given by oxidising agents (halogens, ozone, etc.), and by ammonia, cigar smoke, etc.; a negative result has been stated to indicate the absence of hydrocyanic acid, but the reaction may be affected by the presence of certain reducing agents, and is therefore not conclusive. The phenolphthalein reaction is affected by hydrogen sulphide, which may, however, be removed by a cadmium solution; it is more characteristic than that with guaiacum tincture, as halogens or hydrogen peroxide do not give a red coloration; it is found, however, that these and similar substances may prevent the reaction with the hydrocyanic acid. The silver cyanide reaction is not specific for hydrocyanic acid; in presence of chloride the hydro-

cyanic acid may be redistilled from a borax solution. The amyl iodide reaction is given by various reducing agents; the appearance of a blue coloration on acidifying the solution indicates hydrocyanic acid, the reaction being specific in the aqueous distillate.—F. W. A.

*Potassium salts; Existence of bischofite deposits and the secondary transformations of the Zechstein* —. M. Róza. Z. anorg. Chem., 1917, 101, 276—284.

THERE is no reason to suppose that bischofite ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) was among the primary separation products in the potassium salt deposits. The secondary metamorphoses which are supposed to have taken place can be explained without the assumption of a primary bischofite. The "principal" salt deposit, which is 16 metres thick, contains 56% of carnallite, 13.3% of kieserite, and 28.7% of rock salt. This is certainly a primary deposit, and was at one time of vast extent. In spite of the rapid development of the potassium mining industry, very few sections of the deposits have been opened up to geological and chemical-petrographic examination. (See also J. Chem. Soc., March, 1918.)—E. H. R.

*Sodium sulphate—ammonium sulphate; Invariant equilibria in the ternary system: water* —. C. Matignon and F. Meyer. Comptes rend., 1918, 166, 115—119.

THE co-ordinates of the triple point for each of the possible combinations of three solid phases have been determined and are shown on a trilinear diagram. The authors have determined the temperature of the eutectic of ice and ammonium sulphate and find it to be  $-18^\circ\text{C}$ . (See also J. Chem. Soc., March, 1918.)—W. G.

*Rare earths; Electrolysis of solutions of the* —. III. L. M. Dennis and A. B. Ray. J. Amer. Chem. Soc., 1918, 40, 174—181.

IT has been found that the electrolysis of neutral solutions of the nitrates of the rare earths with a mercury cathode is accompanied by a fractional separation of the rare earth hydroxides at the surface of the cathode. The average atomic weight of the rare earth metals in the successive fractions diminishes as the electrolysis proceeds. Violent agitation of the mercury surface increases the efficiency of the fractionation. When solutions containing the nitrates of thorium and the rare earths are similarly electrolysed, the thorium is concentrated in the early fractions. (See also J. Chem. Soc., March, 1918.)—H. M. D.

*Gluceium [beryllium] nitride*. A. C. Vournasos. Bull. Soc. Chim., 1917, 21, 282—288.

BERYLLIUM nitride may be readily prepared by passing cyanogen over metallic beryllium heated at  $800^\circ\text{C}$ . Another general method of preparation of nitrides is by the partial oxidation of a metallic cyanide, or of a carbide in the presence of nitrogen. Thus by heating zinc cyanide or calcium carbide or barium cyanamide with ammonium nitrate the corresponding nitride is obtained in each case. (See also J. Chem. Soc., March, 1918.)—W. G.

*Mercuric ion; Rapid characterisation of the — in insoluble sulphates. Application to the identification of this ion in general*. G. Denigès. Bull. Soc. Chim., 1918, 23, 36—39.

IF a 10% solution of mercuric nitrate in dilute nitric acid (1 in 100) is added to an insoluble sulphate, yellow turpeth mineral (basic mercuric sulphate) is formed at once in the cold with calcium or mercurous sulphates, slowly in the cold with strontium and lead sulphates, and only slowly on boiling with barium sulphate, and may be identified, if necessary, by microscopical examination.

Soluble sulphates in a mixture may be first precipitated as barium sulphate and then detected as described. (See also J. Chem. Soc., March, 1918.)—W. G.

*Mercury ammonia compounds*. M. C. C. Holmes. Chem. Soc. Trans., 1918, 113, 71—79.

ON saturating an ethereal solution of mercuric chloride with ammonia, the resulting precipitate has a composition approximating to that of "fusible precipitate,"  $\text{HgCl}_2 \cdot 2\text{NH}_3$ . By digesting "infusible precipitate,"  $\text{HgCl}_2\text{NH}_3$ , at  $100^\circ\text{C}$ . with a solution of mercuric chloride (120 to 130%) in a saturated solution of ammonium chloride, filtering the hot liquid from the undissolved precipitate, and allowing it to cool slowly, crystals with the composition  $3\text{HgCl}_2 \cdot 2\text{NH}_3$  are obtained. It is improbable that any stable compound intermediate between this and "fusible precipitate" exists. A repetition of Strömholm's work (Z. anorg. Chem., 1908, 57, 72) has confirmed the existence of the compound,  $\text{Hg}_2\text{Cl}_3\text{NH}_3$ , described by him.—C. A. M.

*Phosphorus; Black* —. A. Smits, G. Meyer, and R. T. Beck. Proc. k. Akad. Wetensch., Amsterdam, 1918, 20, 392—393.

THE further investigation of the relation of Bridgman's black phosphorus (this J., 1916, 838) to the violet modification has shown that the black form is metastable in reference to the violet at temperatures between  $380^\circ$  and  $480^\circ\text{C}$ . Even in presence of iodine, which acts as catalyst, the transformation of black into violet phosphorus takes place, however, very slowly at these temperatures. (See also J. Chem. Soc., March, 1918.)—H. M. D.

#### PATENTS.

*Oralic acid; Process for the production of* —. C. T. Thorssell and H. L. R. Lunden, Gottenborg, Sweden. U.S. Pat. 1,251,938, Jan. 1, 1918. Date of appl., Aug. 13, 1915.

A MIXTURE of sodium oxalate, sodium carbonate, and sodium hydroxide is treated with a solution containing sodium bisulphate and sulphuric acid so as to form acid sodium oxalate and sodium sulphate. A portion of the acid sodium oxalate is separated by crystallising, and the remainder is recovered by neutralising the solution and converting it into an insoluble oxalate.—A. B. S.

*Hydrosulphite; Manufacture of anhydrous sodium* —. E. F. and H. W. Ehrhardt, Birmingham. Eng. Pat. 112,491, Jan. 5, 1917. (Appl. No. 266 of 1917.)

IN Eng. Pat. 8816 of 1905 (this J., 1905, 673), a paste of sodium hydrosulphite (containing water of crystallisation) is heated with a saturated salt solution to  $50^\circ$ – $70^\circ\text{C}$ . and filtered. A further amount of sodium hydrosulphite (containing water of crystallisation) is obtained by saturating the hot filtrate with salt and then cooling; the paste of crystals obtained is treated as in the patent mentioned.—F. W. A.

*Magnesium compounds [chloride]; Process of extracting — from magnesium-bearing rocks*. S. Peacock, Assignor to Bridgewater Chemical Co., Philadelphia. U.S. Pat. 1,250,216, Dec. 18, 1917. Date of appl., Nov. 28, 1916.

A MIXTURE of finely divided magnesium silicate rock with excess of calcium chloride in aqueous solution is heated under a pressure exceeding three atmospheres to produce a solution of magnesium chloride.—W. E. F. P.



*Ferric sulphate and sodium sulphate; Process of producing fused* —. H. B. Kipper, Muskegon, Mich. U.S. Pat. 1,250,471, Dec. 18, 1917. Date of appl., Oct. 26, 1916.

A MIXTURE of ferric oxide and nitre cake is heated to about 400° C. in a horizontal, cylindrical, rotary chamber fired internally with producer gas.  
—W. E. F. P.

*Oxide and hydroxide of barium; Process of making* —. B. Peacock, Philadelphia, Pa., Assignor to Larrowe Construction Co., Detroit, Mich. U.S. Pat. 1,250,642, Dec. 18, 1917. Date of appl., May 27, 1916.

A MIXTURE of barium carbonate and calcium oxide is heated until most of the carbon dioxide is expelled, and then treated with water to produce a solution of barium hydroxide. The insoluble residue is heated with a subsequent charge of mixture.—W. E. F. P.

*Aluminium nitride; Production of* —. P. R. Hershman, Assignor to Armour Fertilizer Works, Chicago. U.S. Pat. 1,250,874, Dec. 18, 1917. Date of appl., Dec. 22, 1914.

A CHARGE containing aluminiferous material, contained in a closed chamber, is heated by and in contact with producer gas, the latter being preheated to a high temperature.—W. E. F. P.

*Cooling apparatus for alkaline aluminate solutions.* R. S. Sherwin, East St. Louis, Ill., Assignor to Aluminum Co. of America, Pittsburgh, Pa. U.S. Pat. 1,251,295, Dec. 25, 1917. Date of appl., July 16, 1913. Renewed Apr. 17, 1917.

IN a circulatory system, the lower part of a deep container is connected by means of a suction device with a vertical, external pipe having two valved branches which enter the container at different points above the normal level of the liquid in the vessel. The upper branch terminates in a sprayer and is disposed at some distance above the normal level in order to effect air-cooling of the descending spray; liquid from the lower branch is returned to the container at a point only slightly above the normal level.—W. E. F. P.

*Phosphate materials; Process of treating* —. H. Blumenberg, jun., Assignor to C. Allen, Los Angeles, Cal. U.S. Pat. 1,251,741, Jan. 1, 1918. Date of appl., June 7, 1917.

FINELY divided tricalcium phosphate is treated with sulphur dioxide in the presence of water to produce calcium sulphite and phosphoric acid.  
—W. E. F. P.

*Iron sulphide; Method of manufacturing* —. N. G. Petinot, Assignor to United States Alloys Corporation, New York. U.S. Pat. 1,252,024, Jan. 1, 1918. Date of appl., Mar. 7, 1917.

IRON sulphide is produced by heating a mixture of iron pyrites and iron oxide, with or without metallic iron.—W. E. F. P.

*Method of treating liquids to remove dissolved salts.* U.S. Pat. 1,257,486. See I.

*Process for purifying clay and other silicious and aluminous earths and ores. Process for purifying aluminous ores, earths, clays, and sand.* U.S. Pats. 1,251,057 and 1,251,058. See VIII.

*Process for producing cement and potassium compounds.* U.S. Pat. 1,250,291. See IX.

*Method and apparatus for recovering volatile metallic values in the form of metallic oxides from molten slag.* U.S. Pat. 1,250,261. See X.

*Process for reclamation of bichromates [from spent tanning liquors].* Eng. Pat. 105,222. See XV.

## VIII.—GLASS; CERAMICS.

*Action of dilute sulphuric acid upon a common glass carboy.* Olle. See VII.

### PATENTS.

*Glass tank.* H. G. Slingluff, Mount Vernon, Ohio, Assignor to Pittsburgh Plate Glass Co., Pittsburgh, Pa. U.S. Pat. 1,251,931, Jan. 1, 1918. Date of appl., Jan. 20, 1914.

A GLASS-MELTING tank, heated regeneratively at one end, supplies the glass to a series of open drawing-tanks at the other end. Deflecting walls projecting from opposite sides of the tank in front of the drawing tanks nearest the heaters prevent the glass from the heated end of the tank from flowing directly into the drawing tanks.—A. B. S.

*Glass-drawing apparatus.* H. G. Slingluff, Mount Vernon, Ohio, Assignor to Pittsburgh Plate Glass Co., Pittsburgh, Pa. U.S. Pat. 1,251,932, Jan. 1, 1918. Date of appl., May 16, 1916.

A GLASS-DRAWING apparatus partly immersed in the molten glass in a glass-tank, is provided with a block of refractory material having an elongated drawing opening, and with separate blocks moulded at its ends, provided with grooved drawing ledges which lie beneath the edges of the sheet being drawn, whereby a flow of air or molten glass may be directed against the sides of the sheet as it is being drawn.—A. B. S.

*Clay and other silicious and aluminous earths and ores; Process for purifying* —. *Process for purifying aluminous ores, earths, clays, and sand.* F. Langford, Eureka, Cal. U.S. Pats. (A) 1,251,057 and (B) 1,251,058, Dec. 25, 1917. Dates of appl., Oct. 12 and Nov. 22, 1916.

(A) FERRUGINOUS clays and other aluminous and silicious materials containing iron are washed with a solution containing tannic acid to remove iron compounds, etc. The insoluble residue is treated with a mineral acid, leaving a second insoluble residue rich in silica. (B) Ferruginous clays and other aluminous materials are treated with hot dilute sulphuric acid containing less than 1% of sulphuric acid, which dissolves the iron compounds and impurities with a minimum action on the aluminous compounds.—A. B. S.

*Tunnel kiln.* J. B. Owens, Metuchen, N.J. U.S. Pat. 1,251,273, Dec. 25, 1917. Date of appl., June 4, 1917.

AN air-circulating space is formed by building a wall along each side of the interior of a tunnel kiln beyond the point of maximum temperature. Air enters this space through an opening in the top of the kiln, and after being heated by radiation from the cooling goods, passes to the furnace and is used for the combustion of the fuel.—A. B. S.

*Ceramic mouldable composition.* W. M. W. Horn, Teplitz, Austria. U.S. Pat. 1,251,888, Jan. 1, 1918. Date of appl., June 9, 1913.

THE material consists of an intimate mixture of equal parts of finely powdered, burnt fireclay and lean clay, low in quartz, lime, magnesia, and alkalis. It contains a high proportion of alumina and is capable, when moistened, moulded, and baked, of producing finely porous, strong articles.  
—A. B. S.

*Enamels for enamelling iron.* R. Koepp und Co., Oestrich, Germany. Eng. Pat. 100,777, Feb. 18, 1916. (Appl. No. 2437 of 1916.) Under Int. Conv., July 1, 1915.

IN order to reduce the cost of enamels for ironware, the expensive borax and boric acid and the felspar are replaced by a soft glass rich in alkali which is melted along with other constituents. If



necessary, clay is added to replace the alumina contained in the felspar. Quartz may also be replaced by glass if clay is added to the batch to counteract the inferior spreading power which results when an enamel contains too much glass. The following mixtures are mentioned:—Ruby glass containing 10–12% of boric acid, 60 parts, clay 16 parts, cryolite 12 parts, and sodium carbonate 7 parts, for a white enamel.  $1\frac{1}{2}$  parts of a melted mixture of glass 80 parts, and sodium carbonate, 40 parts, may be used to replace 1 part of borax and 1 part of quartz. Two parts of a frit made by melting glass 80 parts, sodium carbonate 40 parts, borax 30 parts, and clay 40 parts, may be used to replace 1 part of borax, 1 part of quartz, and  $\frac{1}{2}$  part of felspar. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4055 of 1875, 15,200 and 15,788 of 1886 and 17,878 of 1889.)

—A. B. S.

## IX.—BUILDING MATERIALS.

### PATENTS.

*Cement and potassium compounds; Process of producing* — C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,250,291, Dec. 18, 1917. Date of appl., Jan. 19, 1916.

FINELY divided, potassium-bearing, silicate rock is sprayed with sufficient alkaline-earth chloride solution to convert the whole of the potassium into chloride, and the mixture is caused to traverse an inclined, cylindrical chamber against a counter-current of flame and combustion gases, the bulk of the potassium being volatilised and recovered as chloride. The non-volatile residue is then mixed with suitable calcareous material to form a Portland cement raw mix, and the mixture heated to produce cement clinker. The volatilising point of the alkaline-earth chloride employed is materially above that of potassium chloride.—W. E. F. P.

*Plaster or cement composition and method of making the same. Plaster composition.* H. S. Thatcher, Assignor to Celite Products Co., Los Angeles, Cal. U.S. Pats. (A) 1,251,841 and (B) 1,251,842. Jan. 1, 1918. Dates of appl., June 17 and Aug. 8, 1916.

(A) CALCINED gypsum and porous, non-crystalline silica (uncalcined kieselguhr) are ground together to form an intimate mixture. (B) Calcined gypsum and uncalcined kieselguhr, both in a state of fine division, are mixed together in a dry condition.

W. E. F. P.

*Cement; Manufacture of acid- and water-proof* — C. C. Meigs, Assignor to Electro-Chemical Supply and Engineering Co., Philadelphia, Pa. U.S. Pat. 1,252,013, Jan. 1, 1918. Date of appl., Jan. 3, 1916.

IN the manufacture of acid- and water-proof cement from silicious material and sodium silicate, the silicious material is first mixed with a chloride which, when the silicate is subsequently added, is acted upon by the water in the latter so as to liberate hydrochloric acid. The hardness and density of the cement are thereby increased.

—W. F. F.

*Cement; Manufacture of building* — A. H. Hambloch, Andernach, Germany, Assignor to H. F. Boersma, The Hague, Netherlands. U.S. Pat. 1,252,204, Jan. 1, 1918. Date of appl., Oct. 6, 1913.

SEE Fr. Pat. 449,642 of 1912; this J., 1913, 489.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron; A criterion for allotropic transformations of — at high temperatures.* K. Honda. Sci. Rep. Tôhoku Imp. Univ., 1917, 6, 213–217.

THE physical properties of homogeneous substances change gradually with the temperature, while those of a substance undergoing an allotropic change vary more or less abruptly within the transformation range. At first sight, the difference between the two changes would seem to be one of degree, and hence, in an extreme case of discontinuous change, although the transformation is undoubtedly allotropic, if the change is gradual, its nature is difficult to decide. There is, however, one important difference, namely, that in non-allotropic changes the physical properties are a function of the temperature; whilst an allotropic transformation may, at a certain rate of heating, take place over an interval, yet if a suitable constant temperature be maintained, the transformation will take place at that temperature. An allotropic transformation may then be defined as a passage from one phase to another which takes place at a definite temperature if sufficient time is allowed for the transformation. In the case of pure iron, the changes  $A_3$  and  $A_4$  conform to this definition:  $A_3$  takes place at  $898^\circ\text{C}$ ., the difference between  $A_3$  and  $A_2$  not exceeding  $5^\circ$ ;  $A_4$  is more rapid than  $A_3$ .  $A_2$  proceeds over an interval of temperature, and is not an allotropic change. Carbon steels show two additional changes,  $A_0$  at  $215^\circ\text{C}$ ., and  $A_1$  at about  $700^\circ\text{--}750^\circ\text{C}$ . The latter is a phase-change, i.e., eutectic transformation of cementite and ferrite, while  $A_0$  is of the same nature as  $A_2$ , i.e., an internal change every stage of which is a function of the temperature.

—W. R. S.

*Steel; Thermal expansion of — at high temperatures.* K. Honda. Sci. Rep. Tôhoku Imp. Univ., 1917, 6, 203–212.

THE coefficient of expansion of carbon steels was found to be a function of the carbon content, apparent irregularities being due to manganese which, like carbon, reduces the coefficient. The following values are given for  $15^\circ\text{--}75^\circ\text{C}$ .:—Swedish iron (C 0.04%),  $1.266 \times 10^{-6}$ ; 0.75% carbon steel,  $1.155 \times 10^{-6}$ ; 1.50% carbon steel,  $1.083 \times 10^{-6}$ . The expansion at higher temperatures (to about  $900^\circ\text{C}$ .) is represented graphically, the curves showing hysteresis over the transformation range. The maximum contractions due to the  $A_1$  and  $A_2$  changes occurred in a 0.31% carbon steel. The coefficient of expansion increased rapidly with the temperature, but showed a slight decrease through the transformation range.

—W. R. S.

*Steels; Thermal and electrical conductivities of carbon — at high temperatures.* K. Honda and T. Simidu. Sci. Rep. Tôhoku Imp. Univ., 1917, 6, 219–233.

THE experiments were made with pure Swedish iron and carbon steels containing respectively 0.18, 0.44, 0.64, 0.80, 1.02, 1.30, and 1.50% C. The thermal conductivity decreased slowly at first, then somewhat rapidly, with increasing temperature. The electrical conductivity decreased at a gradually increasing rate up to the critical temperature, the curve being slightly convex with respect to the temperature axis. In the region  $A_1$  the resistance increased abruptly during the range  $A_1$ , and decreased during  $A_2$ ; the difference between  $A_1$  and  $A_2$  was  $50^\circ\text{--}60^\circ\text{C}$ . The irregularity was greatest in 0.8% carbon steel. The product of thermal conductivity and electric resistance was fairly constant for the different specimens (Wiedemann-Franz's law),



but not proportional to the absolute temperature except for the pure Swedish iron and steel containing up to 0.44% carbon (Lorenz's law).

—W. R. S.

*Iron and iron-nickel alloys; Cathodic deposits of — obtained at ordinary temperatures under high hydrogen pressure. Electrolytic deposition of alloys and their metallographic and mechanical investigation. VIII. R. Kremann and H. Breymesser. Monatsh. Chem., 1917, 38, 359—384.*

WHEN iron is deposited electrolytically under ordinary conditions it is charged with hydrogen and admixed with ferric oxide; it is brittle and hard. These conditions are due to the simultaneous liberation of hydrogen at the cathode. It is shown that elevation of the temperature, increase of the acidity or iron concentration of the bath, and increase of the current density accentuate these defects, but that if the electrolysis takes place in neutral solutions under a high pressure of hydrogen, the nature of the deposit is very much improved. To test these theoretical conclusions, a  $N/1$  solution of ferrous sulphate was electrolysed by a current density of 0.25—0.75 amp. per sq. dm. under a pressure of 20 atmos. of hydrogen and the deposits compared with those obtained from solutions containing respectively citric acid and sulphuric acid, and electrolysed under 1 atm. pressure. The current efficiency at the higher pressure is 99.33—99.46%; the deposit contains less hydrogen, it is less brittle, composed of larger crystals, and is softer. It at once gives the equilibrium potential when immersed in  $N/1$  ferrous sulphate solution. Similar experiments with mixed ferrous and nickel sulphate solutions gave nickel-iron alloys which did not show the improvement in the deposit shown in the case of iron. Photomicrographs of the deposits are given. (See also *J. Chem. Soc.*, March, 1918.)—J. F. S.

*Copper; New method of determining —. J. Moir. J. Chem. Met. Soc., S. Afr., 1917, 18, 133—135.*

A WEIGHED quantity of the sample of copper or copper alloy is dissolved in strong nitric acid, and the solution shaken for 1 to 2 mins. to expel nitrogen peroxide, and then diluted to 20 c.c., treated with 0.5 gm. of urea, diluted to 50 c.c., and boiled for 15 secs. to remove all nitrous acid. The liquid, which is then no longer an oxidising agent, is nearly neutralised with sodium hydroxide, and treated with 2 to 3 grms. of sodium acetate, followed by a measured slight excess of standard sodium thiosulphate solution (3.904 grms. = 1 gm. of copper), and, immediately afterwards, by a sufficient excess (1.75 grms. per 1 gm. of copper) of potassium thiocyanate. The white precipitate of cuprous thiocyanate is filtered off by means of a filter of disintegrated paper pulp, roughly washed, and rejected. The filtrate is diluted to about 1 litre, treated with 5 to 10 c.c. of pure sulphuric acid, and the excess of thiosulphate titrated with  $N/10$  iodine solution, with starch as indicator. It is essential that a large excess of thiosulphate should not be used, or the amount of copper found will be too high (102 to 105% for pure copper). None of the common metals, with the exception of iron, interferes with the reaction.—C. A. M.

*Brass; Thermal expansion of  $\alpha$  and  $\beta$  — between 0° and 600° C. in relation to the mechanical properties of heterogeneous brasses of the Muntz metal type. P. D. Merica and L. W. Schad. J. Amer. Inst. Metals, 1917, 11, 396—407.*

IN an investigation into cases of fracture of 60 : 40 brass, it was ascertained that where failure could not be explained by known causes, the articles had been subjected at some time to a very rapid

cooling or quenching. Since such brass is heterogeneous, consisting of about equal proportions of  $\alpha$  and  $\beta$ , it was thought that local stresses, due to unequal thermal contraction of the two constituents, might be developed by rapid cooling. By measuring the thermal expansion of sample ingots suitably heat-treated so as to produce homogeneous structure, it was observed that from 0° to about 300° C. the expansions of  $\alpha$  and  $\beta$ -brass were almost equal; from 300° to 460° C. the expansion of  $\beta$  was almost twice that of  $\alpha$ . Above the transformation temperature the expansion curve of  $\beta$  was almost linear and parallel to that of  $\alpha$ . Hence the stresses during the cooling of 60 : 40 brass are greatest during the interval 500°—300° C. It may therefore be assumed that during slow cooling the constituents yield locally but when quenched they are in a state of stress at the ordinary temperature. The effect of quenching on the mechanical properties of test-pieces was not very apparent, except a lowering of the proportional limit by about 2000 lb. per sq. in.

—W. R. S.

*Mercury still; Silica-glass —. J. C. Hostetter and R. B. Sosman. J. Wash. Acad. Sci., 1918, 8, 11—15.*

THE use of silica-glass for a vacuum still obviates the risk of breakage to which glass stills are liable when overheated. In the still shown in the

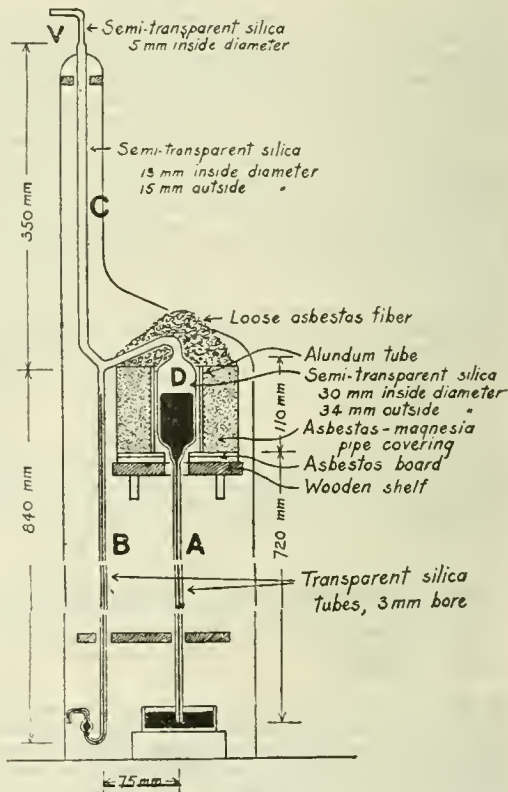


diagram the distillation chamber, D, and the condenser, C, are made of semi-transparent silica-glass, whilst the vertical tubes, A and B, are of transparent silica-glass. At V the still is connected with a suction pump, the joint being made with a hard cement, at a point where it will be cooled as completely as possible by the air. The resistance wire for heating the furnace is wound directly upon an alundum tube, and held in position by means of alundum cement. An



effective method of purifying the mercury is to shake it with cane sugar, and then to filter it through a pinhole in a filter paper.—C. A. M.

*Colloidal gold and platinum; Precipitation of — on metallic surfaces.* E. B. Spear and K. D. Kahn. J. Amer. Chem. Soc., 1918, 40, 181—184.

COLLOIDAL solutions of gold and platinum are coagulated when metal plates of various kinds are brought into contact with the solutions. The rate at which coagulation occurs increases with the electro-positive character of the metal. Plates of zinc, steel, nickel, lead, and copper decrease in activity in the order given. The effect depends also on the physical character of the surface, a rough surface being more active than a polished surface of the same metal. (See also J. Chem. Soc., March, 1918.)—H. M. D.

*Amorphous metal hypothesis: The — and equi-cohesive temperatures.* Z. Jeffries. J. Amer. Inst. Metals, 1917, 11, 300—323.

FINE-GRAINED metals are superior to coarse-grained in mechanical properties, and, up to the present, Beilby's amorphous cement theory accounts for this behaviour in the most satisfactory manner. At ordinary temperatures the amorphous metal cement is much harder than the crystalline phase, and, since the grain boundary surface increases as the grain size decreases, there will be more amorphous material in a fine-grained metal, hence greater cohesion. Additional amorphous metal is believed to be generated along the planes of slip and the grain boundaries during plastic deformation. According to the theory, the amorphous phase is more cohesive than the crystalline phase at ordinary temperature, but at high temperatures the reverse is the case. If so, there must be an intermediate temperature at which the cohesion of the amorphous and crystalline phases is the same; this is called the equi-cohesive temperature. Above this, the coarse-grained metal offers greater resistance to permanent deformation, and *vice versa*. The existence of the equi-cohesive temperature was proved by experiment. Fine wires of various metals were annealed in such a manner as to produce a fine-grained structure in one set, and a coarse-grained structure in another; the wires were examined microscopically to make certain that the desired grain size had been obtained. The resistance of the wires to bending was determined at different temperatures, by measuring the deformation caused by a 0.2 grin. rider. At low temperature the fine-grained wires proved stronger, and at high temperature weaker, than the coarse-grained; the equi-cohesive temperatures were found to be: iron, 550°—600° C.; tungsten, 1350° C.; silver, 250°—275° C.; gold, 275°—300° C.; platinum, 525°—550° C. These temperatures correspond closely with the recrystallisation or "equi-axing" temperatures of these metals after cold working.—W. R. S.

*Temperature determinations by eutectic alloys.* C. P. Steinmetz. J. Amer. Chem. Soc., 1918, 40, 96—100.

IN order to measure the maximum temperatures existing locally at the rim of steel discs revolving at such speeds as to prevent the attachment of any temperature-measuring device, it has been proposed that small holes should be drilled into the rim of the rotating disc and that these should be filled with eutectic alloys melting at different temperatures. The alloys which melt will be ejected by centrifugal action and the required temperature may thus be approximately determined. In this connection, the melting points of some twelve eutectic alloys melting between 69.5° and 194° C. have been investigated. The following are the alloys with their respective

melting points:—Bi 15, Cd 3, Pb 8, Sn 4 (Wood's metal), 69.5°; Bi 7, Cd 1, Pb 6, 90.0° C.; Bi 2, Pb 1, Sn 1 (Rose metal), 93.5° C.; Bi 11, Cd 4, Sn 5, 102.4° C.; Bi 11, Pb 9, 132.0° C.; Bi 2, Sn 1, 132.5° C.; Bi 2, Cd 1, 143.5° C.; Cd 2, Pb 5, Sn 3 (cadmium-solder), 144.8° C.; Sn 4, Ti 3, 166.5° C.; Cd 1, Sn 2, 172.0° C.; Pb 3, Sn 5 (tin solder), 178.3° C.; Sn 10, Zn 1, 194.0° C. It is pointed out that the use of eutectic alloys affords a much closer scale of reference numbers than could possibly be attained by the use of pure metals. (See J. Chem. Soc., March, 1918.)—H. M. D.

#### PATENTS.

*Cast iron and other metals; Flux for brazing —* H. A. Gill, London. From J. H. Nathan, St. Kilda, Victoria, Australia. Eng. Pat. 112,500, Jan. 9, 1917. (Appl. No. 447 of 1917.)

A MIXTURE of sodium baborate 38% (by weight), washing soda 19%, sodium chloride 38%, soda ash 5%, with or without gold chloride 0.04%. —T. H. B.

*Magnetic material [iron or iron alloys] for use in static transformers and other electrical apparatus.* A. F. Berry, London. Eng. Pat. 112,649, Jan. 13, 1917. (Appl. No. 686 of 1917.)

MAGNETIC material (iron or an iron alloy) in sheet or strip form is coated, by suitable heating, with a thin, firmly adherent layer of iron oxide having high electrical resistance, and is then passed while in the heated condition, first through grooving rolls, to form longitudinal grooves extending nearly through the thickness of the sheet, with intervening parallel ribs of metal, then through flattening rolls to close the grooves to lock the oxide film and prevent subsequent detachment. The resulting sheet or strip has a greater electrical resistance in one direction than in another.—T. H. B.

*[Steel] alloy.* J. Churchward, Lakeville, Conn. U.S. Pat. 1,251,341, Dec. 25, 1917. Date of appl., Aug. 8, 1917.

A STEEL alloy contains Ni 1 to 5%, Cr 0.25 to 0.50%, and Mn 0.15 to 0.50%, together with titanium and "an element of the nature of molybdenum"; or Ni 1—3%, Mo 0.25 to 3%, Cr 0.25—1.5%, Ti trace to 0.50%, and Mn 0.15—0.50%. —T. H. B.

*Iron or steel; Method of treating —.* N. G. Petinot, Assignor to United States Alloys Corporation, New York. U.S. Pat. 1,252,023, Jan. 1, 1918. Date of appl., Mar. 7, 1917.

AN alloy of iron, titanium, and silicon is added to molten iron or steel. The alloy contains from 69 to 80% of iron, and the ratio of silicon to titanium is about 1 to 4, so that on oxidation a silicotitanate of low melting point is produced.—W. E. F. P.

*Metals; Devices for the pulverisation of molten or liquid —.* A. Cusquel and H. Goupil, Levallois-Perret, France. Eng. Pats. (A) 106,095 and (B) 112,065, Apr. 24 and 30, 1917. (Appl. Nos. 5782 and 6111 of 1917.) Under Int. Conv., May 1, 1916.

(A) IN a pulveriser for powdering metals having a melting point below 800° C., *e.g.*, tin, lead, antimony, zinc, and particularly aluminium, the molten metal is supplied to a tube having a nozzle controlled by a needle-valve, which is fitted at its rear end with a threaded portion and a stuffing-gland and is moved by an external turn-button. Air or gas under pressure is supplied to an annular chamber around the nozzle, having an annular outlet adjacent to the outlet for the metal. The annular chamber comprises a fixed rear plate, threaded on its periphery, and a nozzle screwing on to the plate so as to adjust the central outlet for the gas. In a modification, the nozzle may be



surrounded by an additional annular chamber, having a number of outlets in the front plate converging to a point on the axis of the jet and supplied with pressure gas, so as to draw the metallic powder away from the hot nozzle by suction. (B) In the apparatus first described in (A), a supplementary air chamber is provided at the rear of the adjustable annular air chamber, the common wall being perforated. The perforations have a total cross-section less than the section of the supply conduit, so as to minimise fluctuations of the air jet.—W. F. F.

*Zinc alloys.* F. Maas, Berlin. Eng. Pat. 112,487, Dec. 28, 1916. (Appl. No. 18,565 of 1916.)

ALLOYS consisting essentially of zinc, copper, and aluminium are made by first melting the copper, then adding aluminium in the amount required in the finished alloy, and allowing the mixture to cool to about 700°–800° C.; zinc, previously heated, is then added in the proportion of one-half of the amount required for the finished alloy, and the mixture allowed to cool to about 500° C., when the remainder of the zinc is added in a molten condition. For 100 parts of zinc, less than 4 parts of copper is employed, and the whole content of copper plus aluminium is between 7 and 8.5%.—T. H. B.

*Copper; Electro-deposition of* —. S. O. Cowper-Coles, Sunbury-on-Thames, Middlesex. Eng. Pat. 112,516, Jan. 18, 1917. (Appl. No. 935 of 1917.)

THE cathodes are mounted vertically in a vat and are caused simultaneously to rotate and to reciprocate in a vertical direction through a distance of several inches, one complete up-and-down displacement being effected while the cathode makes two or three revolutions. The peripheral speed of the cathode is about 50 ft. per minute. The electrolyte is caused to travel in a direction opposite, or at right angles, to that in which the cathode revolves, by squirting it through a number of jets or by blowing air through jets into the electrolyte.—T. H. B.

*Melting furnaces.* I. Hall, Birmingham. Eng. Pat. 112,559, Apr. 13, 1917. (Appl. No. 5216 of 1917.)

A MELTING furnace particularly suitable for melting aluminium and its alloys comprises a metallic melting pot supported at its upper part, a combustion chamber below the pot, and an injector box communicating tangentially with the combustion chamber. The casing enclosing the melting pot has a refractory lining following the contour of the pot. The whole is mounted on trunnions, so that it can be tilted. Over the melting pot is a cover, with or without external flues whereby the products of combustion can be conveyed into the chamber above the melting pot. A perforated plate, adapted to move vertically in the pot, is used for mixing purposes, to ensure homogeneity.—T. H. B.

*Blast furnace bosh.* W. J. Foster, Walsall, Staffs. Eng. Pat. 112,658, Jan. 17, 1917. (Appl. No. 844 of 1917.)

THE bosh is built up of bricks or blocks consisting of a mixture of carbonaceous material such as plumbago, or very finely ground hard coke, with Stourbridge or porcelain clay. The bosh walls are enclosed by a metal casing between which and the outer ends of the blocks a mixture similar to that composing the blocks is applied to form a sealing layer between the casing and the blocks, and also a key. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 13,690 of 1891 and 12,390 of 1912; this J., 1913, 794.)—T. H. B.

*Volatile metallic values; Method of and apparatus for recovering* —, in the form of metallic oxides, from molten slag. H. Witteborg, Caldwell, Idaho. U.S. Pat. 1,250,261, Dec. 18, 1917. Date of appl., Mar. 6, 1917.

THE molten slag, containing a suitable flux and reducing agent, is caused to flow in a comparatively shallow stream through a series of closed, superheating retorts while subjected to the action of blasts of flame and hot combustion gases. Means are provided for oxidising the metallic vapours produced and for collecting the resulting oxides.—W. E. F. P.

*Flotation apparatus.* W. E. Greenawalt, Denver, Colo. U.S. Pat. 1,250,303, Dec. 18, 1917. Date of appl., Dec. 4, 1915.

A TANK containing liquid has an outlet for tailings in its lower portion and a froth overflow at the top. A series of horizontal pipes is suspended in the tank, submerged in the liquid, the pipes extending transversely across the tank, and being surrounded by porous material. The ore is fed into the upper part of the tank and sinks through the liquid, and a gas is supplied to the pipes and escapes through the porous material into the liquid, the volume and pressure of the supply to the respective pipes being varied so as to produce a selective action on the ore from one end of the series of pipes to the other. The pipes are reciprocated transversely of their longitudinal axes and across the path of sinking particles.—T. H. B.

*Ore-roaster; Blast* —. G. H. Deru, Salt Lake City, and T. P. Holt, Silver City, Utah, Assignors to Holt-Christensen Process Co., Provo, Utah. U.S. Pat. 1,251,189, Dec. 25, 1917. Date of appl., Aug. 25, 1916.

A CHARGE holder supported by a built-up bin has a grate made up of independently movable sections; each section is composed of sub-sections adapted to operate in opposite directions, and each complete section can be operated independently of any other section. An air blast is led into the upper portion of the bin and upwards through the grate, and parts of the charge may be dropped into the bin for cooling purposes, remaining there without interference with the blast. A smoke-flue is supported transversely across the charge holder and ore is fed in on either side of the smoke-flue.—T. H. B.

*Lead; Process of treating sulphide ores of* —. R. De Luce, Oakland, Cal. U.S. Pat. 1,251,485, Jan. 1, 1918. Date of appl., May 8, 1917.

SULPHIDE ores of lead containing silver or copper are decomposed by a solution containing ferric chloride, and the residue is leached with an electrically conducting solution which is a solvent of lead chloride but is free from ferric chloride. The two solutions are electrolysed in the anode and cathode compartments respectively of an electrolytic cell having a porous diaphragm. Lead is deposited and the solutions regenerated for the decomposition and leaching of a fresh charge of ore.—W. F. F.

*Solder.* J. R. Kinder, jun., Ladd, Ill. U.S. Pat. 1,252,010, Jan. 1, 1918. Date of appl., May 31, 1917.

THE solder consists of lead 35.7%, pure zinc 24.5%, and tin 39.8%.—W. F. F.

*Alloy; Dental* —. H. K. Sandell, Assignor to H. S. Mills, Chicago, Ill. U.S. Pat. 1,252,038, Jan. 1, 1918. Date of appl., June 18, 1917.

NICKEL is added to molten tungsten and the resulting alloy incorporated with molten gold, the

proportion being 80 parts by weight of gold, 10 of tungsten, 7 of nickel, and a relatively small proportion of carbon.—T. H. B.

*Metal reflectors, mirrors, and other metal articles having a reflecting surface or surfaces; Manufacture of —.* T. Hancock, London. Eng. Pat. 112,881, May 16, 1917. (Appl. No. 6977 of 1917.)

*Steel ingots; Casting —.* H. H. Ashdown, Assignor to Sir W. G. Armstrong, Whitworth and Co., Newcastle-upon-Tyne. U.S. Pat. 1,251,951, Jan. 1, 1918. Date of appl., June 18, 1917.

SEE Eng. Pat. 105,617 of 1916; this J., 1917, 653.

*Process for purifying clay and other silicious and aluminous earths and ores. Process for purifying aluminous ores, earths, clays, and sand.* U.S. Pats. 1,251,057 and 1,251,058. See VIII.

## XI.—ELECTRO-CHEMISTRY.

*Lead standard cell and determination of the potential of the lead electrode.* W. E. Henderson and G. Stegeman. J. Amer. Chem. Soc., 1918, 40, 81—89.

THE cell represented by Pb amalgam—PbSO<sub>4</sub>—Na<sub>2</sub>SO<sub>4</sub>.10H<sub>2</sub>O—Hg<sub>2</sub>SO<sub>4</sub>—Hg with an amalgam containing from 2.5 to 6% Pb gives a constant and reproducible electromotive force, which, for temperatures between 18° and 30° C., is given by the equation  $E = 0.96463 + 0.000174(t - 25) + 0.00000038(t - 25)^2$ . Like many other cells of this type, the E.M.F. shows a small gradual decrease over an extended period of time. (See also J. Chem. Soc., March, 1918.)—H. M. D.

*Electrolysis of solutions of the rare earths.* Dennis and Ray. See VII.

### PATENTS.

*Electrolytic cell.* H. C. Jenkins, London. U.S. Pat. 1,250,183, Dec. 18, 1917. Date of appl., Nov. 22, 1916.

SEE Eng. Pat. 16,643 of 1915; this J., 1917, 31.

*Diaphragm for electrolytical and electro-osmotic purposes.* B. Schwerin, Assignor to Ges. für Elektro-Osmose m.b.H., Frankfurt, Germany. U.S. Pat. 1,252,185, Jan. 1, 1918. Date of appl., Jan. 4, 1913.

SEE Addition of June 22, 1912, to Fr. Pat. 426,072 of 1911; this J., 1912, 1180.

*Process and apparatus for separation of suspended particles from gases.* U.S. Pat. 1,250,088. See I.

*Baking-furnace [for carbons, etc.].* U.S. Pat. 1,251,309. See I.

*Method and apparatus for fractionating hydrocarbons.* U.S. Pat. 1,250,879. See IIA.

*Magnetic material [iron or iron alloys] for use in static transformers and other electrical apparatus.* Eng. Pat. 112,649. See X.

## XII.—FATS; OILS; WAXES.

*Fats; Saponification of —.* II. J. P. Treub. Proc. k. Akad. Wetensch. Amsterdam, 1918, 20, 343—357.

A STUDY of the hydrolysis of trilaurin by sulphuric acid confirms the theory that in the case of saponification in stages the relative concentration of glycerol at any moment is equal to the third power of the relative concentration of the free

fatty acid, provided that the ester groups are equivalent. (See also J. Chem. Soc., March, 1918.) —W. G.

*Studies of drying oils. Properties of some cerium salts obtained from drying oils.* Morrell. See XIII.

*Formation of [lipolytic] ester-hydrolysing substances by the action of alkali on proteins.* Hulton-Frankel. See XIXA.

### PATENTS.

*Fatty acids and their glycerides; Means for adding hydrogen to non-saturated —.* A. Woltman, The Hague. Eng. Pat. 112,293, Dec. 29, 1916. (Appl. No. 18,621 of 1916.)

HYDROGEN under pressure is conducted through a perforated stirring device into the oil or fatty acid previously heated to the required temperature in presence of a catalyst. The agitator may be in the form of a horizontal hollow shaft arranged in an eccentric position and provided with hollow perforated arms, which revolve continuously in the liquid to be hydrogenated, while the outflow of gas may be in any direction relative to that in which the arms revolve.—C. A. M.

*Hydrogenation [of oils]; Manufacture of catalysts for —.* C. Ellis, Montclair, N.J. U.S. Pat. 1,251,201, Dec. 25, 1917. Date of appl., May 26, 1917.

A HYDROGENISING catalyst is obtained by heating the readily reducible soap (e.g., oleate) of a non-noble catalytic metal of atomic weight between 48 and 63.6 (e.g., nickel) in solution in an oil other than a drying oil, whereby the non-catalytic soap is decomposed and the catalyst liberated in a finely divided colloidal state in the oil.—A. DE W.

[Hydrogenation of oils;] *Catalyser-concentrate [for —] and process of making same.* C. Ellis, Montclair, N.J. U.S. Pat. 1,251,202, Dec. 25, 1917. Date of appl., Feb. 6, 1914.

A REDUCIBLE, oil-soluble, non-catalytic compound of a catalytic metal (e.g., nickel carbonyl) is incorporated in an oil not inimical to the activity of the catalyst, in a proportion materially in excess of that required to hydrogenise such oil, i.e., more than one part of nickel to 200 parts of oil. The compound is then reduced by heating at a sufficiently high temperature under superatmospheric pressure in presence of a reducing gas, whereby the catalyst is obtained in a colloidal state.

—A. DE W.

[Hydrogenation of oils;] *Process of making a catalyst [for —].* C. Ellis, Montclair, N.J. U.S. Pat. 1,251,203, Dec. 25, 1917. Date of appl., Jan. 25, 1917.

A PURE fatty acid salt of a metal which in the free state possesses catalytic activity (e.g., nickel acetate) is reduced with a gas containing hydrogen.

—A. DE W.

[Hydrogenation of oils;] *Process of making catalysts [for —].* C. Ellis, Montclair, N.J. U.S. Pat. 1,251,204, Dec. 25, 1917. Date of appl., May 1, 1917.

AN organic compound of a non-noble metal having an atomic weight between 51 and 63.6 (e.g., nickel) is heated out of contact with air (e.g., in a bath of oil) with an inorganic salt of the same metal containing nitrogen and oxygen.—A. DE W.

*Oil [from garbage]; Perculators for use in extracting — by solvents.* G. Harrison, London. From C. O. Bartlett and Snow Co., Cleveland, Ohio, U.S.A. Eng. Pat. 112,590, July 23, 1917. (Appl. No. 10,586 of 1917.)

OIL-CONTAINING material, and in particular garbage and similar refuse, is extracted with a solvent in



a drum which rotates on a horizontal axis, and is provided along a portion of its periphery with a perforated screen enclosing a space, which has valved outlet pipes and a steam or vapour discharge pipe connected with a conduit passing through the axis of the drum to a receiving chamber. To facilitate working, the manholes, the perforated strainer, and the openings of the pipes for the admission of steam, solvent, etc., are arranged on lines at distances about 90° apart on the circumference of the drum.—C. A. M.

*Solidified plastic articles [soap]; Method of rapidly ageing*—. De N. W. B. Young, Narberth, Pa., Assignor to Soap Works Equipment Co. U.S. Pat. 1,250,540, Dec. 18, 1917. Date of appl., Dec. 7, 1915.

A SOLIDIFIED plastic article having semi-crystalline, non-uniform surfaces, e.g., bar soap, is conveyed in a continuous and uninterrupted movement through a gaseous mixture so as partially to modify the surface, then through a static body of heated air of constant temperature to develop an "investing integument of jelly-like consistency," and finally through a cooling atmosphere to fix such integument, the resulting film being uniform, translucent, and "visibly free from crystalline salts."—A. DE W.

*Fatty and other matters; Method for the recovery of*—*from liquids*. T. W. Barber, London. U.S. Pat. 1,251,621, Jan. 1, 1918. Date of appl., Dec. 8, 1915.

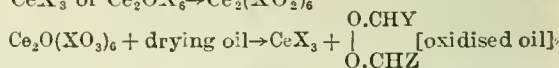
SEE Eng. Pat. 23,741 of 1914; this J., 1915, 1061.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Drying oils; Studies of*—. I. Properties of some cerium salts obtained from drying oils. R. S. Morrell. Chem. Soc. Trans., 1918, 113, 111—124.

CEROUS salts of the principal fatty acids in drying oils and of abietic acid were prepared by the interaction of a solution of a cerous salt and of a solution of the potassium salt of the respective acids. All these salts are colourless and insoluble in water. Cerous stearate, elaidate,  $\beta$ -elaeostearate, and palmitate are insoluble in ether, whilst cerous oleate, linolate, linolenate,  $\alpha$ -elaeostearate, and abietate are soluble in ether and oil of turpentine. In each case the salt is of the normal type,  $CeX_3$ . Ethereal solutions of the cerous salts of unsaturated fatty acids darken on exposure to the air, and in the case of the linolenate yield buff-coloured precipitates giving a peroxide reaction and having a composition corresponding with the formula of a basic ceric oxylinolenate  $CeO(XO_2)_2$ . The brown ethereal solution appears to contain a mixture of the salts  $CeX_3$  and  $CeX_4$ , or a basic salt,  $2CeO(XO_2)_2$ , formed by the oxidation of  $CeX_3$ . After longer exposure of the solution to the air an insoluble salt of the type  $CeO(XO)_2$  is produced. An ethereal solution of cerous oleate only darkens slightly on exposure, and becomes slightly turbid. Olive oil forms cerous salts of the normal type,  $CeX_3$ , soluble in ether, and changing but slowly into the insoluble type, but after being heated in the absence of air it yields not only the normal salt, but also a soluble salt of the  $CeX_4$  type and an insoluble basic ceric salt  $CeOX_2$ . Poppy-seed oil behaves in a similar way, whilst thickened linseed oil from which the polymerised compound has been extracted with acetone gives an increased yield (up to nearly 50%) of the basic ceric salt,  $CeOX_2$ . Raw linseed oil yields the normal salt,  $CeX_3$ , and about 12.4% of the basic ceric salt. These

facts support the author's view (this J., 1915, 105) that there is a change in the intramolecular linkings prior to polymerisation when drying oils are polymerised by heat. Cerous  $\alpha$ -elaeostearate from Chinese wood oil is readily soluble in ether, and yields an acid with the high m.pt. and high iodine value of  $\alpha$ -elaeostearic acid, but after some time the salt becomes insoluble in ether, and then yields a mixture of a crystalline acid (m.pt. 45° to 46° C.) with iodine value 177.8, and a syrupy acid with lower iodine value, which liberates iodine from potassium iodide. These results show that partial oxidation of the cerous salt readily takes place, although the ethereal solution yields no precipitate when subjected to the action of a current of air or oxygen. The presence of mixed unoxidised and oxidised acids in the insoluble salt may be attributed to the formation of an insoluble basic ceric salt of the type  $Ce_2X_6O$ , and subsequent partial oxidation of the acid. The absence of any appreciable amount of  $\beta$ -elaeostearic acid shows that stereoisomeric change does not take place. The oxygen absorption of cerous  $\alpha$ -elaeostearate, the initial stage of which takes more than 10 days, indicates that with the formation of a basic ceric  $\alpha$ -elaeostearate of the type  $Ce_2O(XO_2)_2$ , from  $CeX_3$  or  $Ce_2X_6O$ , one mol. of oxygen per mol. of acid is absorbed, so that the iodine value and iodine-liberation value become approximately equal. It is only after exposure of a year to oxygen that the second double linking of elaeostearic acid becomes peroxidised. The peroxidic acid shows gradual polymerisation until the syrupy liquid has set to a varnish. It is unstable with alkalis, but stable with acid, and is not reduced by zinc dust and acetic acid. The general conclusion drawn from these results is that the function of cerium  $\alpha$ -elaeostearate as a drier may be represented by the following scheme:—  
 $CeX_3$  or  $Ce_2O(XO_2)_2 \rightarrow Ce_2(XO_2)_4$



This representation agrees with the conclusions of Ingle concerning lead linolenate (this J., 1917, 318). Cerium linoleate from linseed oil is a more active drier than lead linolenate, but is inferior to manganese and cobalt linoleates (Mackay and Ingle, this J., 1917, 318). The addition of a cerium drier to a varnish containing a lead drier does not accelerate the time of drying in the same way as does cobalt or manganese linoleate.—C.A.M.

*Colophony; Colloidal nature of*—. II. L. Paul. Kolloid-Zeits., 1917, 21, 148—154.

WHEN certain fractions of the distillate obtained by distilling mixtures of colophony with phenol, resorcinol,  $\alpha$ - and  $\beta$ -naphthol are treated with diazo- or tetrazo-compounds, dyes are produced which are characterised by marked adhesive properties and may be fixed on the fibre without the use of mordants. The preliminary fractional distillation may be replaced by the treatment of the phenolic substance with a solution of a resin soap.—H. M. D.

#### PATENTS.

*Iron pigment; Process of making*—. A. F. Hoffman, Pittsburgh, Pa. U.S. Pat. 1,251,522, Jan. 1, 1918. Date of appl., Apr. 5, 1916.

NITRE cake and silicious material are heated together, whereby a soluble silicate is produced with evolution of sulphur trioxide and sulphuric acid vapours. A solution of ferrous sulphate is treated with this soluble silicate in such proportions that the reacting materials contain about one atom of iron to four atoms of silicon and that at least the major part of the iron is precipitated. The precipitate is separated, oxidised, and calcined till completely dehydrated.—A. DE W.

*Rosin, turpentine, and oils [from wood]; Process of extracting*—J. E. Teeple, Montclair, N.J. U.S. Pat. 1,252,058, Jan. 1, 1918. Date of appl., Jan. 12, 1914.

FINELY comminuted coniferous wood is heated to a temperature below 100° C. and subjected to the action of the mixed vapours obtained by passing steam into a bath of oil of turpentine. The vapours are condensed in such a way that the wood is exposed to the joint action of water and oil of turpentine at their boiling points until the resin and oils are sufficiently extracted. The amount of oil of turpentine used for extraction must be greater than the amount normally present in the wood.—A. DE W.

*Titanium oxide paint and pigment and process of making same*. L. E. Barton, Niagara Falls, N.Y., Assignor to The Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,251,170, Dec. 25, 1917. Date of appl., Mar. 25, 1916.

SEE Eng. Pat. 108,805 of 1917; this J., 1917, 1055.

*[Varnished] decorative surfaces; Treatment of*—G. H. Hadfield, Mitcham, and A. E. Bawtree, Sutton, Assignors to Hadfields (Merton) Ltd., Mitcham. U.S. Pat. 1,252,001, Jan. 1, 1918. Date of appl., Sept. 18, 1917.

SEE Eng. Pat. 109,657 of 1916; this J., 1917, 1139.

*Method of treating mine water containing sulphuric acid and iron salts [and obtaining a pigment]*. U.S. Pat. 1,251,561. See XIXB.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### PATENTS.

*Coagulating [rubber] latex; Process and apparatus for*—Lintner & Co., Ltd., Amsterdam, Netherlands. Eng. Pat. 108,298, Oct. 27, 1916. (Appl. No. 15,342 of 1916.) Under Int. Conv., July 27, 1916.

LATEX treated with sulphur dioxide, either gaseous or in aqueous solution, is transferred to tanks, where it coagulates after the lapse of one hour or more according to the proportion of sulphur dioxide used. An apparatus is described for use with gaseous sulphur dioxide; the gas produced by burning sulphur in a stove is led into a vessel half filled with latex and fitted with a gauge glass. During the treatment with the gas the latex is allowed to flow from the vessel and at the same rate fresh latex enters from a tank, so that the process is continuous. The stream of treated latex flows into a suitable receptacle and is thence transported to the coagulation tanks. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 16,728 and 18,062 of 1913; this J., 1914, 758.)—D. F. T.

*Rubber articles; Process of ornamenting*—F. J. Croque, Cuyahoga Falls, Ohio. U.S. Pat. 1,251,182, Dec. 25, 1917. Date of appl., July 25, 1917.

THE surface of a rubber article is distorted or wrinkled whilst on its form by immersion in a rubber solvent; when the article is subsequently dipped in a colouring solution the swollen portions absorb more of the colour than do the portions which remain tight on the form.—D. F. T.

*Rubber substitute and process of making the same*. W. W. Carter, Needham, Mass. U.S. Pats. (A) 1,251,862 and (B) 1,251,863, Jan. 1, 1918. Dates of appl., Mar. 29 and 30, 1917.

(A) A RUBBER substitute is prepared by mixing a sulphonate and a polymerisable oil together

with a substance adapted to neutralise any free acid, this substance being provided with a protective coating. The sulphonate and polymerisable oil give rise to a gelatinous product, and the protective coating on the neutralising substance prevents this from exerting its influence until after the completion of the main reaction. (B) The reaction product of a polymerisable oil, such as Chinese wood oil, with sulphonates derived from tar acids, is submitted to the action of formaldehyde or some equivalent substance; it is then washed and dried.—D. F. T.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Gelatin; Swelling of*—in polybasic acids and their salts. M. H. Fischer and M. O. Illoker. J. Amer. Chem. Soc. 1918, 40, 272—292.

THE absorption of water by gelatin was determined in different concentrations of the primary, binary, and ternary salts of phosphoric, citric, and carbonic acids, and in solutions varying from pure acid to pure alkali (sodium hydroxide) for the same three acids. The absorption is greatest in pure acids and alkali, decreasing considerably in the presence of salts, and with certain salts at high concentrations, it is less than in pure water. From a certain minimum there is a progressive increase in the absorption with increase of the acid or alkali content of the mixture. (See also J. Chem. Soc., March, 1918.)—L. A. C.

*Fibrin; Swelling of*—in polybasic acids and their salts. M. H. Fischer and M. Berzinger. J. Amer. Chem. Soc., 1918, 40, 292—303.

EXPERIMENTS in the absorption of water by fibrin in acid and alkaline solutions containing the acid salts (using phosphoric and citric acids) gave essentially the same results as when gelatin was used (see preceding abstract), with the exception that the points of minimum absorption corresponded to a different acid content in the solution. (See also J. Chem. Soc., March, 1918.)—L. A. C.

*Gelatin; Liquefaction or "solution" of*—in polybasic acids and their salts. M. H. Fischer and W. D. Coffman. J. Amer. Chem. Soc., 1918, 40, 303—312.

EXPERIMENTS showed that there is a progressive increase in the tendency of gelatin to dissolve in mixtures of the salts of polybasic acids as the amount of acid or alkali in these mixtures is increased from a given low point. This shows that hydration and "solution" of a protein are not the same thing; if they were, the addition of an acid or alkali to a solution should cause it to gel, for this causes increased hydration (see preceding abstracts). (See also J. Chem. Soc., March, 1918.)—L. A. C.

*Fibrin; Swelling of*—in acids. I. Colloidal behaviour; The molecular mechanism of—R. C. Tolman and A. E. Stearn. J. Amer. Chem. Soc., 1918, 40, 264—271.

EXPERIMENTS were performed to determine the swelling of fibrin in various solutions of acids and salts and the adsorption of acids from the solutions by the fibrin, with a view to investigating the molecular mechanism by which such swellings and reductions in swellings are brought about. The main points in the theory advanced, which is in accordance with the results obtained, are: Colloids have a marked tendency to adsorb hydrogen ions from acid solutions and hydroxyl ions from alkaline solutions. In an acid solution, the adsorbed hydrogen ions, together with a corresponding number of anions, form a "double layer" on the walls of the pockets in the interior



of the gel and this leads to swelling and imbibition of water by electrostatic repulsion. The addition of a strong electrolyte furnishes ions in the interior of the pockets which will tend to arrange themselves so as to neutralise the electric fields of the adsorbed layers, bringing about a reduction of the swelling. The addition of a neutral salt to an acid solution tends to neutralise the electric field of the adsorbed acid, and hence makes it easier for more acid to get to the surface of the pockets, leading to increased adsorption. Salts with multivalent ions are more effective in reducing swelling than salts with univalent ions, a divalent ion taking up no more room than a univalent ion, but being twice as effective in neutralising the electric field. (See also J. Chem. Soc., March, 1918.)—L. A. C.

#### PATENTS.

*Bichromates [from spent tanning liquors]; Process for reclamation of* —. C. W. Tucker, North Andover, Mass., and T. A. Kelley, Lynn, Mass. Eng. Pat. 105,222, Jan. 25, 1917. (Appl. No. 1291 of 1917.) Under Int. Conv., Mar. 25, 1916.

SODIUM or potassium bichromate is reclaimed from spent chrome tanning liquors by mechanically removing foreign matters in suspension, e.g., by means of aluminium hydroxide, and evaporating to remove the less soluble and deleterious salts whilst retaining the chromates in solution. An alkali carbonate may be used to precipitate the alkaline earths before evaporating the solution.

—F. W. A.

*Glue [from starch]; Process of making* —. F. G. Perkins, Lonsdale, Pa., Assignor to Perkins Glue Co. U.S. Pat. 1,251,275, Dec. 25, 1917. Date of appl., Dec. 26, 1913.

JOINER'S glue is made by stirring starch, water, and "solvent" in a water-jacketed vessel. Steam is injected into the water in the jacket so as to heat the mixture to a temperature below that of the bursting point of the starch, and the heating is checked subsequently so as to prevent overcooking of the glue after it has been dissolved.

—A. B. S.

### XVI.—SOILS ; FERTILISERS.

*Soil; Absorption of cations and anions by* —. A. De Dominicis, G. Mammano, and L. Diaferia. Annali della Regia Scuola Supr. di Agric. Portici, 1916, 13, 26. Bull. Agric. Intell., 1917, 8, 1064.

FIVE different soils were used; chlorides of ammonium, potassium, sodium, calcium, magnesium, aluminium, and trivalent iron were tested for the absorption of the cations, and, for the absorption of the anions, chloride, nitrate, nitrite, sulphate, carbonate, silicate, monohydrogen phosphate, and aluminate of sodium were used. The results show that soil absorbs the cations without exception, those most largely absorbed being iron and aluminium. The anions were also absorbed without exception, the absorption in some cases equalling, and even exceeding, that of the cations, though with certain soils the chloride and nitrate ion showed a negative absorption. The anions are absorbed by amorphous compounds charged with positive electricity and the cations by amorphous compounds charged with negative electricity. Both in the case of the anions and the cations the univalent ions are less absorbed than the divalent and these in turn less than the trivalent ions. The results show that, in the absorbent capacity of the soil, the physical character is the decisive factor.—W. G.

*Soil acidity and the hydrolytic ratio in soils.* C. H. Spurway. J. Agric. Res., 1917, 11, 659—672.

A NUMBER of acid and alkaline soils from glacial formations were examined and a definite relationship was found between the ratio of calcium to iron and aluminium soluble in N/5 hydrochloric acid, and the soil reaction. All acid soils have ratios  $\text{CaO} : (\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3)$  above 1:1.3 and all the alkaline soils have ratios below this figure. The lime requirement of such acid soils may be computed by calculating the amounts of calcium oxide necessary to add to the soil to bring this ratio to 1:1.3. The results so obtained agree closely with those from the indirect titration method. An indirect titration method is described in which certain fixed quantities of a standardised calcium hydroxide solution are allowed to react separately with varying amounts of soil. The concentrations of soil and solution giving a neutral reaction are determined either by electrical resistance measurements or by the colour reaction with phenolphthalein. (See also J. Chem. Soc., March, 1918.)—W. G.

*Basic slag and rock phosphates; Trials on grass land with open-hearth* —. G. S. Robertson. J. Bd. Agric., 1918, 24, 1077—1086.

THE results are given for the first two years of a six years' comparative field test of the relative value of low and high citric-soluble phosphates when applied to grass land. A high-grade basic slag, Gafsa rock phosphate, open-hearth basic slag (fluorspar slag, 45% citric-solubility), and two low-grade, high citric-soluble slags were compared on three clay soils. On all three soils the fluorspar slag gave very satisfactory results. On only one soil did the high citric-soluble slag give results appreciably superior to the low citric-soluble phosphate. The author advocates that, as far as possible, open-hearth basic slag should be utilised for improving poor pasture and meadow hay land. Fineness of grinding is essential.—W. G.

*Phosphates; Conversion of insoluble into "soluble"* —. A. M. Johnston. J. Chem., Met., Min. Soc., S. Afr., 1917, 18, 140—141.

A SAMPLE of Saldanha Bay phosphate contained 20.6% of total and 0.4% of "soluble" phosphoric anhydride. On fusing a finely powdered mixture of 25 grms. of the sample with 15 grms. of calcium carbonate for 1½ hours at 1100° C. the resulting mass (29 grms.) contained 17.2% of total and 6.4% of "soluble" phosphoric anhydride. A mixture of 25 grms. of the sample, 17½ grms. of sodium bisulphate, and 1½ grms. of powdered coke was finely pulverised and heated in a crucible for about an hour at 1100° C. (see Hall's "Fertilisers and Manures," p. 135). The friable mass (36 grms.) was powdered, and treated with three successive portions of about 200 c.c. of water. The dried insoluble portion (34 grms.) contained 13.8% of total and 12.4% of "soluble" phosphoric anhydride, the "soluble" phosphate being determined by agitating 2 grms. of the material for 30 mins. with 200 c.c. of 2% citric acid solution. The total phosphate left in the mass after washing with water represented 91% of the "soluble" phosphate and 81.8% of the total phosphate in the mineral. On a large scale the conversion would be best effected by the use of a reverberatory pan furnace, or by converting the mixture into briquettes, with or without the addition of molasses, and burning it in an oven.—C. A. M.

*Germination; Influence of metallic salts on* — in the presence of calcium. L. Maquenne and E. Demoussy. Comptes rend., 1918, 166, 89—92.

THE chlorides of sodium, potassium, strontium, barium, manganese, and lead, and the sulphates of ammonium, magnesium, zinc, and copper,

function as antagonists to calcium in germination, in the same way as calcium behaves as anti-toxic to them. (See also J. Chem. Soc., March, 1918.)

—W. G.

#### PATENTS.

*Phosphate material; Process of treating* —. II. Blumenberg, jun., Assignor to C. Allen, Los Angeles, Cal. U.S. Pat. 1,251,742, Jan. 1, 1918. Date of appl., June 21, 1917.

FINELY powdered rock phosphate is intimately mixed with an ammonium salt and heated to form ammonium phosphate. Claim is also made for a fertiliser comprising powdered rock phosphate and ammonium sulphate.—W. G.

*Acid calcium phosphates [superphosphate]; Process of making* —. II. Blumenberg, jun., Assignor to C. Allen, Los Angeles, Cal. U.S. Pat. 1,252,318, Jan. 1, 1918. Date of appl., June 7, 1917.

CALCAREOUS phosphate material or phosphate rock is treated with ammonium carbonate or with ammonia and carbon dioxide in the presence of water, calcium carbonate and ammonium phosphate being formed. The ammonium phosphate is separated from the mixture and heated to drive off ammonia and leave phosphoric acid, with which is mixed powdered phosphate material in proportion to produce acid calcium phosphate.

—W. G.

*Bacterial product [fertiliser] and process of preparing same*. G. H. Barp-Thomas, Glen Ridge, N.J. U.S. Pat. 1,252,332, Jan. 1, 1918. Date of appl., Mar. 30, 1917.

THE product consists of granules of humus inoculated with bacteria beneficial to soil, e.g., nitrobacteria, and coated with a protective substance, the adherent coating being superficially treated to prevent the granules from sticking together. An acid or basic mineral fertiliser may be mixed with the coated particles.—W. G.

*Fertilisers*. J. G. Lipman, New Brunswick, N.J., U.S.A. Eng. Pat. 104,171, Feb. 7, 1917. (Appl. No. 1881 of 1917.) Under Int. Conv., Feb. 17, 1916.

SEE U.S. Pat. 1,222,112 of 1917; this J., 1917, 560.

### XVII.—SUGARS; STARCHES; GUMS.

*Sugars; Auto-oxidation of* —. L. Berczeller and E. Szego. Biochem. Zeits., 1917, 84, 1—36.

METHYLENE-BLUE and some other substances promote the oxidation of sugar when shaken in alkaline solution with air. Charcoal was found to promote oxidation by alkaline copper solutions. (See also J. Chem. Soc., March, 1918.)—S. B. S.

*Monosaccharides; Influence of different compounds on the destruction of* — by sodium hydroxide and on the inversion of sucrose by hydrochloric acid. H. I. Waterman. Proc. K. Akad. Wetensch. Amsterdam, 1918, 20, 382—391.

AMINO-ACIDS retard on the one hand the destructive action of sodium hydroxide on dextrose, and on the other hand the inversion of sucrose by hydrochloric acid. Amines are without influence on the destruction of dextrose by alkali but retard the inversion of sucrose by acid. (See also J. Chem. Soc., March, 1918.)—W. G.

*Lactose*. A. Snits and J. Gillis. Proc. K. Akad. Wetensch. Amsterdam, 1918, 20, 520—532.

A DISCUSSION of the relationship between the  $\alpha$ - and  $\beta$ -anhydrous forms and the hydrate of lactose. (See also J. Chem. Soc., March, 1918.)

—W. G.

*Adsorption compounds and adsorption. I. The starch-iodine complex*. L. Berczeller. Biochem. Zeits., 1917, 84, 106—117.

THE presence of potassium iodide is not necessary for formation of the complex. Starch takes up more iodine at lower than at higher temperatures, and the adsorption equilibrium is attained more rapidly in dilute than in concentrated solution. (See also J. Chem. Soc., March, 1918.)—S. B. S.

*Dextrins; Analysis of commercial* —. M. C. Lamb and A. Harvey. J. Soc. Dyers and Col., 1918, 34, 10—11.

THE following determinations are carried out: *Moisture*. 5—6 grms. is dried at 100° C. in an air-oven for 4 hrs. *Ash*. The residue from the preceding determination is incinerated at as low a temperature as possible. *Water-soluble matter*. 10 grms. is shaken with cold water, allowed to stand overnight, an aliquot portion of the filtrate evaporated, and the residue dried. *Reducing sugar*. Determined in a portion of filtrate from preceding estimation, using Fehling's solution and calculating to dextrose (maltose is only present in dextrins manufactured by a diastase process, a method not frequently used commercially). *Starch*. Free starch is tested for by the iodine reaction in the portion insoluble in cold water. The results of a number of analyses show that dextrins are liable to considerable variation in composition, and the following analytical standards for commercial dextrins are suggested: *Moisture*, 8—10%; *ash*, 0.1—0.3%; *water-soluble matter*, 75—85%; *dextrose*, up to 8%. After allowing for any dextrose and soluble ash, the water-soluble matter may be taken to represent the dextrin. The sample should give a neutral reaction. The darker coloured samples contain the highest proportion of water-soluble matter; very dark samples frequently contain a high proportion of dextrose; buff-coloured dextrins are preferable to white samples, as they are not so likely to contain a large quantity of unconverted starch.—F. W. A.

#### PATENTS.

*Decolorising agents [charcoal]; Process of making* — [from molasses] and the products thereof. H. L. Lyon and S. S. Peck, Honolulu, Hawaii. U.S. Pat. 1,251,546, Jan. 1, 1918. Date of appl., Sept. 20, 1915. Renewed May 26, 1917.

A DECOLORISING material is produced by treating molasses with sulphuric acid until charring takes place and removing the soluble substances. Whenever activity diminishes, the treatment with sulphuric acid may be repeated. A "body-giving agent" may be coated or impregnated with the carbon.—L. A. C.

*Process of making glue [from starch]*. U.S. Pat. 1,251,275. See XV.

### XVIII.—FERMENTATION INDUSTRIES.

*Starch; Precipitation of* — by diastase. L. Berczeller. Biochem. Zeits., 1917, 84, 37—41.

THE amount of starch precipitated when its solutions are treated with diastase depends on the previous treatment of the solutions. The author ascribes the action to the aggregation of colloidal particles. Diastatic action diminishes the viscosity and thus promotes precipitation of the larger particles as they are formed. (See also J. Chem. Soc., March, 1918.)—S. B. S.

*Diaslases; Action of oxidising and reducing substances on* —. L. Berczeller and E. Fodor. Biochem. Zeits., 1917, 84, 42—49.

DILUTE permanganate, iodine, and hydrogen peroxide solutions inhibit the action of diastase.



The slight acceleration by formalin is due to the presence of formic acid. (See also J. Chem. Soc., March, 1918.)—S. B. S.

*Enzymes: Periodic phenomena in the action of — as a result of their colloidal nature.* J. T. Groll. Kolloid-Zeits., 1917, 21, 138—148.

PERIODIC variations in the activity of urease, extracted from soya beans, have been observed in experiments, in which the activity was measured from day to day by Van Slyke's method. The enzyme preparations were kept at constant temperature varying from 35° to 65° C. The periodicity is no longer apparent at the last-mentioned temperature and the activity then decreases continuously. The observations seem to show that the decomposition of the enzyme which follows a normal course at 65° C. is complicated at lower temperatures by a periodic change which is supposed to be connected with the colloidal nature of the enzyme. Other enzymes, for example lipase, appear to show the same phenomenon, and as the result of the investigation of colloidal metals with respect to their catalytic activity towards hydrogen peroxide, it is said that these inorganic enzymes show similar effects under certain conditions. To explain the periodicity it is suggested that the colloids are subjected to periodic changes in respect of their degree of dispersity.—H. M. D.

*Water-soluble accessory growth-promoting substance in yeast.* Drummond. See XIXa.

*Formation of [lipolytic] ester-hydrolysing substances by the action of alkali on proteins.* Hulton-Frankel. See XIXa.

## XIXa.—FOODS.

*Flour: Soluble nitrogenous substances as an index of the baking value of —* E. Rousseaux and M. Sirot. Comptes rend., 1918, 166, 190—192.

THE most favourable proportion of soluble nitrogen with respect to total nitrogen in flour is about 16—17%. Flours with this proportion of soluble nitrogen absorb water well and the dough has a good plasticity. If the proportion of soluble nitrogen rises or falls too much the flour is likely to show bad baking qualities. If the total nitrogen rises too much the flour is worked with great difficulty.—W. G.

*Milk: Proteins of cow's —.* T. B. Osborne, A. J. Wakeman, C. S. Leavenworth, and O. L. Nolan. J. Biol. Chem., 1918, 33, 7—17.

WHEN precipitated lactoglobulin containing 2.2% of phosphorus is digested with alcohol, a phosphatide appears to be split off and the remaining coagulated lactoglobulin contains only 0.24% of phosphorus. A similar cleavage of phosphatide occurs in the case of vitellin, and the authors suggest that these so-called globulins are really lecithalbumins or mixtures of proteins. Lactalbumin is practically free from phosphorus. Although traces of proteoses can always be isolated from cow's milk, it is uncertain whether these substances are present as such in the fresh milk or are formed from the proteins by the reagents employed for their isolation. Siegfried's "nucleon" (Z. physiol. Chem., 1895-6, 21, 360) is probably a mixture of uncoagulable protein and a still unidentified organic substance which yields phosphoric acid on hydrolysis. By extraction of precipitated caseinogen with alcohol, a new protein is obtained which resembles the gliadin of wheat in its solubility in 50%—70% alcohol. (See also J. Chem. Soc., March, 1918.)—H. W. B.

*Milk: Adenine and guanine in cow's —.* C. Voegtlin and C. P. Sherwin. J. Biol. Chem., 1918, 33, 145—149.

Cow's milk contains 5 mgrms. of adenine and 10 mgrms. of guanine per litre. These values may be considered as minimum values, because the method of isolation of these aminopurines is not quantitative.—H. W. B.

*Lactic organisms: Proteolytic activity of —.* III. and IV. C. Gorini. Atti R. Accad. Lincei, 1917, [V], 26, ii, 195—199, 223—227.

IN investigations on the proteolytic and coagulating actions of lactic acid bacteria on milk, it is essential that the latter be not sterilised by heating too long or at too high a temperature, since otherwise it is not readily acted on by the enzymes of the micro-organisms. The sterilisation should modify as little as possible the original white colour of the milk and is best effected in steam at the ordinary pressure for 15—20 minutes on each of three successive days; before use the milk should be tested to ascertain if it is free from bacteria.

—T. H. P.

*Dietary-essential, water-soluble B; Study of the — in relation to its solubility and stability towards reagents.* E. V. McCollum and N. Simmonds. J. Biol. Chem., 1918, 33, 55—89.

THE authors describe experiments designed to reveal a method for the isolation of the food accessory substance, termed "water-soluble B," the lack of which leads to the development of polyneuritis. Polyneuritis can be produced in rats by feeding on a diet which is complete in all respects except in content of water-soluble B. Extracts of a material rich in water-soluble B, which had been prepared in various ways, were administered to rats before and after the onset of polyneuritis, and the results clearly indicated that water-soluble B is not extracted directly from beans, wheat germ, or pig's kidney by ether, benzene, or acetone, but is readily extracted by alcohol, and that after being removed by alcohol and deposited on an inert material, such as dextrin, it is then soluble in benzene and slightly soluble in acetone.—H. W. B.

*Yeast: Water-soluble accessory growth promoting substance in —.* J. C. Drummond. Biochem. J., 1917, 11, 255—271.

EXPERIMENTS were made with young rats fed on a basal diet free from water-soluble accessory foodstuff, so that growth was inhibited. Addition of small quantities of dried yeast or of yeast extract ("Marmite") to the diet caused a resumption of normal growth (cp. Seidell, this J., 1917, 562). Dried yeast retained this stimulating power practically unimpaired after extraction with ether or absolute alcohol, though 70% alcohol appeared to remove some of the active substance. A loss of activity followed dialysis of the yeast extract, i.e., the accessory factor passed through the septum. Heating of the yeast or juice for 30 mins. at 100° C. had no effect, but heating at 120° C. in an autoclave caused a partial loss of stimulating power. The activity of the yeast extract was scarcely weakened by boiling with 1% of hydrochloric acid or by the prolonged action of 5% sodium hydroxide at ordinary temperatures. Commercial meat extract ("Lemco") was found to be destitute of the water-soluble accessory growth-promoting factor. The repeated failures to isolate substances of this class in the pure condition are attributed more to the fact that they are readily removed from solution by precipitates, than to their reputed instability. (See also J. Chem. Soc., March, 1918.)—J. H. L.

*Kafirin: Nutritive properties of*—A. G. Hogan. J. Biol. Chem., 1918, 33, 151—159.

**KAFIRIN** (see this J., 1917, 158) is the chief protein in kafir, one of the sorghum grains of America. It is deficient in lysine and cystine, and rats fed on a diet in which kafirin is the only protein do not grow, unless lysine is also added. The subsequent addition of cystine slightly increases the rate of growth.—H. W. B.

*Ester-hydrolysing substances; Formation of [tipolytic]—by the action of alkali on proteins.* F. Hulten-Frankel. J. Biol. Chem., 1917, 32, 395—407.

**FALK** (this J., 1917, 974) has shown that some proteins when treated with alkali acquire ester-hydrolysing properties. Experiments with alkali of different concentrations showed that in the case of casein, gelatin, and egg albumin, the greatest ester-hydrolysing activity is produced by the action of 3*N* alkali. The solutions are all most active at a hydrogen ion concentration less than  $10^{-7}$ , i.e., when the reaction is slightly alkaline. The activity of the solutions is not much affected by the duration of the action of alkali on the protein nor by the temperature at which such action takes place, except when the temperature is unduly high (80° C.). The solutions are not rendered inactive by boiling and their action on different esters shows marked differences. Falk (*loc. cit.*) has suggested that the ester-hydrolysing activity of the hydrolysed proteins is due to the presence of an enol-lactin structure, and the differences in activity may be due to the presence of different groupings attached to the sides of the enol-lactin linkage. The following proteins when acted upon by alkali showed ester-hydrolysing properties: casein, gelatin, egg albumin, castor bean globulin, zein, phaseolin, edestin, ovitellin, egg globulin, pseudoglobulin, euglobulin, soya bean globulin and glutelin, gliadin, and lactalbumin. Solutions obtained by hydrolysing protein with acid do not exhibit ester-hydrolysing properties

*Hæmoglobin [in blood]; Colorimetric estimation of*—W. W. Palmer. J. Biol. Chem., 1918, 33, 119—126.

THE blood is diluted with a hundred volumes of 0.4% ammonia and is then saturated with carbon monoxide. It is compared in a Duboscq colorimeter with a standard carbon monoxide hæmoglobin solution prepared from blood of known oxygen capacity. The accuracy of the estimation is usually within 1%. (See also J. Chem. Soc., March, 1918.)—H. W. B.

*Lactose.* A. Smits and J. Gillis. See XVII.

*Lecithin. I. "Hydrolecithin" and its bearing on the constitution of cephalin.* Levene and West. See XX.

#### PATENTS.

*Antiseptic or sterilising agents for use in the preservative treatment of meat.* A. E. Sherman. London. Eng. Pat. 112,473, Oct. 5, 1916. (Appl. No. 14,138 of 1916.)

BEFORE being chilled for cold storage treatment, meat is exposed to an atmosphere containing an antiseptic gas which is obtained by vaporising stearoptene,  $C_{10}H_{16}O$ , derived from *Carum coplicum*, *Thymus vulgaris*, *Monarda punctata*, or similar herbs. A still more active agent is obtained by vaporising a chlorinated or saline solution containing a stearoptene of the above-mentioned formula. By this means the freshness and the "bloom" of the meat are retained during the cold storage treatment. The stearoptene may

be incorporated, with a binding agent, and made into tablets graduated in size so as to yield a sufficient quantity of antiseptic gas for a chamber of given capacity.—C. A. M.

*Coffee extract.* L. Etaix, Paris. U.S. Pat. 1,251,359, Dec. 25, 1917. Date of appl., Feb. 25, 1915.

SEE Eng. Pat. 2722 of 1915; this J., 1915, 1268.

*Apparatus for cooling and drying compressed materials and substances, seeds and the like and for removing dust therefrom.* Eng. Pat. 112,512. See I.

*Method of preservation.* U.S. Pat. 1,252,090. See XIXB.

### XIXB.—WATER PURIFICATION; SANITATION.

*Water; Identification and determination of lead in*—R. Meldrum. Chem. News, 1918, 117, 49—50.

ONE hundred c.c. of the sample is treated with 1 c.c. of 20% acetic acid, and, after 2 mins., 1 c.c. of saturated hydrogen sulphide solution is added. The coloration obtained, if any, is compared with that given by a known amount of lead under the same conditions. In preparing this comparison solution, the same water as the sample, but free from lead, must be used, since the intensity of the lead sulphide coloration is largely influenced by the saline constituents, etc., of the water. Copper, if present, is detected by means of ammonia, and in this case the lead must be identified and estimated by the bichromate method. (See also J. Chem. Soc., March, 1918.)—W. P. S.

*Alkalis in drinking water; Determination of*—M. Wagenaar. Pharm. Weekblad, 1917, 54, 1454—1455.

THE method of Fresenius involving the separation of the alkali metals is too tedious, and in the indirect method the errors of analysis accumulate in the result (compare Pharm. Weekblad, 1917, 1115). The abbreviated method described by the author (Pharm. Weekblad, 1916, 233) is preferable. After freeing the total solids from silica, iron, and alumina, calcium is precipitated as oxalate and weighed as  $CaO$ , the residue is evaporated with several drops of sulphuric acid, and the ignited residue of alkali sulphates and magnesium sulphate weighed; the magnesium content is then determined. Kolthoff has endeavoured further to simplify the method by using the insolubility of calcium sulphate in 50% alcohol, in which the alkali sulphates and magnesium sulphate dissolve; it was found difficult to extract the calcium sulphate satisfactorily, and errors are introduced in calculating the percentage of alkali. As the potassium content is calculated as sodium, it is recommended that the "atomic weight" of the alkali metal be taken as 24 instead of 23.—F. W. A.

*Dichloramine T; Solvents for*—H. D. Dakin and Dunham. Brit. Med. J., Jan. 12, 1918. Pharm. J., 1918, 100, 82.

**CHLORINATED** paraffin wax ("chlorocosane") is the most satisfactory solvent for dichloramine T, solutions of 8.5 to 10% strength being obtainable. The solvent is prepared by passing chlorine into paraffin wax, of m. pt. 50° C. or higher, at 120° C., until an increase in weight of 45 to 55% is attained.

*Case of poisoning by hydrofluosilicic acid and detection of this acid in animal organs.* Spaeth. See VII.



*Colorimetric estimation of hæmoglobin [in blood].*  
Palmer. See XIXA.

#### PATENTS.

*Water; Filtration of* —. F. P. Candy, Cheam, Surrey. Eng. Pat. 112,672, Jan. 22, 1917. (Appl. No. 1061 of 1917.)

IN a mechanical gravity filter of the type in which the water passes at a high speed through an open filter provided with means for cleaning the filtering medium by a reverse flow of water, the water to be filtered is delivered into a measuring tank above the filter from which it flows by slotted weirs into the filter. A float in a chamber in the measuring tank is connected by a flexible cord to a dial indicator which shows the volume of water passing over the weirs. The filter is maintained full of water by a regulating valve placed on the outlet pipe for the filtered water and connected with a float on the water at the top of the filter. The valve also regulates the rate of outflow according to the level of the water above the filtering material, and at its extreme positions the float brings an alarm bell into action. Rapidity of filtration is effected by connecting the outflow pipe to a suction pump.—J. H. J.

*Water containing sulphuric acid and iron salts; Method of treating* — [and obtaining a pigment]. W. M. Parkin, Pittsburgh, Pa. U.S. Pat. 1,251,564, Jan. 1, 1918. Date of appl., Apr. 5, 1916.

MINE waters containing iron salts and free acid are treated by adding pulverised limestone in sufficient quantity to neutralise the acid and to precipitate the greater part of the iron. The precipitate is separated and heated to 179°–338° C., whereby it is converted into a finely divided pigment without undergoing any grinding operation.—J. H. J.

*Preservation; Method of* —. W. R. Clayton, Evanston, Ill. U.S. Pat. 1,252,090, Jan. 1, 1918. Date of appl., Dec. 2, 1915.

THE substance to be preserved is first partially desiccated and is then placed in a closed chamber in the presence of calcined gypsum. A circulation of heated air is kept up in the chamber.—J. H. J.

*Water; Method and apparatus for obtaining pure* — from salt water. F. T. Evans, Newport, R.I., U.S.A. Eng. Pat. 112,679, Jan. 25, 1917. (Appl. No. 1294 of 1917.)

SEE U.S. Pat. 1,225,118 of 1917; this J., 1917, 665.

*Percolators for use in extracting oil [from garbage] by solvents.* Eng. Pat. 112,590. See XII.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Orimes; New method of dehydration of* — by catalysis. A. Mailhe and F. de Godon. Bull. Soc. Chim., 1918, 23, 18–20.

TWO aldoximes, isovaleraldoxime and oenanthaldoxime, and two ketoximes, isobutyronoxime and isovaleronoxime, were dehydrated by passing their vapours over aluminium oxide or thorium oxide heated at 340°–360° C. (See also J. Chem. Soc., March, 1918.)—W. G.

*Hydrogenation under the influence of colloidal catalysers and how to account for this process.* J. Böeseken and H. W. Hofstede. Proc. K. Akad. Wetensch., Amsterdam, 1918, 20, 424–431.

MEASUREMENTS of the rate at which hydrogen is absorbed by solutions of ethyl undecylenate and of cinnamic acid and its methyl and ethyl esters in presence of colloidal palladium as catalyst, show that the hydrogenation does not follow a normal course. The irregularities are probably caused in part by impurities in the hydrogen and by coagulation of the sol. (See also J. Chem. Soc., March, 1918.)—H. M. D.

*Phenylethylamine; Reduction of* —. A. Weinlagen. Biochem. J., 1917, 11, 272–276.

AQUEOUS solutions of phenylethylamine hydrochloride, prepared from phenylalanine by splitting off carbon dioxide, were shaken with hydrogen in presence of finely divided platinum at the ordinary temperature (*cp.* Willstätter and Hatt, this J., 1912, 634). The calculated quantity of gas was slowly absorbed and the product was identified as hexahydrophenylethylamine. Commercial synthetic phenylethylamine, under similar conditions, absorbed no hydrogen whatever, though the platinum used was afterwards tested and found to be active. Negative results were also obtained with phenylalanine, tyrosine, and *p*-hydroxyphenylethylamine (from tyrosine). (See also J. Chem. Soc., March, 1918.)—J. H. L.

*Fullers' earth; Utilisation of the adsorption power of* — for chemical separations. [Adsorption of quinine and Methylene Blue.] A. Seidell. J. Amer. Chem. Soc., 1918, 40, 312–328.

THE relative adsorption capacities of some thirty-six samples of fullers' earth and similar clays have been examined by experiments in which the samples were shaken up for half an hour with solutions of quinine bisulphate and Methylene Blue of varying concentration. The free bases are readily adsorbed in both cases and the quantities adsorbed per 100 grms. of the earth afford a basis for the comparison of the adsorption capacities. In general there is a parallelism between the series of numbers obtained for the alkaloid and the dye. The numbers range from 20.0 to 0.2 in the case of quinine and from 27.8 to 0.2 for Methylene Blue. The English samples of fullers' earth had nearly the same adsorption capacity for quinine, which was considerably greater than that of the American samples examined. Several of these were found to have little adsorbent power, although a sample of unknown origin from Utah gave very good results. (See also J. Chem. Soc., March, 1918.)—H. M. D.

*Gitalin (x-digiloxin); Rotatory power and molecular weight of* —. J. Burmann. Bull. Soc. Chim., 1917, 21, 290–293.

GITALIN carefully prepared and purified at a temperature not exceeding 0° C. has  $[\alpha]_D^{25} = -25.2^\circ$  (in chloroform) and  $-18.8^\circ$  (in alcohol). Its molecular weight as determined by the ebullioscopic method, with chloroform as solvent, is 539. (See also J. Chem. Soc., March, 1918.)—W. G.

*Lecithin. I. "Hydrolecithin" and its bearing on the constitution of cephalin.* P. A. Levene and C. J. West. J. Biol. Chem., 1918, 33, 111–117.

HYDROLECITHIN is readily prepared by the reduction of lecithin with hydrogen in the presence of palladium. After recrystallisation from methyl ethyl ketone, it softens, on heating, between 80° and 90° C., turns brown about 100° C., starts to

melt about 200° and runs down the tube, giving a dark red liquid, at 235° C. As thus prepared, the hydrolecithin is not pure, but contains 20% of a substance containing amino-nitrogen, which is probably hydro-cephalin. (See also J. Chem. Soc., March, 1918.)—H. W. B.

*Nitriles; New preparation of aliphatic—by catalysis.* A. Mailhe. Comptes rend., 1918, 166, 121—123.

ALIPHATIC as well as aromatic nitriles may be prepared by the action of ammonia on the corresponding esters in the presence of thorium oxide or aluminium oxide at 480°—490° C. (See also J. Chem. Soc., March, 1918.)—W. G.

*Nitriles; New method of formation of—by catalysis.* A. Mailhe and F. de Godon. Comptes rend., 1918, 166, 215—217.

ALIPHATIC or aromatic nitriles may be prepared by passing the vapours of the corresponding aldehyde along with ammonia over thorium oxide heated at 420°—440° C. Water and some condensation products of the aldehyde and ammonia are formed at the same time. (See also J. Chem. Soc., March, 1918.)—W. G.

*Sea-sapphire from different parts of France; Essential oil of—.* M. Delépine and G. de Belsunce. Bull. Soc. Chim., 1918, 23, 24—35.

OIL of sapphire obtained from plants grown in different parts of France always contained the three characteristic constituents, 5,6-dimethoxy-3,4-methylenedioxy-1-allylbenzene, the methyl ether of thymol, and crithmene. Samples from different sources differed considerably, however, in the relative amounts of these three constituents present, and some of the oils contained in addition *p*-cymene, *d*-pinene, and a paraffin, m. pt., 63° C. (See also J. Chem. Soc., March, 1918.)—W. G.

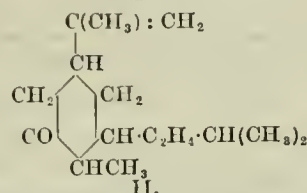
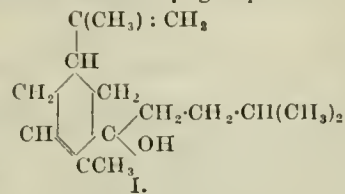
*Essential oils; Constituents of—.* Gum ammoniac oil, and *synthetical experiments on the nature of its constituents.* F. W. Semmler, K. G. Jonas, and P. Roenisch. Ber., 1917, 50, 1823—1837.

THE gum-resin which is obtained from the umbelliferous plant, *Dorema ammoniacum*, and several *Ferula* species, yields about 0.3% of an oil on distillation. This is found to contain linalyl and citronellyl acetates; a unicyclic, dihydrosesquiterpene, "ferulene,"  $C_{15}H_{26}$ , closely associated with a bicyclic sesquiterpene,  $C_{15}H_{24}$ , a fraction with b.pt. 124°—126° C. at 7 mm. being a mixture of these in the approximate ratio 3:1; an ethylenic, sesquiterpene-ketone, "doremone,"  $C_{15}H_{24}O$ , b.pt. 145°—155° C. at 12 mm., amounting to about 22% of the oil, which may be reduced by means of platinum and hydrogen to tetrahydrodoremone, or by sodium and alcohol to the ethylenic alcohol, "doremol," b.pt. 145°—150° C. at 12 mm.; dorenyl acetate, b.pt. 155°—165° C. at 12 mm.; and cetyl alcohol, in the highest fraction. This is the first recorded instance of the occurrence of cetyl alcohol in a plant, and doremone is the first example of a sesquiterpene-ketone. The unicyclic sesquiterpene, "isoamyl- $\alpha$ -dehydrophellandrene,"  $C_{15}H_{24}$ , b.pt. 130°—132° C. at 11 mm., may be obtained by the action of magnesium isoamyl iodide on carvone, and this may be hydrogenated to "isoamylmethane,"  $C_{15}H_{32}$ , b.pt. 131°—133° C. at 14 mm. Farnesol may be hydrogenated to inactive hexahydrofarnesol,  $C_{15}H_{32}O$ , b.pt. 145°—155° C. at 15 mm., which is evidently closely related to the above "doremol."—J. C. W.

*Essential oils; Constituents of—.* *Synthetical experiments in the unicyclic sesquiterpene series.* F. W. Semmler, K. G. Jonas, and K. Oelsner. Ber., 1917, 50, 1838—1842.

THE product of the action of magnesium isoamyl

iodide on carvone (see preceding abstract) contains not only isoamyl- $\alpha$ -dehydrophellandrene but the normal carbinol, "isoamylcarveol," (I), b.pt. 155°—170° C. at 12 mm., which loses water when treated with potassium bisulphate, yielding the phellandrene derivative, and also "isoamyldihydrocarvone" (II), b.pt. 144°—148° C. at 10 mm., which is produced by the introduction of the isoamyl group at the double bond in the carvone ring instead of at the carbonyl group.



Iso-amylcarveol is the first synthetic unicyclic sesquiterpene alcohol, and isoamyldihydrocarvone is the first synthetic sesquiterpene-ketone.—J. C. W.

*Mercury ammonia compounds.* Holmes. See VII.  
*Colorimetric estimation of hæmoglobin [in blood].* Palmer. See XIXA.

*Solvents for dichloramine T.* Dakin and Dunham. See XIXB.

### XXIII.—ANALYSIS.

*Testing and standardisation of motor fuel.* Lomax. See IIA.

*Constituents of wood which give colour reactions.* Wiehelhaus and Lange. See V.

*Case of poisoning by hydrofluosilicic acid and detection of this acid in animal organs.* Spaeth. See VII.

*Detection of small amounts of hydrocyanic acid.* Kolthoff. See VII.

*Rapid characterisation of the mercuric ion in insoluble sulphates. Application to the identification of this ion in general.* Denigès. See VII.

*New method of determining copper.* Moir. See X.

*Silica-glass mercury still.* Hostetter and Sosman. See X.

*Temperature determinations by eutectic alloys.* Steinmetz. See X.

*Soil acidity and the hydrolytic ratio in soils.* Spurway. See XVI.

*Adsorption compounds and adsorption. I. The starch-iodine complex.* Berczeller. See XVII.

*Analysis of commercial dextrans.* Lamb and Harvey. See XVII.

*Proteolytic activity of lactic organisms.* Gorini. See XIXA.

*Colorimetric estimation of hæmoglobin [in blood].* Palmer. See XIXA.

*Identification and determination of lead in water.* Meldrum. See XIXB.

*Determination of alkalis in drinking water.* Wageenaar. See XIXB.



## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Bennis. Heat treatment of liquids and gases. 3673. Mar. 2.  
 Boulton. Drying apparatus. 3637. Mar. 1.  
 British Dyes, Ltd., Parker, and Turner. Filter-press. 3516. Feb. 28.  
 British Thomson-Houston Co. (General Electric Co.). Manufacture of sheets by aggregation of flake materials. 3317. Feb. 25.  
 Fadden. Apparatus for producing mineral distillates of definite composition. 2810. Feb. 18.  
 Giesecke. 3369. *See X.*  
 Hope. Apparatus for drying sand, etc. 3054. Feb. 20.  
 Mangelsdorff. Dehydrating. 3144. Feb. 21.  
 Meihuizen. Drying apparatus. 3035. Feb. 20. (Holland, Mar. 31, 1917.)  
 Paterson. Filtering apparatus. 3701. Mar. 2.  
 Roberts. Apparatus for automatically taking samples of liquids. 2827. Feb. 18.  
 Rodda. Apparatus for reclaiming heat, oils, fats, etc. 2811. Feb. 18.  
 Sadezky. Producing a drying medium. 2888. Feb. 19.  
 Soar. Production of permanent natural colour effects by the action of light rays on chemical matter. 3275. Feb. 25.  
 Tuckfield. Continuous fractional distillation apparatus. 3356. Feb. 26.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 16,283 (1916). Wallace and Tiernan. Treatment of liquids with gases or purification of water. (113,288.) Feb. 27.  
 2290 (1917). Rasmussen, and Ernst Ges. Separation of air or gases from liquids. (104,183.) Feb. 27.  
 2579 (1917). Gee. Centrifugal separators. (113,326.) Feb. 27.  
 3444 (1917). Barbet et Fils et Cie. Cooling-plates for continuous rectification apparatus. (105,549.) Mar. 6.  
 6140 (1917). Perry. Apparatus for distilling. (113,373.) Feb. 27.  
 6175 (1917). Wallace and Tiernan. Diffusers for use in diffusing gases in liquids. (113,374.) Feb. 27.  
 12,380 (1917). Hofmann. Apparatus for drying material in bulk. (109,267.) Feb. 27.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

- Boizard. Burning inferior or anthracitic fuel in furnaces or automatic stokers. 3006. Feb. 20. (Fr., Dec. 14, 1917.)  
 Climie. Gas-producers. 2994. Feb. 20.  
 Day. Extraction of hydrocarbon products from coals and shales. 3521. Feb. 27.  
 Dela Devesa. Compounds for increasing the calorific value of coal, etc. 3447. Feb. 27.  
 Fadden. 2810. *See I.*  
 Fairweather. Washing coal gas, etc., with oil. 3506. Feb. 27.  
 Firth, Blakely, Sons, and Co., and Shaw. Arrangement and operation of vertical gas-retorts. 3240. Feb. 23.

Kimber and Palmer. Retort for treating oil shale. 3173. Feb. 22.

- Lynn. Producer-gas plant. 3715. Mar. 2.  
 Marks (Soc. Anon. Ital. G. Ansaldo & Co.). Combustion processes. 2851. Feb. 18.  
 Marks (Soc. Anon. Ital. G. Ansaldo & Co.). Simultaneously producing gas from lignite and other combustibles, and calcium phosphate for agricultural purposes. 2924. Feb. 19.  
 Moorhouse and Simpson. Carbonisation and distillation of coal, etc., by electricity. 2878. Feb. 19.  
 Nitrogen Products and Carbide Co., and Perkin. Destructive distillation of carbonaceous materials. 2920. Feb. 19.  
 Rew. Water-gas producer. 3282. Feb. 28.  
 Rideal and Taylor. Removal of sulphur and sulphur-containing bodies from gases. 3702. Mar. 2.  
 Russell. Apparatus for distilling coal, shale, peat, etc. 3232. Feb. 23.  
 Smith. Scrubbers, cleaners, and coolers for gas. 2832. Feb. 18.

#### COMPLETE SPECIFICATION ACCEPTED.

- 16,126 (1917). Rose. Apparatus for producing combustible fuel. (113,585.) Mar. 6.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATION.

- Day. Hydrocarbon products. 3521. *See II.*

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATION.

- Corbidge. Dyes. 2808. Feb. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 2776 (1917). British Dyes, Ltd., Turner, and Segaller. Manufacture of N-dihydro-1,2,2',1'-anthraquinoneazine. (113,489.) Mar. 6.  
 3372 (1917). Holliday and Clayton. (Production of yellow colouring-matter. (113,510.) Mar. 6.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

- Bindley and Weller. Production of dope or substitutes for celluloid, vulcanite, etc. 3502 and 3503. Feb. 27.  
 Dagnall. Treatment of paper. 3598. Feb. 28.  
 Dreyfus. Manufacture of unflammable celluloid. 3336. Feb. 25. (Fr., Mar. 22, 1917.)  
 Dreyfus. Manufacture of solvents for cellulose acetate, and of dopes, solutions, films, etc., therewith. 3337. Feb. 25.  
 Gilchrist. Treating cotton, etc., preparatory to the production of yarns and threads. 2999. Feb. 20.  
 Moore. Material for paper-making. 3202. Feb. 22.  
 Murata, Ogawa, and Okubo. Production of zinc-chloride solution of cellulose. 3139. Feb. 21.  
 Rafsky. Coated paper. 3235. Feb. 23.  
 Zdanowich. Nitrocellulose solutions. 3172. Feb. 22.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 1224 (1916). Meiro. Treatment of flax fibre. (113,285.) Feb. 27.  
 2982 (1917). Schaufelberger. Digesters for use in the manufacture of cellulose, etc. (113,494.) Mar. 6.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

- Ashton, and Calico Printers' Assoc. Mordanting vegetable fabrics and fibres. 3287. Feb. 25.  
 Dudley. Dyeing machines. 3233 and 3234. Feb. 23. (U.S., Mar. 29 and 31, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

8050 and 8051 (1916). Larivei. Machines for dyeing and finishing textile fibres. (113,453 and 113,454.) Mar. 6.

2610 (1917). Calico Printers' Assoc., and Fourniaux. Production of black on vegetable fibres. (113,484.) Mar. 6.

12,511 (1917). Annicq. Dyeing or similarly treating yarns in the form of hanks, skeins, or bundles. (113,575.) Mar. 6.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Cote, Thompson, and Spencer, Chapman, and Messel. Solutions of sulphuric anhydride. 3245. Feb. 23.

Curtis. Composition for manufacture of lining of acid pots, etc. 2904. Feb. 19.

General Chemical Co. Converting atmospheric nitrogen into a compound containing combined nitrogen. 3607. Feb. 28. (U.S., Nov. 3, 1917.)

Haynes and Williams. Synthetic production of ammonia. 2914. Feb. 19.

Lindblad. Production of nitrogen compounds, especially nitrates. 3057. Feb. 20.

Llewellyn, Spence, and Spence and Sons. Production of aluminous compounds. 3152. Feb. 22. Marks. 2921. *See* II.

Mineral Products Corporation. Purification of alumina. 3528. Feb. 27. (U.S., Aug. 11, 1917.)

Nitrogen Corporation. Production of substantially pure cyanogen compounds and their derivatives. 3415. Feb. 26. (U.S., June 12, 1917.)

Soar. 3275. *See* I.

Taylor. Catalysts for use in the production of hydrogen. 3703. Mar. 2.

Titan Co. Aktieselskabet. Production of titanium compounds. 3624. Mar. 1. (Norway, Mar. 23, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

16,822 (1916). Castner-Kellner Alkali Co., and Moore. Manufacture of bleaching-powder. (113,457.) Mar. 6.

6841 (1917). Skoglund. Concentration of sulphuric acid. (113,551.) Mar. 6.

15,982 (1917). Wenman. Manufacture of phosphorus. (113,584.) Mar. 6.

17,036 (1917). Raffin et Fils. *See* X.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Barron and Barron. Artificial millstones, and manufacture thereof. 2848. Feb. 18.

Carborundum Co. (Urtel). Manufacture of abrasive articles. 3707. Mar. 2.

## IX.—BUILDING MATERIALS.

## APPLICATION.

Borch. Manufacture of Portland cement from raw materials with hydraulic properties. 2958. Feb. 19. (Denmark, Feb. 24, 1917.)

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Arzano and Clerici. Manufacture of iron by electrolysis. 3431. Feb. 26. (Ital., Mar. 19, 1917.)

Berglund. Extraction of lead. 2936. Feb. 19. (Sweden, Mar. 10, 1917.)

Cito. Treatment of copper sweepings and scraps. 3591. Feb. 28.

Edwards. 3536. *See* XXIII.

Fairholme and Hatfield. Refining molten metals in the process of manufacture. 3620. Mar. 1.

Giesecke. Process for solidifying conglomerates of fine ores, flue dust, purple ore, etc. 3369. Feb. 26.

Hloworth (Norsk Hydro-Elektrisk Kvaestof-aktieselskab). Connecting cast tubes of silicon-iron alloys. 3524. Feb. 27.

Humbert. Manufacture of metals in electric furnaces. 3420. Feb. 26.

Kamfield. Preparation to prevent penetration of carbon into portions of steel objects during case-hardening. 2823. Feb. 18.

Stobie. Electric steel manufacture. 2970. Feb. 20.

Sundberg. Separation of mineral values from ore by flotation. 2964. Feb. 19.

Zourieff. 3505. *See* XI.

## COMPLETE SPECIFICATIONS ACCEPTED.

3285 (1917). Griffith. Electro-chemical treatment of ores. (113,508.) Mar. 6.

3398 (1917). Deutsch-Luxemburgische Bergwerks u. Hütten A.-G., and Klinkenberg. Manufacture of ingot iron and steel. (113,512.) Mar. 6.

3721 (1917). Larsen. Production of iron-sponge. (113,351.) Feb. 27.

9053 (1917). Van Gulck. Furnace especially for the treatment of zinc. (113,393.) Feb. 27.

9852 (1917). Grondal. Concentration of ores by flotation. (108,308.) Mar. 6.

10,379 (1917). Berglund. Extraction of zinc by the electro-thermal process. (109,435.) Feb. 27.

11,522 (1917). Metals Disintegrating Co. Reducing metals to a finely-divided state. (109,257.) Feb. 27.

15,682 (1917). Moll et Cie. Process for soldering aluminium. (110,758.) Feb. 27.

17,036 (1917). Raffin et Fils. Treatment of ilmenites or titanic iron ores. (111,668.) Mar. 6.

276 (1918). Richmond Gas Stove and Meter Co., and Thompson. Metal-heating, metallurgical, and like heating furnaces. (113,594.) Mar. 6.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

Antonoff. Carbon conductors. 3532. Feb. 27.

Antonoff. Electric batteries. 3660. Mar. 1.

Arzano and Clerici. 3431. *See* X.

Bibby. Electric furnaces. 3726. Mar. 2.

Chloride Electrical Storage Co., and Dean. Electric accumulators. 3414. Feb. 26.

Humbert. 3420. *See* X.

Macdonald and Macdonald. Electrolytic gas process. 2877. Feb. 19.

Moorhouse and Simpson. 2878. *See* II.

Rogers. Electric resistance furnaces. 3070. Feb. 21.

Silberrad. Manufacture of electric cells. 3452. Feb. 27.

Stobie. 2970. *See* X.

Travis, and Watson and Co. Supply of electric energy to furnaces. 3460. Feb. 27.

Zourieff. Accumulators, etc. 3430. Feb. 26.

Zourieff. Production of spongy lead. 3505. Feb. 27.

## COMPLETE SPECIFICATIONS ACCEPTED.

3285 (1917). Griffith. *See* X.

3807 (1917). Svenska Accumulator Aktiebolaget Jungner. Electric batteries. (108,849.) Mar. 6.

10,379 (1917). Berglund. *See* X.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Nairn. 2875. *See* XIII.

Rodda. 2811. *See* I.

## COMPLETE SPECIFICATION ACCEPTED.

4711 (1917). Downs and Bellwood. Apparatus for extracting oil from seeds and like oleaginous materials. (113,530.) Mar. 6.



### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

- Bindley and Weller. Synthetic shellac, resin, etc., and methods of producing same. 3501. Feb. 27.  
Corticine Floor Covering Co., and Thompson. Cement, and method of making same for use in the manufacture of linoleum, etc. 3348. Feb. 26.  
Darrin and Sperr. Manufacture of resins. 3664. Mar. 1.  
Murray. Manufacture of water paint, etc. 3394. Feb. 26.  
Nairn. Treatment of fatty acids for use in manufacture of linoleum. 2875. Feb. 19.  
Titan Co. Aktieselskabet. Pigments. 3549. Feb. 28. (Norway, Feb. 28, 1917.)

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

- Bindley and Weller. 3501. *See* V.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 12,085 (1917). Peachey. Agents for accelerating vulcanisation of rubber, and method of producing them. (113,570.) Mar. 6.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATION.

- Heyl. Drying japanned or patent leather. 3053 and 3143. Feb. 20 and 21. (Ger., May 22. and Apr. 27, 1916.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 2197 (1917). Röhm. Tanning processes. (104,338.) Feb. 27.  
13,036 (1917). D'Angelo. Apparatus for tanning hides. (113,408.) Feb. 27.  
14,862 (1917). Röhm. Tanning or tawing by means of aluminium salts. (110,750.) Mar. 6.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

- Marks (Soc. Anon. Ital. G. Ansaldo & Co.). 2924. *See* II.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 2924 (1917). Borghesani and Stampa. Fertilisers. (113,493.) Mar. 6.  
12,186 (1917). Richards. Treatment of straw and other vegetable substances for manurial purposes. (113,571.) Mar. 6.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATION.

- Herriot (Deerr.). Apparatus for macerating bagasse. 3471. Feb. 27.

### XVIII.—FERMENTATION INDUSTRIES.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 2683 (1917). Stevenson. Production of a blended yeast. (113,486.) Mar. 6.  
8677 (1917). Hadley. Non-intoxicating beer, and process of brewing the same. (113,560.) Mar. 6.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

- Bugler. Manufacture of egg substitute. 3357. Feb. 26.  
Dantzig, and Naaml. Vennootsch. Chemisch and Pharmaceutisch Laboratorium. Preservation of vegetables and fruits. 3132. Feb. 21.

- Dowding. Treatment of potatoes for obtaining food products. 3523. Feb. 27.

- Growcott. Treatment of grain, etc. 3510. Feb. 27.

- Lyle. Combined food and beverage. 3625. Mar. 1.

- Mackay. Disinfecting and refrigerating. 3364. Feb. 26.

- Mangelsdorff. Preparing potatoes for dehydration. 3145. Feb. 21.

- Norton. Manufacture of margarine. 3609. Feb. 28.

- Stokes. Incinerating furnaces. 3385. Feb. 26.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 16,283 (1916). Wallace and Tiernan. *See* I.  
2761 (1917). Jones, and Jones and Attwood. Purification of sewage and other liquids. (113,333.) Feb. 27.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

- Comp. des Prod. Chimiques d'Alais et de la Camargue. Preparation of acetic aldehyde from acetylene. 3589. Feb. 28. (Fr., Jan. 12, 1917.)

- Comp. des Prod. Chimiques d'Alais et de la Camargue. Preparation of acetic acid by oxidation of ethylaldehyde. 3590. Feb. 28. (Fr., Mar. 6, 1917.)

- Garroid and Nielsen. Catalytic manufacture of formaldehyde, etc., from hydrocarbons. 3632. Mar. 1.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 2151 (1917). Imray (Soc. Chem. Industry in Basle). Manufacture of a new physiologically active substance and compounds containing nitrogen from ovaries, corpus luteum, or placenta. (113,311.) Feb. 27.

- 2896 (1917). Parke, Davis, and Co. Anæsthetic bodies. (106,087.) Mar. 6.

- 2928 (1917). Parke, Davis, and Co. Anæsthetic bodies. (105,746.) Mar. 6.

- 3244 (1917). Simpson. Treatment of liquids for curative and therapeutical purposes. (113,505.) Mar. 6.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATIONS.

- Bateman. Kinematograph films. 3491. Feb. 27.  
Camiller and Lyell. Films, screens, or plates for natural-colour photography. 3115. Feb. 21.

- Camiller and Lyell. Films or screens for kinematography. 3116. Feb. 21.

- Hess-Ives Corporation. Coloured photographic image, and method of producing same. 2910. Feb. 19. (U.S., Feb. 20, 1917.)

- Hess-Ives Corporation. Colour photography. 2911. Feb. 19. (U.S., Feb. 20, 1917.)

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATIONS.

- Holloway and Summers. Matches ignited by friction. 2865. Feb. 18.

- Tonge. Matches. 2976. Feb. 20.

- Zdanowich. 3172. *See* V.

### XXIII.—ANALYSIS.

#### APPLICATIONS.

- Edwards. Testing hardness of metals, and instrument therefor. 3536. Feb. 28.

- Michell. Determining viscosity of fluids. 3124. Feb. 21. (Australia, July 12, 1917.)

- Roberts. 2827. *See* I.

## I.—GENERAL; PLANT; MACHINERY.

## PATENTS.

*Cooling and otherwise treating gases with liquids; Apparatus for —.* A. W. Mathys, London. Eng. Pat. 111,113, Nov. 8, 1917. (Appl. No. 16,389 of 1917.) Under Int. Conv., Nov. 8, 1916.

IN an apparatus for humidifying or cooling air by contact with water, a tank containing water is divided by vertical partitions into compartments which communicate at the bottom. Each compartment contains a number of vertical plates spaced apart to form narrow passages and mounted in skeleton frames or cages, each pair of adjacent frames being suspended by a cable or chain passing over a pulley. The pulleys are oscillated so that the plates are alternately raised from and lowered into the water, the velocity of the frames being at a minimum at the two ends of their travel. Air enters at the side of the casing and passes between the plates in each frame in succession, openings being provided in the partitions above the water level at different heights alternately, so that the air is forced to take a zigzag path.—W. F. F.

*Furnaces or mufles; Gas heated —.* J. M. Wallwin, Warwick. Eng. Pat. 112,747, Sept. 4, 1917. (Appl. No. 12,646 of 1917.)

THE body of the furnace is formed of a tubular member, having openings in the sides for the admission of the material to be heated and the fuel, i.e., gas and air. The ends are recessed to receive top and bottom plates, which are clamped in position between metal flanges. Outlet openings for the products of combustion are provided at the top and inlet openings at the bottom of the second and subsequent furnaces when two or more furnaces are arranged in tiers. The lowermost metal flange is provided with a bracket to which the furnace door is hinged. The top member is provided with a plain dished flange with a hole in the centre. The furnaces are held together by bolts passing through from top to bottom.—J. H. P.

*Furnaces and the like.* I. Hall, Birmingham. Eng. Pat. 112,827. (Appl. Nos. 1557, Jan. 31, and 2484, Feb. 20, 1917.)

THE furnace is provided with four forced-draught burners for burning coal gas, producer gas, petrol gas, high pressure gas, or liquid fuel, discharging tangentially into the heating or melting chamber. Two of the burners are set 180° apart, but not at the same level, at the lower end of the chamber, and the other two are similarly placed at a higher level, the lines joining each pair being at right angles in plan. The burners are preferably arranged on an imaginary helical line. The escape of the combustion gases is delayed by providing one or more helical grooves or channels in the refractory lining of the heating chamber, or by means of projecting helical ribs. Two reservoirs may be provided for the gas and air respectively, with separate outlets for each of the burners.

—W. F. F.

*Kiln.* C. W. Bowsman, Joliet, Ill. U.S. Pat. 1,251,333, Dec. 25, 1917. Date of appl., July 12, 1917.

THE baking chamber is heated by means of peripheral combustion chambers, and the waste gases are discharged through slots in the floor of the chamber into radial flues leading to a discharge tunnel. By suitable adjustment of the area and capacity of the several slots and flues a substantially equal volume of the gases is drawn through each flue.—J. H. P.

*Kiln.* J. L. Harper, Niagara Falls, N.Y., U.S.A. U.S. Pat. 1,253,487, Jan. 15, 1918. Date of appl., Apr. 29, 1910. Renewed Dec. 1, 1917.

SEE Fr. Pat. 404,208 of 1909; this J., 1910, 98. Flues are provided through which gas may be introduced into the electrically heated middle portion of the kiln.

*Separating solids from liquids; Means for —.* G. A. Spratt, Chingford, Essex. Eng. Pat. 112,791, Jan. 3, 1917. (Appl. No. 151 of 1917.)

IN an apparatus for separating a liquid from solid matter suspended in it, e.g., for purifying cutting and cooling oils from metallic particles, a centrifugal pan of oval shape in vertical cross-section is provided with a wide central opening at the top into which the liquid is supplied and over the rim of which the purified liquid overflows. A small central opening is provided in the bottom of the pan and a flanged sleeve, projecting upwards into the vessel and closed at the top, is secured over the opening. A bearing plate is secured to the underside of the pan which rests on a washer supported on a horizontal flange carried by the vertical driving shaft. The shaft is extended upwards into and concentric with the closed sleeve, leaving an annular space between the two. The pan is rotated by frictional contact with the washer, and rotates about its centre of gravity so that vibration is avoided. The pan is surrounded by a casing in which the purified oil collects. In a modification, the pan is rigidly connected to the driving shaft and the liquid is supplied through a central opening in the shaft. A cover may be provided, the purified liquid then escaping through perforations in the wall of the pan close to the edge of the cover.—W. F. F.

*Mixing and cooling machine.* F. A. Connelly, Pompton Lakes, N.J. U.S. Pat. 1,250,435, Dec. 18, 1917. Date of appl., Jan. 22, 1917.

A DRUM provided with internal vanes arranged obliquely, and a central hollow shaft are mounted in a frame so that they can be rotated in opposite directions. Parallel radial hollow blades are mounted on the shaft and are connected together at their outer ends. A cooling medium passes through the shaft and blades, and means are provided for supplying material to and removing it from the drum.—J. H. P.

*Fractional distillation and condensation; Process and apparatus for —.* J. W. Aylsworth, East Orange, N.J., Assignor to Condensate Co. of America, Bloomfield, N.J. U.S. Pat. 1,250,760, Dec. 18, 1917. Date of appl., Nov. 9, 1915.

THE apparatus comprises a still connected to one end of a horizontal condensing coil contained in a closed receptacle, the coil having a condensing surface at least ten times as great as the evaporating surface of the still. The other end of the coil is connected to a second water-cooled coil, and means are provided for observing the temperature in the connecting pipe as well as in the receptacle. The liquids condensing in the bottom loops of the horizontal coil are led back to the still. A heating fluid is rapidly circulated from a heater through the receptacle and around the horizontal coil by means of a pump, the supply of heat being regulated by an adjustable thermostat governed by the temperature of the fluid in the receptacle, so that this is maintained at any desired one of several temperatures, in accordance with the temperatures indicated in the connecting tube between the coils and in the receptacle.—B. N.



*Liquids having different boiling points; Art of separating* — G. R. Sanford, T. H. Miller, and B. Torrey, jun., Syracuse, N.Y., Assignors to Somet-Solvay Co., Solvay, N.Y. U.S. Pat. 1,252,725, Jan. 8, 1918. Date of appl., Mar. 29, 1916.

THE mixture is distilled and the vapour condensed in a dephlegmating column, so that the upper part of the column contains only liquid having the lowest boiling point, the other condensates passing downward to the lower part of the column. The distillation is stopped when all the liquid of lowest boiling point has been distilled from the charge, the residue is removed, and the operation is repeated with a fresh charge.—W. F. F.

*Drying machine.* C. Crouch and G. J. Hewlett, Cleveland, Ohio. U.S. Pat. 1,250,980, Dec. 25, 1917. Date of appl., Sept. 9, 1916.

A FLUID-JACKETED revolving drum is provided with stationary and gland-bearing heads. Fluid conduits communicate with the latter and with the drum, and means are provided in some of the conduits for trapping liquid out of the lower portion of the drum at regular intervals. The material to be dried passes through the drum in an opposite direction to the fluid in the jacket, and means are provided for increasing or decreasing the resistance offered to the passage of the material and fluid through the drum and jacket respectively.—J. H. P.

*Drying materials; Art of* — A. F. Wright, East Orange, N.J. U.S. Pat. 1,251,317, Dec. 25, 1917. Date of appl., Mar. 18, 1916.

THE material to be dried is distributed over superposed shelves having heaters placed adjacent thereto. A heated drying atmosphere is circulated through the spaces between the shelves and the heaters, and, by means of deflectors, eddy currents are set up, whereby intimate contact of the drying atmosphere with the material and with the heaters is obtained.—J. H. P.

*Refrigeration.* F. G. Keyes, Hoboken, N.J., Assignor to National Automatic Refrigerator Co. U.S. Pat. 1,251,538, Jan. 1, 1918. Date of appl., May 27, 1916.

A LIQUID refrigerating composition consists of water, an active absorbent such as ammonium nitrate, and a volatile refrigerating component, such as ammonia, the proportions being such that on applying a vaporising temperature of 100° C. the ratio of water to ammonia in the vapour evolved is not greater than in the critical boiling mixture thereof at 0° C.—J. H. P.

*Precipitation of particles from fluid streams; Art of electrical* — C. W. Girvin, Assignor to U. G. Scott, Philadelphia, and H. G. Seitz, New York. U.S. Pat. 1,252,104, Jan. 1, 1918. Date of appl., Mar. 22, 1917.

OPPOSING electrode systems are so arranged that an electric field is produced having permanently located active and inactive portions, and means are provided for rotating the collecting-electrode system on its axis to move a deposit-collecting face between an active and an inactive portion and for removing the deposit whilst it is in the inactive zone.—J. H. P.

*Air; Apparatus for treating — with liquids.* J. H. Lorimer, Philadelphia, Pa. U.S. Pat. 1,253,401, Jan. 15, 1918. Date of appl., July 9, 1914.

A HORIZONTAL conduit is provided at its lower part with a tank for containing liquid, and at its upper part with a deflecting plate extending obliquely downwards and backwards. A number of transverse shafts are provided above the tank geared to rotate together and carrying sets of parallel discs on each shaft partly dipping into the liquid.

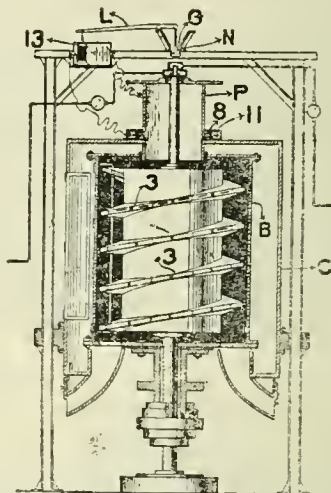
The plane of the discs is thus parallel to the air current, which is caused to flow through the conduit by a fan, and the discs on one shaft are out of alignment with and overlapping those on the adjacent shaft. The upper edges of the discs fit closely to the deflecting plate.—W. F. F.

*Washing and drying machine; Centrifugal* — R. C. McCaleb, Chicago, Ill. U.S. Pat. 1,253,403, Jan. 15, 1918. Date of appl., Apr. 3, 1917.

IN a centrifugal washing and drying machine a cylindrical container is provided with an internal basket mounted on a vertical rotating shaft. A member movable vertically carries a bucket which may be moved upwards and downwards through a central opening in the bottom of the basket. The basket and shaft are adapted to be tilted into two different positions, in which the shaft engages with worm and spur driving gear respectively, so that the basket may be rotated at two different speeds.—W. F. F.

*Centrifugal machine.* L. Salcines, Guantánamo, Cuba. U.S. Pat. 1,253,428, Jan. 15, 1918. Date of appl., Jan. 3, 1916.

A CASING, C, contains a rotating basket, B, to which the material to be treated is fed from the trough, 3, through the cylindrical chamber, P.



A valve, 11, controlling the discharge from the trough, 3, to the chamber, P, is reciprocated by the electromagnet, 13, through the pivoted lever, L. A series of cylinders, 8, is arranged radially around the chamber, P, and each is provided with a piston normally pressed into the cylinder, P, by a spring. The pistons are pressed outward by the pressure of the material in the chamber and thus complete the electric circuit of the electromagnet, 13, which then opens the valve, 11. The feed of the material is thus controlled by the pressure of the material in the feeding chamber, P.—W. F. F.

*Filters; Process of cleaning — in connection with the purification of sugar, oils, and chemicals.* P. L. Wooster, Yonkers, N.Y., U.S.A. Eng. Pat. 109,795, Aug. 10, 1917. (Appl. No. 11,541 of 1917.) Under Int. Conv., Aug. 17, 1916.

SEE U.S. Pat. 1,240,290 of 1917; this J., 1917, 1121.

*Desiccation [of liquids]; Process of — and apparatus therefor.* H. Wade, London. From G. A. Buhl, Chicago, U.S.A. Eng. Pat. 112,965, Dec. 29, 1916. (Appl. No. 18,632 of 1916.)

SEE U.S. Pat. 1,215,889 of 1917; this J., 1917, 376.

*Distillation of liquids.* O. Söderlund and T. Boberg, Assignors to Techno Chemical Laboratories, Ltd., London. U.S. Pat. 1,252,962, Jan. 8, 1918. Date of appl., Dec. 10, 1914.

SEE Eng. Pat. 28,537 of 1913; this J., 1915, 68.

*Machine for mixing concrete and the like.* Eng. Pat. 107,575. See IX.

*Process of drying grain and other coarse granular materials.* U.S. Pat. 1,250,496. See XIX A.

## II A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Fuels; Determination of total carbon in — and its importance in the study of fuel economy.* E. Damour and M. de la Morinière. Rev. Mét., 1917, 14, 789—800.

IN considering the heat economy of a fuel the total carbon content has an importance not generally recognised. Few laboratories are capable of estimating this content rapidly, and in general a commercial analysis including ash, fixed carbon, volatile matter, and sometimes calorific power is considered sufficient. In making a balance-sheet of heat production and consumption in an industrial furnace, it is impossible to calculate the amount of heat lost outside the furnace without using a term of comparison common to the fuel and to the gaseous waste products of combustion. From the content of total carbon in the fuel it is possible to calculate the volume or weight of gaseous products of combustion. The apparatus used by the authors for the determination of total carbon is a combination of the Mahler-Goutal bomb (fitted with an additional outlet) with the usual absorption tubes generally associated with the combustion furnace in organic analysis. 0.5 gm. of the fuel, contained in a silica capsule, is introduced into the apparatus. After the explosion, and on opening the connection to the tared absorption vessels, the gases circulate through sulphuric acid, soda-lime, potash, and pumice stone moistened with sulphuric acid. A large Maquenne tube charged with 50 c.c. of de Nolly's solution (alkaline solution containing about 4 grms. of soda per litre) indicates any loss of carbon dioxide due to failure to regulate the speed of gas flow. In this way it is possible to determine in 1½ hours the total carbon of the fuel. Tests have shown that the losses due to the residue from combustion, and formation of carbon monoxide or of hydrocarbons are negligible. A correction is made for the carbon dioxide remaining in the bomb. To ensure complete combustion and to reduce the error possible from the amount of space in the bomb, the authors recommend a smaller bomb of 250 c.c. capacity and adapted for a pressure of 25 atmospheres. From the results of tests conducted as above, the authors give elaborate calculations leading to a thermal balance-sheet of a battery of boilers and of an open-hearth furnace respectively.—J. E. C.

*Water-gas; Production of — in continuous vertical retorts.* H. M. Balsam. Gas J., 1918, 141, 247—248.

THE author discusses the advantages of making water-gas, *in situ*, in a continuously worked vertical retort, the speed of the descent of the charge being adjusted to leave an ample depth of incandescent coke, and steam at 40 lb. per sq. in. being introduced somewhat on the lines indicated by West (see this J., 1917, 1265: 1918, 26 A). The following results are given relating to normal working and to "combination" working respectively: Gas per ton, 14,120 and 27,390 cub. ft.;

calorific value, 540 and 436 B.Th.U.; candle power, 14.5 and 5.0; gas produced per retort in 24 hours, 738 and 950 cub. metres.—J. E. C.

*Illuminating gas; Washing of — with anthracene oil.* M. Raffo and G. Scagliarini. Annali Chim. Appl., 1917, 8, 114—121.

THE increase in the sp. gr. of anthracene oil which has been used for several months for extracting benzene, etc., from gas is not due to naphthalene. Fractional distillation of a sample of anthracene oil which had thus been used gave the following results:—Between 200° and 230° C. a liquid distilled which yielded 2.09% of naphthalene and 7.75% of liquid residue, whilst between 230° and 270° C. there distilled 6.16% of an oil yielding 1.08% of naphthalene. These results compared with those given by a sample of saturated anthracene oil show that the amount of naphthalene left in solution in the tar is somewhat greater than that dissolved by the oil. The increase in the sp. gr. of the anthracene oil is due to the presence of various products boiling between 270° and 330° C., principally fluorene, phenanthrene, and acenaphthene. The increase in the sp. gr. of the oil has no material influence on the extraction of light oils from the gas, and after 4 months' use an oil will extract almost the same amount of benzene as when first used. Anthracene oil can be used for extracting light oils until mechanical conditions prevent its movement within the washing chamber.—C. A. M.

### PATENTS.

*Alcohol; Method of rendering — more readily ignitable when used as fuel in internal combustion engines.* P. H. King and J. A. Stoneham, London. Eng. Pat. 112,741, Aug. 10, 1917. (Appl. No. 11,478 of 1917.)

ALCOHOL is vaporised and mixed with an equal volume of acetylene. The mixture is then condensed in the ordinary manner, the liquid holding the acetylene in solution.—J. E. C.

*[Gas retort] furnace; Regenerative —.* S. B. Russell, Assignor to Parker-Russell Mining and Manufacturing Co., St. Louis, Mo. U.S. Pat. 1,251,426, Dec. 25, 1917. Date of appl., Jan. 15, 1917.

IN a gas retort setting heated by producer gas, the primary air circulates round a waste heat flue in the recuperator. Steam is introduced through steam jets or injecting nozzles in close proximity to the ports in the division wall between the gas producer and the recuperator so as to cause a mixture of heated primary air and steam to be supplied to the producer.—J. E. C.

*Gas producers.* F. Thuman, Westminster. Eng. Pat. 112,909, Sept. 25, 1917. (Appl. No. 13,770 of 1917.)

IN a gas producer, a steam- and blast-distributing grate projects through an eccentrically disposed aperture in the ash-pan into the fuel bed and is caused by the rotation of the ash-pan to gyrate, means being provided for coupling the grate to the ash-pan when desired, so that it will then also rotate about its axis.—J. E. C.

*Gas producer.* J. R. George, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pat. 1,251,011, Dec. 25, 1917. Date of appl., Feb. 6, 1915.

A GAS producer is fitted with a rotating ash support and a scraper which normally rotates with this support. By means of gear responsive to changes in the level of the fuel bed, a stop can be interposed in the path of the scraper to prevent its rotation.—J. E. C.



*Gas producer.* E. A. W. Jefferies, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pats. (A) 1,251,038 and (B) 1,251,039, Dec. 25, 1917. Dates of appl., Aug. 2, 1913, and Dec. 16, 1914.

(A) A GAS producer comprises a fuel section, to which fuel is fed during the operation of the producer, a fuel levelling device resting on the surface of the fuel and capable of adjustment to the level of the bed, with means for causing relative rotation between the fuel section and the levelling device. (B) A bed of fuel is supported in a gas producer, and the surface levelled by a swinging arm pivoted at one end on a horizontal axis and resting by gravity on the surface of the fuel bed. Means are provided for ensuring relative rotation between the levelling arm and the fuel bed.—J. E. C.

*Gas generators; Steam and air mixer for* —. F. Logan, Phoenixville, Pa. U.S. Pat. 1,251,252, Dec. 25, 1917. Date of appl., June 16, 1916.

A STEAM and air mixer consists of a casting having an upper hollow portion, to which air and steam are supplied through separate pipes, and an integral annular retarding chamber forming the lower portion of the casting. There are openings in the opposite side walls of this chamber, one opening being closed by a plug and the other being fitted with a discharge pipe which extends through the wall of the generator to the space beneath the grate.—J. E. C.

*Fuel; Apparatus for producing combustible* —. J. R. Rose, Edgeworth, Pa. U.S. Pat. 1,252,032, Jan. 1, 1918. Date of appl., Nov. 29, 1915.

A PAIR of vertical and parallel flues, each filled with refractory material in chequered formation, is arranged in a generator, the upper ends being connected by a transverse passage. A combustible fluid, together with a regulated supply of air, is admitted to the lower end of one of the flues, and a liquid hydrocarbon, preheated in a passage between the two flues, is admitted about half-way up the flue.—J. E. C.

*Fuel; Process of manufacturing gaseous* —. J. R. Rose, Edgeworth, Pa. U.S. Pat. 1,252,033, Jan. 1, 1918. Date of appl., May 19, 1916.

SUPERHEATED steam and a hydrocarbon fluid are passed through a duct heated to at least 2200° F. (1200° C.) to induce dissociation. A heated mixture of liquid hydrocarbon and superheated or dried steam is then added, the final mixture passing through another duct also heated to at least 2200° F. (1200° C.). Carbon dioxide is then removed from the gaseous product.—J. E. C.

*Distilling hydrocarbons; Art of* —. J. W. Coast, jun., Tulsa, Okla. Assignor to The Process Co. U.S. Pat. 1,252,401, Jan. 8, 1918. Date of appl., Nov. 11, 1916.

PETROLEUM distillates having a boiling point of over 300° F. are mixed with steam and forced at a velocity greater than 2600 ft. per minute through a coil of small pipe at a pressure of 50 to 150 lb. per sq. in., and between 850° and 1500° F., into a cracking chamber into which is also injected a finely divided and relatively cool petroleum distillate. This latter is thereby also cracked; the fractions of high boiling point are condensed, and carbon or coke removed from the vapours. The remaining vapours are led off and condensed.—L. A. C.

*Hydrocarbonaceous or bituminous product and process of making the same.* T. M. Hart, Assignor to P. M. Stewart, New York. U.S. Pat. 1,252,433, Jan. 8, 1918. Date of appl., Sept. 30, 1915.

ASPHALT is mixed with alum, whereby the water

of emulsification is converted into water of crystallisation. The mixture is heated below the temperature at which water is evolved.—L. A. C.

*Distilling oil and other liquids; Process of and apparatus for* —. W. Thomas, Nahaimo, British Columbia. U.S. Pat. 1,253,747, Jan. 15, 1918. Date of appl., Feb. 2, 1917.

GAS, heated in a regenerator situated below the heating chamber of the still, is passed under pressure into an "injector-circulating device" situated within the still, drawing with it the top layer of liquid and foam into the body of the liquid, and thus assisting vaporisation and preventing boiling over. Waste hot products from the regenerator are led into the heating chamber of the still.—L. A. C.

*Cracking hydrocarbons; Art of and apparatus for* —. J. W. Coast, jun., Assignor to The Process Co., Tulsa, Okla. U.S. Pats. 1,252,999 and 1,253,000, Jan. 8, 1918. Date of appl., May 22, 1917.

A HOLLOW movable shield, cooled by water, is placed between the still and the heating medium in order to protect overheated portions of the still.—L. A. C.

*Coke-oven.* T. von Bauer, Tautenburg, Germany, Assignor to B. Zwillinger, New York. U.S. Pat. 1,253,445, Jan. 15, 1918. Date of appl., July 11, 1913.

SEE Ger. Pat. 262,116 of 1912; this J., 1913, 901.

*Process of making sodium ferrocyanide.* U.S. Pats. 1,252,711 and 1,252,742. See VII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Destructive distillation of Philippine wood.* A. H. Wells. Philippine J. Sci., 1917, 12, A, 111—125.

THE experimental plant consisted of a cylindrical iron retort capable of holding 10 to 15 kilos. of wood and heated electrically in such a manner that the inside temperature could be controlled over a range of 500° C. The vapours passed into a tar trap, a piece of iron pipe 10 cm. in diameter, capped at each end and fitted with a pet-cock at the bottom, and then into a condenser and scrubber consisting of a coil of lead piping 2.5 cm. in diameter, fitted into a barrel and leading to a small tower containing pieces of glass tubing. Both fast and slow distillations were made on each kind of wood; in the former the current was not controlled, except at the beginning of the exothermic reaction (265°—275° C.), during which period it was shut off in order to prevent loss by overtaxing the condensing system: in the latter, the current was so applied that the apparent moisture was driven off first, a pause in the distillation marking the end of this stage (121°—180° C.); the current was then gradually increased and subsequently was shut off for the period of the exothermic reaction, after the completion of which it was raised slowly to about 330° C., and then rapidly to 550° C. The highest yields of wood alcohol and acetic acid were obtained from hard mangrove woods, the lowest from soft woods with the highest resinous content. Controlled distillations in all cases gave higher yields of methyl alcohol and generally of acetic acid. Acetone, aldehyde, and higher ketonic compounds were found in most of the distillates. The tars obtained were fractionally distilled, yielding below 100° C. colourless inflammable oils with a strong characteristic odour and having sp. gr. 0.958—0.998. The fraction from 150° to 250° C. varied from pale yellow to brown, and quickly darkened in colour; sp. gr. 1.010—1.028. The green and blue oils began to pass over at 230° C.: these were accompanied by a small amount of

water, indicating decomposition, and contained a high percentage of substances which reacted with alkali. The fractions above 250° C. were semi-solid and usually red. The pitch was vitreous, with conchoidal fracture and of rather high melting point. All of the specimens of charcoal would be suitable for domestic purposes, and some of them for iron smelting. The author discusses costs and working expenses for wood distillation on the large scale in the Philippine Islands. Percentage yields of various products from different woods are given in the following table:—

### III.—TAR AND TAR PRODUCTS.

#### PATENT.

*Distilling hydrocarbons or liquids likely to form emulsions; Apparatus for treating or —.* F. M. A. A. Melehoir, Assignor to Soc. Anon. des Combustibles Industriels, Paris. U.S. Pat. 1,253,411, Jan. 15, 1918. Date of appl., Sept. 13, 1912.

SEE Eng. Pat. 17,954 of 1912; this J., 1913, 904.

Wood.			Pyroli- gneous distillate.	Tar.	Non-con- densable gases.*	Charcoal.	Methyl alcohol.	Acid (as 100% acetic acid).
Botanical name.	Commercial name.	Hardness.						
<i>Rhizophora</i> sp.....	Bacauan	Very hard	39.5	4.1	21.2	35.2	2.12	5.16
<i>Bruguiera parviflora</i> W. & A. . .	Langarai	"	40.8	4.2	22.9	32.1	1.84	4.95
<i>Avicennia officinalis</i> L. ....	Api- <i>api</i>	"	43.4	5.2	19.0	32.4	1.71	4.66
<i>Intsia bijuga</i> (Colebr.) O. Ktze.	Ipi	Hard	32.4	6.8	19.1	41.7	1.61	4.40
<i>Hopea</i> sp. ....	Yacal	"	34.6	6.6	20.3	38.5	0.93	4.61
<i>Shorea guiso</i> (Blanco) Blume ..	Guljo	"	35.5	6.2	20.0	38.3	1.55	4.70
<i>Liptocarpus</i> sp. ....	Apitong	Medium hard	39.7	12.6	15.1	32.6	0.91	3.80
<i>Shorea polysperma</i> (Blanco) Merr.	Tangull	"	39.8	7.5	18.8	33.9	1.23	2.92
<i>Pterocarpus</i> sp. ....	Narra	"	39.4	7.8	16.0	36.8	1.36	4.37
<i>Pentacme contorta</i> (Vid.) M. & R.	White lapan	Soft	36.6	6.8	23.4	33.2	1.20	2.90
<i>Anisoptera thurifera</i> Blanco ..	Palosapis	"	39.9	3.8	19.7	36.6	0.74	3.83
<i>Pinus insularis</i> Endl. ....	Benguet pine	"	38.9	10.3	15.1	35.7	0.82	2.12

\* By difference.

—L. A. C.

#### PATENTS.

*Distillation of coal, oil shales, and other similar carbonaceous materials.* S. B. Billbrough, Johannesburg, Transvaal. Eng. Pat. 112,793, Jan. 11, 1917. (Appl. No. 576 of 1917.)

THE material is distilled at a high temperature (800° to 1200° C.) and the vapours are withdrawn through a passage remote from the walls of the retort. This passage may consist of a central perforated tube, decreasing in cross-sectional area from the top to the bottom of the retort, and may be rotated if desired. The perforated tube may be covered with wire gauze or netting if desired. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 3934 of 1875, 2312 of 1883, 11,581 of 1890, 15,486 of 1894, 30,673 of 1897, 19,143 of 1900, 16,925 and 29,313 of 1909, and 4598 of 1915; this J., 1891, 689; 1895, 645; 1899, 255; 1901, 1100; 1910, 1049, 1366; 1916, 919.)—J. E. C.

*Oil shales; Distillation of —.* G. E. Heyl, London. Eng. Pat. 112,997, Feb. 6, 1917. (Appl. No. 1865 of 1917.)

BEFORE distillation, shale is soaked for a long period, e.g., 24 hrs., in a relatively small volume of a liquid hydrocarbon, such as mineral oil or coal-tar oil. The distillation is preferably effected in two or more stages, a further quantity of the hydrocarbon being added between each. For example, a volume of coal-tar oil distilling up to 220° C., equal to 10% of the mass of the shale, is used to soak the shale and the mixture distilled to 220° C. After cooling, the still residue has added to it a further 10% of the oil and the distillation is continued to 400° C. The yield of oil, less the added 20% of coal-tar oil, will be greater than would be obtained without the addition of the oil.—L. A. C.

*Heat-developing apparatus.* W. A. Bone and J. W. Wilson, Leeds, and C. D. McCourt, Assignors to Radiant Heating, Ltd., London. U.S. Pat. 1,253,342, Jan. 15, 1918. Date of appl., Feb. 20, 1912. Renewed June 11, 1917.

SEE Eng. Pat. 2401 of 1911; this J., 1912, 110.

### IV.—COLOURING MATTERS AND DYES.

#### PATENTS.

*Dye stuffs and dyes; Manufacture of —.* N. Malcolmson, London. Eng. Pat. 112,813, Jan. 24, 1917. (Appl. No. 1228 of 1917.)

CERTAIN carbonaceous materials, such as plants of the algæ family, ferns, peat, or used tea-leaves, are heated with a concentrated mineral acid, and the saturated solution thus produced is evaporated to dryness or to a paste, any residual acid being neutralised by sodium carbonate, caustic soda, or other suitable neutralising agent. To increase the solubility of the dye, sodium chloride, sugar, or other soluble material may be added either to the mixture in the retort or to the paste or powder, also, other dyestuffs, mordants, or topping bath materials may be added in the same way. The acid vapours evolved are condensed and the resulting liquid used again.—L. A. C.

*Colouring bodies; Preparation of — for use in food, pharmaceutical and other preparations.* A. Boake, Roberts and Co., Ltd., and T. Rendle, London. Eng. Pat. 112,886, May 17, 1917. (Appl. No. 7105 of 1917.)

THE dye, or other colouring matter, is dissolved in a heated salt such as sodium thiosulphate, sodium carbonate, sodium sulphate, sodium phosphate, magnesium sulphate, alum, and others which on heating melt in their own water of crystallisation. The mixture is cooled, allowed to set, and formed into blocks, slabs, or masses, or reduced to powder. A solution containing up to 15 to 20% of the colouring matter can be obtained. —L. A. C.

*[Anthracene] vat dyes; Bluish-green — and process of making them.* M. Kardos, Charlottenburg, and P. Nawiaskey, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,253,252, Jan. 15, 1918. Date of appl., Apr. 15, 1914.

SEE Addition of Feb. 13, 1914 to Fr. Pat. 458,949 of 1913; this J., 1915, 22.



*Method of preparing nitroso compounds from ligninsulphonic acid.* Eng. Pat. 103,653. See V.

*Nitration of cotton cellulose and influence of prolonged bleaching on its transformation into dinitrocellulose.* Namias. See XXI.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### PATENTS.

*Fulling and materials for use therein.* K. Mohs, Allach, Germany. Eng. Pat. 110,343, June 25, 1917. (Appl. No. 9118 of 1917.) Under Int. Conv., Oct. 12, 1916.

A COMBINATION of organic colloids with inorganic protecting colloids is prepared in a solid and soluble form and a small proportion of the colloid powder is added to the fulling liquor, whereby quicker felting and less shrinkage are obtained. For the preparation of the colloid a mash is made with a material, such as Indian seaweed, "Indian bread or Tuckakoe," Japanese seaweed, or Iceland moss, and a solution containing about 2% of potassium carbonate, which is raised to 55° C. The filtered infusion is evaporated under vacuum with a certain proportion of colloidal silicic acid, and when concentrated to a suitable consistence, the mixture is discharged and mixed with a further portion of the silicic acid so that the mixture is solidified and obtained as a dry powder. For instance, 1000 kilos. of Iceland moss yields about 300 kilos. of air-dry organic colloid substance, which is combined with about 200 kilos. of colloidal silicic acid yielding 500 kilos. of air-dry solid colloid powder.—J. F. B.

*Cellulose; Process of dissolving*—. E. C. R. Marks, London. From International Cellulose Co., Reno, Nevada, U.S.A. Eng. Pat. 104,173, Jan. 31, 1917. (Appl. No. 1564 of 1917.)

SEE U.S. Pat. 1,218,954 of 1917; this J., 1917, 450. Sulphuric acid may be used instead of phosphoric acid, and the hydrochloric acid should contain not less than 25% by weight of hydrogen chloride. When it is desired to obtain a solution of sugars (dextrose and pentoses) by hydrolysis of the cellulose, the amount of water present in the initial mixture of cellulose and acids should not exceed one-fifth of the weight of cellulose.

*Cellulose solutions; Spinning copper oxide ammonia*—. E. Elsaesser, Langerfeld, Germany. Eng. Pat. 113,010, Feb. 19, 1917. (Appl. No. 2438 of 1917.)

IN apparatus for spinning threads from cuprammonium solutions of cellulose, in which flowing liquids are used for feeding the threads, air bubbles in the precipitating liquid disturb the formation of the thread. To prevent these bubbles the precipitating liquid is freed from dissolved gases, for instance, by subjecting it, preferably while hot, to the action of a vacuum.—J. F. B.

*Viscose; Process of making*—. N. E. Katz, Meridian, Miss. U.S. Pat. 1,251,237, Dec. 25, 1917. Date of appl., Aug. 28, 1917.

ALKALI-CELLULOSE containing an excess of alkali is caused to react directly with carbon bisulphide associated with an organic plasticising agent and a solvent for the same.—J. F. B.

*Paper-making and like machines; Felt-driers for*—. W. A. Aitken, Gravesend. Eng. Pat. 112,678, Jan. 25, 1917. (Appl. No. 1240 of 1917.)

THE lower drying felt of a paper machine is carried round a large auxiliary drying cylinder situated at

the wet end of the range of paper-drying cylinders, so that the felt is dried immediately before it comes in contact with the paper.—J. F. B.

*Translucent paper; Reinforced*— and process for making same. E. Bellan, Paris. Eng. Pat. 113,062, July 14, 1917. (Appl. No. 10,233 of 1917.) Under Int. Conv., Apr. 20, 1917.

REINFORCED translucent paper is manufactured from two sheets of paper between which is fixed a reinforcement of textile or metallic thread; the adhesive which is employed for sticking the sheets together contains an oily or fatty body, e.g., 30–40% of linseed oil, in such proportion that the adhesive and translucent effects are produced in one operation.—J. F. B.

*Vegetable substances; Method of treating*— for the recovery of their values. M. W. Marsden, Logan Station, Pa. U.S. Pat. 1,251,261, Dec. 25, 1917. Date of appl., Apr. 26, 1916.

FIBROUS vegetable material is cleansed and softened by immersion, then subjected to squeezing in order to loosen the fibres lengthwise and crush the material; the crushed mass is washed with the use of steam and leached while open to the atmosphere, to remove the extractive matter. After thorough rinsing, the material is digested until the fibres are reduced to the uniformity required for paper-making.—J. F. B.

*Sulphite acid liquor and the like; Means for making*—. G. D. Jessen, New York. U.S. Pat. 1,251,533, Jan. 1, 1918. Date of appl., Mar. 24, 1917.

THE apparatus comprises a pair of towers each having near the lower end a partition supporting blocks of acid-proof material to retard the downward flow of liquor, and some distance above this partition a second partition supporting a layer of limestone. Water is supplied to the upper ends of the towers and sulphur dioxide gas to the lower ends. Gas-outlet pipes at the upper ends of the towers are connected with a pipe through which the gas can be supplied again to the lower ends of the towers. The liquor withdrawn from the lower ends of the towers is returned to the towers, below the partition supporting the limestone, for re-treatment.

*Ligninsulphonic acid or its salts; Method of preparing nitroso compounds from*—. E. Oman, Stockholm. Eng. Pat. 103,653, Jan. 4, 1917. (Appl. No. 204 of 1917.) Under Int. Conv., Jan. 14, 1916.

SODIUM ligninsulphonate, 100 grms. of 60% purity, obtained by saturating sulphite-cellulose waste lye with sodium chloride, is dissolved in 600 grms. of water; a solution of 60 grms. of sodium nitrite in 100 of water is added, the mixture is cooled with ice and treated with a solution of 60 grms. of concentrated sulphuric acid in 400 of water. After some hours the nitroso compound is separated by the addition of salt and purified by washing with a saturated solution of sodium chloride. If desired, the free ligninsulphonic acid, precipitated by hydrochloric acid, may be dissolved in water and treated for the production of the nitroso compound. An impure nitroso compound may be prepared direct from the sulphite waste lye. The product has a yellowish-brown colour and may be used as a colouring matter. (See also this J., 1918, 121 A.)—J. F. B.

*Wood pulp sulphite plants; Process of treating by-product liquors from*—. C. Marchand, Kimberly, Wis., Assignor to The Marchand Process Co., Chicago, Ill. U.S. Pats. (A) 1,253,853 and (B) 1,253,854, Jan. 15, 1917. Dates of appl., (A) Nov. 26, 1915, and (B) Aug. 14, 1916. Renewed Nov. 19, 1917.

(A) SULPHITE wood pulp waste liquor is treated

in a tank with sulphuric acid to convert the sulphites into sulphates and precipitate the lime; a predetermined quantity of the treated liquor is discharged into a preliminary heater and charge-measurer, where it is maintained at a temperature below boiling, and subsequently discharged into an evaporator. The liquor is evaporated under vacuum to remove a portion of the sulphur dioxide and is then passed to a second evaporator at a lower temperature and higher vacuum until most of the sulphur dioxide is vaporised. The vapour from the evaporators is absorbed in a reagent to produce a sulphite and the concentrated liquor is treated in a tank with an oxidising agent to produce sulphuric acid and then fermented. (B) Waste sulphite liquor is treated to convert the sulphites into the corresponding chlorides and sulphur dioxide. The latter is expelled, the residue fermented, the alcohol distilled off, the residual liquid evaporated, the solid residue incinerated, and the chlorides are recovered by repeated crystallisation.—J. F. B.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid situation in the United States.* L. B. Skinner. Met. and Chem. Eng., 1918, 18, 82–85.

Prior to the war sulphuric acid in the United States was made exclusively from the gases produced by roasting zinc blende and pyrites or by the blast-furnace treatment of Tennessee copper ores. The demand for acid during the war has stimulated production which, however, has never yet met all requirements. To increase the output from zinc-acid plants brimstone has been added to the ore fed into the burners (Hegeler kilns) whereby the chamber output has been increased 20%. Australian concentrates are being used and may continue to be used after the war. The production from plants now using or which previously used pyrites has also increased, to some extent due to the substitution of brimstone for the now scarcer Spanish ore. There is, however, no commercial advantage, even under present conditions, in doing this. The development of the contact processes in America has been restricted by the onerous conditions of licences and royalties imposed by European patent owners. When fine concentrates are used, the roaster gases carry much undesirable dust. The Cottrell process has failed to work satisfactorily under the very severe practical conditions of acid plants where it has been tried. The lead tower in chamber plants is giving way to cheaper masonry designs and the stoneware fittings to silicon-iron castings. The increased demand for sulphuric acid has been due to the production of explosives and to the demand of oil refineries largely caused by the growth of use of motor vehicles under war conditions. The quantity available for the manufacture of fertilisers has been insufficient for the demand. In the future, acid production will be an adjunct to the metallurgical industry. Pyrites and also brimstone burning for acid will probably decline. Much will depend on the transport facilities which should be arranged in the national interest to favour the utilisation of the waste and noxious gases produced by smelting works.—H. J. H.

*Selenium in sulphuric acid; Detection of*—. L. P. J. Palet. Ann. Chim. Analyt., 1918, 23, 25–26.

If a small crystal of aspidospermine is added to a few drops of sulphuric acid containing selenium, and the mixture then heated until fumes appear, the crystal, and afterwards the whole mixture, is coloured an intense violet. A coloration is not

obtained in the absence of selenium, but a red coloration develops if the acid contains an oxidising substance such as lead peroxide or potassium chlorate.—W. P. S.

*Phosphorous, hypophosphoric, and phosphoric acids in mixtures; Estimation of*—. R. G. van Name and W. J. Huff. Amer. J. Sci., 1918, 45, 91–102.

A METHOD for the estimation of phosphorous acid in presence of hypophosphoric acid depends on the reactivity of the former towards iodine in presence of disodium hydrogen phosphate, which is added in sufficient quantity to leave the solution alkaline when the reaction is completed. The solution containing the phosphorous acid is made approximately neutral, the requisite quantity of disodium hydrogen phosphate added, followed by the addition of an excess of a standard solution of iodine. The solution is then left in the dark for at least two hours and the residual iodine determined by titration with thiosulphate after the solution has been acidified with phosphoric acid. The method gives accurate and concordant results and is a considerable improvement on Sievert's method (this J., 1909, 1169) in which the reaction takes place in presence of potassium bicarbonate. According to the authors' experiments this method always gives results which are somewhat too high. The new method may also be used in the estimation of hypophosphoric acid if this is previously hydrolysed by heating the solution at 100° C. in presence of hydrochloric acid. The reaction takes place according to the equation:  $H_4P_2O_6 + H_2O = H_3PO_3 + H_2PO_4$ . Phosphorous and hypophosphoric acids, when present in the same solution, may be estimated by the application of the iodometric method before and after hydrolysis. Solutions containing phosphorous, hypophosphoric, and phosphoric acids may also be examined by the method, the iodine absorption being determined before and after hydrolysis and the total phosphoric acid determined in the oxidised solution. A modification of Kühling's permanganate method (this J., 1900, 1149) for the estimation of phosphorous acid is also described, zinc sulphate being added to prevent the solution from becoming alkaline. The mixture, to which an excess of standard permanganate is added, is heated for 90 minutes in a steam bath, cooled, and treated with an acidified solution of potassium iodide, the liberated iodine being titrated with thiosulphate. The relation between the permanganate and thiosulphate solutions is determined by a blank experiment.—H. M. D.

*Hypophosphoric acid solutions; Rate of hydrolysis and electrical conductivity of*—. R. G. van Name and W. J. Huff. Amer. J. Sci., 1918, 45, 103–118.

MEASUREMENTS have been made of the velocity of hydrolysis of hypophosphoric acid in aqueous solutions at different temperatures. Acid solutions of hypophosphates cannot be boiled without appreciable loss by hydrolysis. (See also J. Chem. Soc., Mar., 1918.)—H. M. D.

*Lead acetate; Solubility of*— in water. Y. Osaka and R. Hara. Mem. Coll. Sci., Kyoto, 1917, 2, 147–150.

THE solubility of lead acetate in pure water has been found to be 51.38 parts per 100 of water at 25°, 87.77 at 35°, and 154.25 at 45° C. (See also J. Chem. Soc., Mar., 1918.)—H. M. D.

*Metaphosphoric acid; Thermal dissociation of*—. D. Balarew. Z. anorg. Chem., 1918, 102, 34–40.

By the dehydration of orthophosphoric acid by



heat, metaphosphoric acid is formed and, when subjected to prolonged heating, volatilises slowly. (See also J. Chem. Soc., Mar., 1918.)—E. H. R.

*Polish; Italian leucitic lavas a source of —.*  
H. S. Washington. Met. and Chem. Eng., 1918, 18, 65—71.

ATTENTION is drawn to the leucitic lavas of the Italian volcanoes which form perhaps the most extensive accumulation of potash-containing igneous rocks on the earth's surface. Leucite ( $\text{KAlSi}_2\text{O}_6$ ) contains theoretically 21.5%  $\text{K}_2\text{O}$  and is readily decomposed by acids, whereas orthoclase feldspar contains only 16.9%  $\text{K}_2\text{O}$  in a form extremely resistant to processes of extraction. The leucite trachytes—essentially soda feldspar and leucite—are the richest of the lavas and contain on an average about 10%  $\text{K}_2\text{O}$ , while the average  $\text{K}_2\text{O}$  content of 44 lavas analysed, including non-leucitic kinds, was 8.31%. Seven Italian volcanoes characterised by leucitic lavas have been examined geologically and chemically, their cubical content calculated and probable quantity of  $\text{K}_2\text{O}$  therein. They are Bolsena, Vico Volcano, Bracciano, Alban Hills, Hermian Volcanoes, Rocca Monfina, and Vesuvius—the only one still active. It is estimated that they contain 105,340 million metric tons of leucitic lavas containing between 7.5% and 9%  $\text{K}_2\text{O}$ , or 8786 million tons of  $\text{K}_2\text{O}$ . The situations are favourable to exploitation and transport facilities are good.—H. J. H.

*Oceanic salts; A complete review of solutions of —.* III. E. Jänecke. Z. anorg. Chem., 1918, 102, 41—65.

The effect of water or, which amounts to the same thing, sodium chloride, as a variable on the equilibrium diagram for the system  $(\text{Na}_2, \text{Mg})(\text{Cl}_2, \text{SO}_4)$  is considered with respect to the simple salts  $\text{MgCl}_2$ ,  $2\text{KCl}$ ,  $\text{Na}_2\text{SO}_4$ , represented by the corners of a triangle in the original diagram, and also with respect to the three double systems represented by the sides of the triangle. (See also J. Chem. Soc., 1918.)—E. H. R.

*Bleaching powder; The best proportions for the manufacture of — in mechanical apparatus.*  
H. Ditz. Z. anorg. Chem., 1918, 102, 66—80.

THE quality of the bleaching powder produced in mechanically operated plant is influenced by the concentration of chlorine in the incoming gas mixture and by the presence of water. The most favourable chlorine concentration is 10—12% to obtain a bleaching powder containing 36—37% of available chlorine, the diluting air serving to moderate the reaction velocity and to conduct away the heat of reaction. The gas mixture must be dried by passing through sulphuric acid, a wet gas causing rise of temperature and consequent decomposition of the bleaching powder. The slaked lime used, however, should contain 5—6% excess (hygroscopic) moisture (see Schütz, this J., 1917, 546.) The function of this water appears to be to cool the reaction mixture by its own evaporation. Supposing the air leaving the plant at 40° C. to be saturated with water vapour, a simple calculation shows that about 21% of the heat produced by the reaction is carried off by this means, the remainder being lost by conduction and radiation. The 5—6% hygroscopic moisture in the lime is not sufficient, however, to saturate the air, but there is sufficient water set free by the first phase of the reaction, namely

$2\text{Ca}(\text{OH})_2 + \text{Cl}_2 = \text{CaO} \cdot \text{Ca}(\text{Cl})\text{OCl} \cdot \text{H}_2\text{O} + \text{H}_2\text{O}$ .  
to make up the deficiency. If, however, the lime were dry at the start, water would be obtained from the above basic compound, bringing about its decomposition with liberation of chlorine.

The initial water in the lime therefore serves to prevent this secondary decomposition. It appears possible that, by using lime containing up to 8—9% excess water at the start, a better bleaching powder containing up to 39—40% of available chlorine might be obtained, but apparently with so much water the lime is liable to form lumps into which the chlorine cannot permeate. The conditions obtaining in the chamber process are different, since here the lime is not in motion. If diluted Deacon chlorine is used, the gas must be dried, according to Hurter. Carbon dioxide appears to have a more deleterious effect in the mechanical apparatus than in the chamber process. The decomposition of bleaching powder by carbon dioxide is favoured by high temperature and the presence of water, and these conditions obtain in the higher tubes of the mechanical apparatus.—E. H. R.

*Boron; Preparation of amorphous —.* W. Kroll. Z. anorg. Chem., 1918, 102, 1—33.

THE most satisfactory method of reducing boron trioxide to amorphous boron is the magnesium-thermite process with addition of sulphur. The reaction mixture consists of 1 part each of magnesium and boron trioxide and 0.8 part of flowers of sulphur, the reaction being carried out in a fireclay or magnesia crucible. The product contains over 90% boron, the chief impurity being magnesium boride. The purest form of boron was obtained by subjecting a mixture of boron trichloride and hydrogen to the action of a high-tension arc, using platinumised copper electrodes. The process is impracticable on a large scale. A sample made by this process contained 98.8% boron and had the sp.gr. 1.731. By heating boric acid with magnesium nitride, a boron nitride was obtained having the composition  $\text{B}_3\text{N}$ , whilst BN was obtained by the action of calcium cyanide on boron trioxide. (See also J. Chem. Soc., Mar., 1918.)—E. H. R.

*Filtration of silica [in analysis].* Nicolardot and Koenig. See X.

*Borax as destructive of vegetation.* Roberts and others. See XVI.

*Gravimetric determination of sulphuric acid and barium as barium sulphate.* Karaoglanow. See XXIII.

#### PATENTS.

*Sulphuric acid; Process of manufacturing —.*  
M. L. Hanahan, Dothan, Ala. U.S. Pat. 1,253,238, Jan. 15, 1918. Date of appl., Mar. 2, 1917.

IN a sulphuric acid plant a dust chamber is provided, communicating with the pyrites furnace or roaster, and sulphur dioxide is produced in the dust chamber by burning sulphur or sulphur-containing material.—B. V. S.

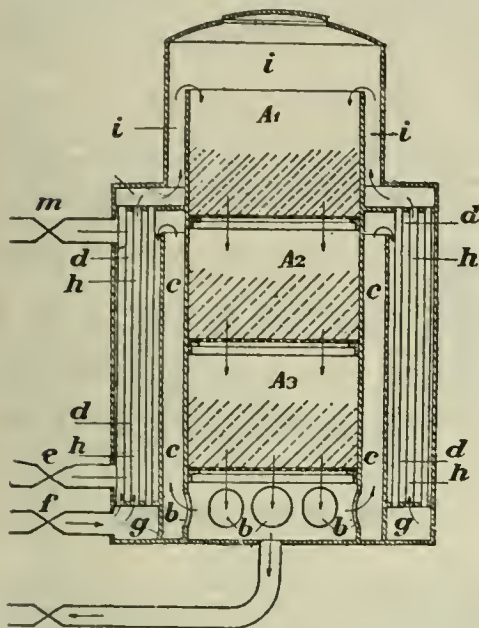
*Nitric acid from ammonia; Process for making —.*  
A. A. Wells, Caldwell, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,252,976, Jan. 8, 1918. Date of appl., Mar. 7, 1917.

IN a process of oxidising ammonia to produce oxides of nitrogen, with a minimum formation of nitrogen, a mixture of ammonia and air is passed through a heated chamber the interior surfaces of which are coated with catalytic material having oxidising properties. A series of superposed chambers or compartments may be employed in which the gaseous mixture is caused to pass through layers of the catalytic material in alternate compartments. In this case the more highly oxidised gas product and that from less advanced stages of reaction are caused to traverse adjacent chambers to effect an interchange of heat.

—W. E. F. P.

**Sulphuric anhydride; Apparatus for the catalytic preparation of —.** W. J. Mellersh-Jackson, London. From Bombrini Parodi-Delfino, Rome. Eng. Pat. 113,017, Mar. 1, 1917. (Appl. No. 3054 of 1917.)

THE reacting gases pass into the reaction chambers ( $A_1, A_2$ , and  $A_3$ ) through the valve,  $f$ , chamber,  $g$



pipes,  $h$ , and chamber,  $i$ , and the waste gases pass out through  $b$  and  $c$  to one or both of the valves,  $m$  and  $e$ . By regulation of these two valves and thus of the amount of waste gases passing through  $d$ , the heating of the gases in pipes,  $h$ , is controlled.—B. V. S.

**Chemical reactions [synthesis of ammonia]; Process and apparatus for accelerating —.** W. A. Schmidt, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,252,726, Jan. 8, 1918. Date of appl., June 22, 1916.

NITROGEN and hydrogen are caused to combine in a reaction chamber to form ammonia gas, which passes with the uncombined nitrogen and hydrogen to another chamber containing a substance which combines with ammonia to form a cloud of suspended particles. The suspended particles are precipitated by the application of an electrical difference of potential, and deposited, and the uncombined nitrogen and hydrogen are returned to the reaction chamber for further treatment.

—W. F. F.

**Polash and other values; Process for extracting — from tobacco.** W. H. Kennedy and G. E. Bailey, Los Angeles, Cal., Assignors to American Safety Explosives Co. U.S. Pat. 1,253,497, Jan. 15, 1918. Date of appl., Mar. 19, 1917.

TOBACCO is treated with alcohol and the solution removed. The residue is distilled to extract gums and resins, and the final residue roasted with charcoal so as to produce potassium carbonate, which is removed by leaching with hot water.

—W. F. F.

**Polash extraction [from felspar].** E. L. Anderson, Pittsburgh, Pa. U.S. Pat. 1,253,560, Jan. 15, 1918. Date of appl., Apr. 8, 1916.

FELSPAR or the like is ground and mixed with an insoluble electrically conducting material such as

carbon. The mixture is electrolysed in a solution containing a substance which combines with the potash to convert it into a form which is easily electrolysed.—W. F. F.

**Aluminous compounds; Production of —.** W. B. Llewellyn, H. Spence, and P. Spence and Sons, Ltd., Manchester. Eng. Pat. 112,881, May 11, 1917. (Appl. No. 6687 of 1917.)

SMALL lumps of bauxite, e.g., such as will pass through a mesh of  $\frac{1}{2}$ -in. or  $\frac{3}{4}$ -in., are placed in suitable vessels and solutions of sulphuric acid or of aluminium sulphate (containing a higher ratio of  $SO_3$  to  $Al_2O_3$  than is desired) obtained from previous operations are caused to circulate through the mass. The vessels are fitted with false bottoms through which the liquor percolates, the bauxite acting as a filter for suspended particles. The bauxite is preferably treated first with a solution of aluminium sulphate containing little or no free acid, then with solutions containing progressively more free acid, and finally with weak liquors and water, these last being used to dilute the sulphuric acid for further operations. Before treatment, the bauxite is usually ignited at about a low red heat, although this is not necessary if it contains a very high percentage of silica. By adopting the above procedure, extraction of the bauxite is more complete than by the old method; cost of powdering the bauxite is avoided; varieties of high silica content can be economically dealt with, in fact, are preferable; filter-pressing of the residue is avoided; solutions in a highly basic condition can readily be obtained without increased loss of alumina in the residue; and the solutions obtained are substantially free from sediment. The solutions may be purified from iron or converted into saleable form, or may be used for preparing other compounds of aluminium. The bauxite may be placed in vessels together with calcined unground shales and the process continued as described in Eng. Pats. 3805 and 22,950 of 1912 (this J., 1913, 363, 1066).—L. A. C.

**Alumina; Art of producing —.** M. Barnett and L. Burgess, New York. U.S. Pat. 1,252,384, Jan. 8, 1918. Date of appl., Dec. 18, 1916.

A SOLUTION of aluminium sulphate is evaporated until the boiling point of the liquid is just below  $120^\circ C.$ , after which the heating is continued, with agitation, until the anhydrous sulphate is obtained. The latter is then decomposed by heating at  $550^\circ$ – $650^\circ C.$ , alumina being produced and the sulphur recovered as sulphuric acid.—W. E. F. P.

**Aluminium sulphate [from solutions of aluminium sulphate containing iron]; Art of producing —.** M. Barnett and L. Burgess, New York. U.S. Pat. 1,252,648, Jan. 8, 1918. Date of appl., Dec. 18, 1916.

THE iron in solution is oxidised by means of ozone or ozonised air, then precipitated as hydroxide and separated by filtration.—W. E. F. P.

**Aluminium nitride; Art of producing —.** M. Barnett and L. Burgess, New York. U.S. Pat. 1,252,649, Jan. 8, 1918. Date of appl., June 1, 1917.

ALUMINIUM carbide is treated with nitrogen at  $900^\circ$ – $1200^\circ C.$ , and the product allowed to cool in the gas.—W. E. F. P.

**Sodium ferrocyanide; Process of making —.** A. R. Tillinghast, Assignor to Semet-Solvay Co., Solvay, N.Y. U.S. Pats. (A) 1,252,741 and (B) 1,252,742, Jan. 8, 1918. Dates of appl., Dec. 20, 1916 and Jan. 10, 1917.

(A) IN the recovery of cyanogen from coal gas, after the removal of ammonia, the gas is scrubbed with a solution of soda ash containing ferrous



carbonate in suspension. The scrubbing medium is then treated with milk of lime and filtered, the solution being concentrated for the recovery of sodium ferrocyanide by crystallisation. (B) The mother liquor from the crystallisation (containing sodium ferrocyanide, carbonate, and hydroxide) is treated with ferrous sulphate and the product employed for scrubbing a further quantity of gas. —W. E. F. P.

*Alkali perborates; Process of making* —. O. Liebknecht, Frankfort, Germany, Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,253,060, Jan. 8, 1918. Date of appl., May 31, 1916.

A SOLUTION of alkali borate and alkali carbonate, kept saturated with both salts, is electrolysed with a current density approximately the same at both cathode and anode.—B. V. S.

*Reducing agent [containing ferrous and calcium sulphides] and method of producing same.* C. S. Bradley, New York. U.S. Pat. 1,253,775, Jan. 15, 1918. Date of appl., Mar. 13, 1913. Renewed June 7, 1917.

THE reducing agent is a mixture of calcium and ferrous sulphides, containing also calcium sulphate, produced by heating together to a low red heat pyrites and burnt lime.—B. V. S.

*Radio-active mineral-water salts; Method of manufacturing* —. J. H. H. Jacobsen, Herlev, Denmark. U.S. Pat. 1,253,830, Jan. 15, 1918. Date of appl., May 8, 1915.

A SOLUTION of a radium salt and a salt of the mineral water which does not precipitate radium is evaporated to dryness. The resulting salt is freed from water of crystallisation and pulverised with the other salt constituents of the mineral water.—B. V. S.

*Alunite; Separation of — from associated rock and/or gangue.* C. F. Hagedorn, Chicago, Ill., Assignor to Mineral Products Corporation, New York. U.S. Pats. (A) 1,253,590 and (B) 1,253,591, Jan. 15, 1918. Dates of appl., Nov. 13, 1915, and Oct. 26, 1917.

(A) To separate rhyolite or the associated rock or gangue from alunite, the mixture is crushed to one-quarter inch mesh and calcined or roasted in a rotary inclined kiln at a low temperature and at such a rate that the specific gravity of the alunite is lowered, but that of the rock or gangue is not affected, and volatilisation of the potash does not take place. The soluble potash salt is extracted and the rock or gangue finally separated by gravity from the undissolved calcined alunite. (B) Silica or silicious minerals such as rhyolite or the like are separated from alunite by roasting or calcining the mixture in a rotary kiln by means of an injected fuel not containing silicon. The silicious material is then separated from the calcined alunite.—W. F. F.

*Borosilicates; Manufacture of* —. R. Boehringer, Newark, N.J., U.S.A. Eng. Pat. 104,008, Feb. 9, 1917. (Appl. No. 2003 of 1917.) Under Int. Conv., Oct. 26, 1915.

SEE U.S. Pat. 1,205,509 of 1916; this J., 1917, 83.

*Aluminium chloride; Art of producing* —. Standard Oil Co., Assignees of M. Barnett and L. Burgess, New York. Eng. Pat. 109,790, July 23, 1917. (Appl. No. 10,554 of 1917.) Under Int. Conv., Aug. 14, 1916.

SEE U.S. Pat. 1,218,588 of 1917; this J., 1917, 456.

*Alkali perborates; Process of making* —. O. Liebknecht, Frankfort, Germany, Assignor to The Roessler and Hasslacher Chemical Co. U.S. Pat. 1,253,061, Jan. 8, 1918. Date of appl., May 31, 1916.

SEE Eng. Pat. 101,620 of 1916; this J., 1917, 137.

*Crude ammoniac; Treatment of the residue from gas purification known as* —. L. C. Bouneau, St. Denis, and V. E. Hasenfratz, Levallois-Perret, France. U.S. Pat. 1,253,571, Jan. 15, 1918. Date of appl., Apr. 21, 1914.

SEE Fr. Pat. 468,535 of 1913; this J., 1914, 962.

*Nitrogen; Process of producing — simultaneously with oxides of nitrogen.* M. Rohmer, Gersthofen, Assignor to Farbwerke vorm. Meister, Lucius, u. Brüning, Höchst, Germany. U.S. Pat. 1,253,534, Jan. 15, 1918. Date of appl., Apr. 16, 1914.

SEE Addition of Apr. 17, 1914, to Fr. Pat. 453,845 of 1913; this J., 1915, 552.

*Hydrogen; Manufacture of pure* —. E. B. Maxted, Walsall. U.S. Pat. 1,253,622, Jan. 15, 1918. Date of appl., July 17, 1916.

SEE Eng. Pat. 12,698 of 1915; this J., 1916, 1060.

*Means for making sulphite acid liquor and the like.* U.S. Pat. 1,251,533. See V.

## VIII.—GLASS; CERAMICS.

### PATENTS.

*Furnace-lining material and the process of producing same.* F. A. Jones, Lakewood, Ohio, U.S. Pat. 1,251,535, Jan. 1, 1918. Date of appl., Nov. 15, 1915.

A MATERIAL containing magnesium oxide, e.g., dolomite, is crushed so that the particles will pass through a screen of  $\frac{1}{2}$  in. mesh but not through one of  $\frac{3}{4}$  in. mesh. The granulated material is mixed with a wet paste containing a substance which is not acted on by moisture, e.g., furnace flue dust, and the mixture is heated until the dolomite is calcined and the flue dust adheres to the surface of the calcined dolomite.—J. F. B.

Kiln. U.S. Pat. 1,251,333. See I.

## IX.—BUILDING MATERIALS.

*Cement; The effect of calcium sulphate on* —. J. C. Witt and F. D. Reyes. Philippine J. Sci., 1917, 12 A, 133—143

THE addition of calcium sulphate, either as gypsum or plaster of Paris, to cement, to the extent of 1.5 to 2%  $\text{SO}_3$ , had the effect of retarding the setting time without decreasing the tensile strength; further additions again accelerated the time of setting and also caused a decrease in tensile strength. Usually the loss of strength was noticeable with anything higher than 2%  $\text{SO}_3$ , but with certain samples there was no loss until about 5%  $\text{SO}_3$  was present. The percentage loss was greater with mortar than with the neat cement, and, with the latter, decreased after seven days. The soundness of the cement (five hours in steam) was unaffected by any addition investigated, i.e., up to about 10%  $\text{SO}_3$ . The tests on the strength of the cements have so far been carried out over a period extending to ninety days, but additional briquettes have been made for testing over periods ranging from six months to five years. The influence of carbon dioxide on the setting time was tested by the method of Hentschel (this J., 1912, 387), and no relation between the  $\text{SO}_3$  content and the effect of exposure to carbon dioxide was apparent. It

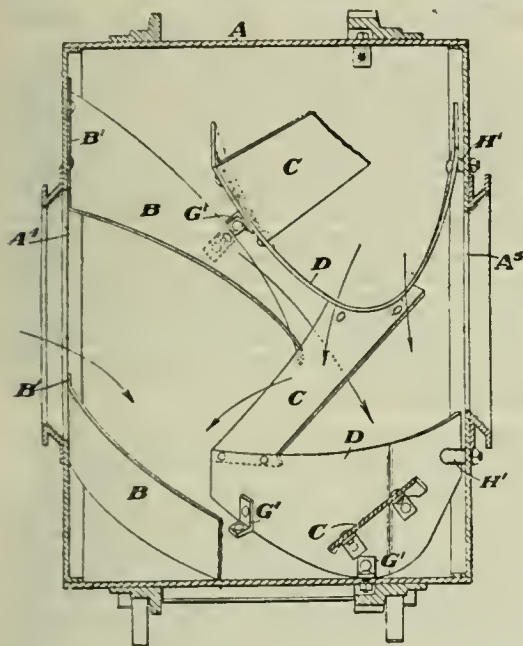
was also shown that the formation of alkali carbonates is not an important factor in the change of physical properties of cement during storage.

—L. A. C.

#### PATENTS.

*Concrete and the like; Machine for mixing* —. Ransome Concrete Machinery Co., New York, Assignees of A. W. Ransome, Plainfield, N.J., U.S.A. Eng. Pat. 107,575, Mar. 28, 1917. (Appl. No. 4505 of 1917.) Under Int. Conv., June 30, 1916.

A ROTATING drum, A, is provided with tapering blades, B, resembling propeller blades, attached to the end wall at B', and extending obliquely to



points near the axis. Blades, D, of stirrup shape, tapering towards both extremities, are secured to the casing at G' and H'. Further blades, C, are attached at their two ends to the inner extremity and the middle portion respectively of two adjacent blades, D, as shown, in such a way that they are substantially horizontal at their lowest position. The material to be mixed is fed into the opening, A', on to the blades, B, and part of this material falls from the narrow inner ends on to the blades, C, and passes thence to the scoop blades, D. A complex movement is given to the materials during mixing, and the mixture is discharged at the opening, A'', by inserting a suitable shoot to catch it as it falls from the blades, D.—W. F. F.

*Road-surfacing material; Process of producing* —. G. Ross, Kansas City, Mo. U.S. Pat. 1,252,722, Jan. 8, 1918. Date of appl., July 21, 1917.

EARTHY material is mixed with water to produce a thin mud, and bitumen and a solvent for the latter added, the mixture being agitated. Crushed rock, gravel, or sand is then added and the mixture compacted on a roadway. In a modification, the earthy material, water, and bitumen are mixed and spread on the road, and after drying, the bitumen solvent is applied to the surface.—W. F. F.

*Wood and other cellulose-containing materials; Art of treating* —. G. W. Herbein, San Francisco, Cal., U.S.A. Eng. Pat. 113,209, June 1, 1917. (Appl. No. 7869 of 1917.)

SEE U.S. Pat. 1,235,895 of 1917; this J., 1917, 1014.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steel projectiles; Principal defects in — and method of avoiding them.* G. Sirovich. *Annali Chim. Appl.*, 1917, 8, 122—158.

IN Italy hard steel intended for certain munition purposes is required to have a tensile strength exceeding 80 kilos. per sq. mm. In the case of ordinary steels poor in manganese and silicon, an increase in the carbon content beyond a certain limit increases the hardness but reduces the tensile strength. Hence to produce a steel of high strength, which can also be worked readily, it is necessary to raise the proportion of manganese, whilst restricting that of the carbon. Steel rich in manganese shows less segregation than a corresponding steel of low manganese content; it also is less liable to deterioration due to overheating during heat treatment. Common steels with high manganese content are largely composed of a series of mixed crystals which solidify within narrow limits of temperature, and such steel is not liable to deteriorate during heat treatment. In Italy, steel for projectiles is made almost exclusively in the basic Martin furnace. In addition to pure aluminium, alloys of iron with various elements are used for the de-oxidation of the steel, and especially ferro-manganese with 50% Mn, or ferro-silicon with 50% Si. The de-oxidation of steel with high manganese content is more troublesome than that of steel poor in manganese, and care is required to prevent the occlusion of products which are not able to reach the surface before complete solidification of the ingot. In general, the main difficulty in the manufacture and working of hard steel is the presence of minute particles of slag ("sonims"), such as silica, iron silicate, and manganese silicate. The use of ferro-silicon gives a less oxidised melt, and produces phases in which basic elements predominate to a less extent than when manganese alone is used. It is practically impossible to eliminate "sonims" from steel with high manganese content, but their occlusion may be restricted by regulating the reactions which take place in the production of the steel.—C. A. M.

*Silicon; Determination of — in ferrosilicon.* P. Nicolardot and J. Koenig. *Rev. Mét.*, 1917, 14, 772—775.

0.470 GRM. of powdered ferrosilicon is mixed with 3 grms. of soda-nitre mixture (5:2) and transferred to a platinum crucible in which 2 grms. of the fusion mixture has been melted, so as to protect the metal. Heat is applied gradually until a violent reaction sets in; the heat is then raised until quiet fusion is attained (10 minutes). The subsequent operations are the same as for the determination of silica in silicates, with the precautions recommended in the following abstract.

—W. R. S.

*Silica; Filtration of — [in analysis].* P. Nicolardot and J. Koenig. *Rev. Mét.*, 1917, 14, 767—771.

THE experiments were undertaken in order to test new filters made in France (Brunin and Durieux brands), taking into account Le Chatelier's views according to which silica separated by an acid is quite insoluble but cannot be quantitatively recovered because, being in a state of extreme subdivision, it passes through the filter; the effect of repeated evaporation may therefore be regarded as to agglomerate the silica rather than to render it insoluble. The first half at least of the filtrate should always be poured back through the filter; only the filtrate itself need be evaporated a second time, not the washings. The quantity of silica lost is independent of the absolute quantity



present; hence the relative error is decreased by operating on a larger amount of material. The quality and kind of filter paper was found not to influence the results, provided a good homogeneous paper was used. Two evaporations with hydrochloric acid in a porcelain dish, followed by a single filtration, the decanted solution being again poured through the filter, were found to give sufficiently accurate results in a reasonably short time.—W. R. S.

[Gold] ores; Cyaniding graphitic.—P. T. Bruhl. Eng. and Min. J., 1918, 105, 197.

THE recovery of gold from graphitic ores by cyaniding does not as a rule exceed 50%, the gold being prematurely precipitated from solution by the graphite. By submitting the ore pulp to flotation, the graphite passes into the froth together with any auriferous pyrite and mispickel. The flotation concentrate is roasted, added to the tailing, and the product cyanided. The tailings from the flotation process are relatively free from carbonaceous matter and pass to the cyanide slimes plant.—W. R. S.

Gold and silver; Metallurgy of.—[in 1917]. H. A. Megraw. Eng. and Min. J., 1918, 105, 102—104.

No development of fundamental importance took place in the metallurgy of gold; existing processes were improved, and economies in working aimed at. The increased price of silver stimulated production, though the Mexican output was only about 40% of the normal figure because of unsettled political conditions and the stoppage of supply of German cyanide. At Pachuca, the substitution of flotation for cyaniding gave promising results, installation and working costs being smaller than for the cyanide process. The flotation concentrate (argentine) is smelted or cyanided. Flotation has also been introduced at Cobalt, where it is a valuable adjunct to concentration and cyaniding, adding 10% and more to the recovery, at a cost of less than 20c. per ton. In crushing the ores, the tendency is to replace stamps by ball mills.—W. R. S.

Silver; Rate of solution of.—in chromic acid. R. G. Van Name and J. U. Hill. Amer. J. Sci., 1918, 45, 54—58.

EXPERIMENTS on the rate of solution of silver in chromic acid solutions containing sulphuric acid show that there is an initial disturbance which is attributed to the physical state of the metal. The rates at which cadmium and silver dissolve become more nearly equal with diminishing sulphuric acid concentration. (See also J. Chem. Soc., March, 1918.)—H. M. D.

Copper; Metallurgy of.—[in the United States in 1917]. A. L. Walker. Eng. and Min. J., 1918, 105, 94—96.

ONE new plant was put in commission, viz., the New Cornelia Copper Co.'s leaching and electrolytic plant at Ajo, Ariz. The ore is leached with 3% sulphuric acid, and the latter regenerated; part of the regenerated solvent must be discarded, to prevent the accumulation of iron and aluminium. The Utah Copper Co. is installing a similar plant for the oxidised ores of Bingham, Utah. The ammonia leaching plants at Calumet and Kennecott (Alaska) are being greatly enlarged. Flotation for fines and slimes has now been introduced at all the large concentrating plants.—W. R. S.

Zinc; Metallurgy of.—[during 1917]. W. R. Ingalls. Eng. and Min. J., 1918, 105, 96—99.

AT Bartlesville, Okla., a process is about to be put in operation which consists in distilling the roasted ore with a small proportion of fuel. The residue

rich in zinc and lead will be burned on Wetherill grates. Several large smelters have installed oxide plants to recover zinc from distillation residues. A 60% recovery can be made from residues containing 6% Zn without the use of any fuel other than that present in the residues. The manufacture of high-grade zinc dust, prepared by atomising the metal with compressed air, has been started by the Anaconda Copper Co., the New Jersey Zinc Co., and several other companies. Several new electrolytic extraction works started operations, but none attained large production. Much spelter was again refined by re-distillation, the refined metal containing as little as 0.10% Pb.—W. R. S.

Lead; Metallurgy of.—[in 1917]. H. O. Hofman. Eng. and Min. J., 1918, 105, 93—94.

MIXED lead-zinc ores are being treated by sulphatizing roasting followed by leaching with sulphuric acid. The solution is purified, nearly neutralised, and electrolysed with lead anodes and aluminium cathodes; the yield up to the present is only 60%. A new smelting works started operation at Kellogg, Idaho; it comprises one Wedge roaster, 3 Dwight-Lloyd sintering machines, and 3 blast furnaces (48×180 in.). Leaching experiments at Kellogg (chloridising roast and leaching with acidulated brine) gave a yield of 85—95% lead and 80—90% silver. An automatic bag filter is in operation at Depue, Ill. The bags of each division are alternately inflated and deflated as well as subjected to an up-and-down and undulating motion. The cleaning of the cloth is thus accelerated and the filtering capacity increased.—W. R. S.

Mercury; Metallurgy of.—[in 1917]. M. Innes. Eng. and Min. J., 1918, 105, 110—111.

THE production of mercury in the United States has been steadily declining for 40 years, due chiefly to inability to compete with the European output, and to the poor grade of ore now mined in California, the average recovery being 6 lb. per ton of ore. The chimney losses of the Scott furnace (which produces 95% of the metal in California) were found to be negligible. In spite of its rather high first cost and moderate capacity, this furnace has the advantage of general efficiency and low fuel cost, and treats coarse (2-in. mesh) ore, thus avoiding the drawbacks of fine grinding. A new rotary furnace of the cement-kiln type, which is being installed by the New Idria Co., will, it is expected, handle an increased tonnage at lower cost.—W. R. S.

Flotation; Progress of.—in 1917. H. A. Megraw. Eng. and Min. J., 1918, 105, 99—102.

IN the Joplin district (Missouri), the use of a mixture of turpentine and rosin was tried; 1—5 lb. of mixture (rosin 20—30, turpentine 80—70%) per ton of ore assaying 1—2% of zinc in an acid pulp (ratio 5:1), gave rough concentrates containing 37.66% of zinc, the recovery being 50—80%. Cold solutions give a higher recovery than warm, but the grade of product is not so good. The introduction of flotation in Missouri lead milling has not greatly altered local practice. It is applied to galena finer than 200-mesh, as the tables deal efficiently with coarser sizes. The frothing agent is creosote: the tailing from the first treatment is re-treated, giving final tailings. At Humboldt, Ariz., the advent of flotation rendered treatment of much poor copper ore possible, while improving the recovery in concentration and the grade of concentrate. With clean sulphides, the recovery sometimes reaches 95%. The Murex process is used at Darwin, Cal., for the concentration of lead carbonate ores, at a cost of \$1.78 per ton. In this process a magnetic material is mixed with oil to form a paint, which is agitated with the ore to be



treated. The paint adheres to the particles of valuable mineral and the latter are recovered by means of a magnetic separator. The advantage of the process is that the ore need only be ground fine enough to liberate the particles of ore from gangue.—W. R. S.

*Flotation: Effect of addition agents in —. II.* M. H. Thornberry and H. T. Mann. *Met. and Chem. Eng.*, 1918, 18, 71—77. (See also this J., 1918, 93 A.)

THE experiments recorded in the previous paper have been extended to include hydroxides and nitrates. The hydroxides employed—alkalis and alkaline earths—behaved on the whole similarly to the sulphates, the relative extractions being practically the same, but the grade of concentrate slightly lower. Calcium hydroxide had most effect on the character of the froth. The results in presence of nitrates depended largely on the oil used and showed great divergence. With the flotation oil No. 1, the extraction was scarcely altered, while the grade of concentrate was lowered about 17%. With the Naval Stores oil No. 17 and with cresylic acid, the extraction was lowered slightly, but the grade of concentrate was raised slightly with the latter and lowered with the former. Mercury and uranyl nitrates were deleterious, but most of all cadmium nitrate. Nitrates exerted considerable effect on the character of the froth. Nitric acid caused such copious frothing that the total quantity of oil could not be added, and the experiments with it had to be abandoned.

—H. J. H.

*Tellurium and aluminium; Metallographic investigation of the system —.* M. Chikashigi and J. Nosi. *Mem. Coll. Sci.*, Kyoto, 1917, 2, 227—232.

ALUMINIUM and tellurium form the compounds  $Al_2Te_3$  and  $Al_3Te_2$ . The reaction which occurs when aluminium and tellurium are heated together is very violent, and in the case of mixtures which contain 50 to 100% Te it is advisable to melt the tellurium and add the aluminium in small portions at a time. The compound,  $Al_2Te_3$ , melts at 895° C. and forms mixed crystals with tellurium which exist in two different forms. The eutectic points at 414° and 621° C. correspond with 2.8 and 97% Al respectively. Conglomerates which consist of the second eutectic mixture and aluminium or the telluride,  $Al_3Te_2$ , undergo transformation when the temperature falls to 551° C., with the formation of  $Al_2Te_3$ . Both compounds are decomposed by water with the liberation of hydrogen telluride. (See also J. Chem. Soc., Mar., 1918.)—H. M. D.

*Selenium and antimony; Metallographic investigation of the system —.* M. Chikashigi and M. Fujita. *Mem. Coll. Sci.*, Kyoto, 1917, 2, 233—237.

ANTIMONY and selenium combine to form the selenide,  $Sb_2Se_3$ , which melts at 572° C. The eutectic at 497° C. on the antimony side corresponds with 46.5% Se, and on the selenium side coincides with the freezing point of selenium. The results of micro- and thermal analyses are in agreement. (See also J. Chem. Soc., Mar., 1918.)

—H. M. D.

*Cadmium and selenium; Metallographic investigation of the system —.* M. Chikashigi and R. Hikosaka. *Mem. Coll. Sci.*, Kyoto, 1917, 2, 239—244.

WHEN cadmium and selenium are heated to a sufficiently high temperature, the compound,  $CdSe$ , is formed. The selenide is infusible up to 1350° C., and has sp.gr. 5.81 at 10° C. It does not dissolve in either of the molten elements, which

are themselves practically immiscible. The micro-structure of solidified melts is illustrated by photographs. (See also J. Chem. Soc., Mar., 1918.)

—H. M. D.

*Zinc and selenium; Metallographic investigation of the system —.* M. Chikashigi and R. Kurosawa. *Mem. Coll. Sci.*, Kyoto, 1917, 2, 245—248.

ZINC selenide,  $ZnSe$ , is formed when the two elements are heated together. It is infusible up to 1100° C., is brilliant yellow in colour and has sp.gr. 5.29 at 21° C. The two fused elements are immiscible with each other and with the compound. (See also J. Chem. Soc., Mar., 1918.)—H. M. D.

*Aluminium and selenium; Metallographic investigation of the system —.* M. Chikashigi and T. Aoki. *Mem. Coll. Sci.*, Kyoto, 1917, 2, 249—254.

ACCORDING to cooling curve observations, aluminium and selenium combine to form the compound  $Al_2Se_3$ , which melts at about 950° C. This compound crystallises out from all fused mixtures of the two elements, and the eutectics correspond with pure aluminium and pure selenium respectively. Photomicrographs are reproduced which show the structure of the crystalline regulus obtained from various fused mixtures. The combination of aluminium with selenium is very violent and an explosion occurs readily when mixtures containing up to 80% Se are heated together. (See also J. Chem. Soc., Mar., 1918.)

—H. M. D.

*Acidity determination in water, leach liquors, mine waters, etc.* Rankin. See XXIII.

#### PATENTS.

*Iron; Alloy of —.* O. B. McMillin, Assignor to Pittsburgh Rolls Corporation, Pittsburgh, Pa. U.S. Pat. 1,252,596, Jan. 8, 1918. Date of appl., July 29, 1916. Renewed May 26, 1917.

THE alloy contains Si 0.25 to 1.0% (e.g., 0.5%), Cr 0.25 to 3.0% (1%), S 0.05 to 2.0% (0.12%), P 0.05 to 2.0% (0.08%), Mn 0.20 to 1.0% (0.3%), total C 1.5 to 3.5% (2.4%), Fe 95.6%.—T. H. B.

*Steel; Welding and annealing high speed —.* E. J. von Henke, Assignor to Thomson Electric Welding Co., Lynn, Mass. U.S. Pat. 1,252,746, Jan. 8, 1918. Date of appl., May 14, 1917.

AFTER electrically welding high-speed steel to machinery steel, the clamps on the high-speed steel are opened, and a heating current is immediately passed through from a point at the rear of the clamped section to and through the weld to bring the high-speed end of the tool to the desired annealing temperature.—T. H. B.

*Steel, iron, or other magnetisable metal; Method of and means for indicating the condition of — during heat treatment.* L. W. Wild and E. P. Barfield, London. Eng. Pat. 112,894, June 26, 1917. (Appl. No. 9170 of 1917.)

IN an apparatus for determining the temperature to which steel or other magnetic metal should be heated for hardening, tempering, and annealing, by means of an indication depending on the magnetic properties of the metal in a magnetic field at that temperature, the current which generates the field is also used to heat the metal. The metal is placed in a receptacle of silica, partly filled with a molten salt such as a mixture of potassium and sodium chlorides. A heating and magnetising coil is wound round the receptacle and connected to a pair of metal bands also surrounding it. The receptacle is placed in an outer vessel and the intermediate space filled with a



heat and electrical insulating substance, such as quartz powder. A secondary coil is wound on the outer vessel and two pairs of metal bands or clips are provided on this vessel to which the primary and secondary coils are connected. A galvanometer or other suitable recorder is included in the secondary circuit. When the metal reaches the temperature of decalescence it becomes non-magnetic and the consequent change in the magnetic field induces a current in the secondary coil which is indicated by the galvanometer or other apparatus.—W. F. F.

*Manganese steel articles; Method of and apparatus for the heat treatment of*—F. Schaffer, Leobersdorff, Austria. Eng. Pat. 112,986, Feb. 1, 1917. (Appl. No. 1655 of 1917.)

ARTICLES made of steel containing about 1% of carbon and more than 12% of manganese are heated to 1000°—1100° C. and cooled in accordance with a definite curve of time and temperature which will produce austenitic structure, and selected to obtain the structure and mechanical properties required. The cooling medium is applied by an automatic electrical device to the various parts in proportion to their area and the quantity of heat absorbed, so that the drop of temperature is uniform. The cooling is done automatically in stages each of which comprises cooling and subsequent equalising of differences in temperature.—W. R. S.

*Furnace; Electrical — for hardening steel and the like.* L. W. Wild and E. P. Barfield, London. Eng. Pat. 112,731, June 26, 1917. (Appl. No. 9169 of 1917.)

THE lower part of a crucible of silica or other suitable material to contain the molten salt bath in which the metal is immersed, is enclosed in a coil or winding and the ends of the coil are connected to metal bands around the crucible. The metal bands are connected to a source of electricity through similar metal bands on an outer casing or jacket, the crucible and jacket being separated by heat-resisting and non-conducting material.

—B. N.

*Crucible furnace.* F. E. Whitham, Sowerby Bridge, Yorks. Eng. Pat. 113,036, Apr. 17, 1917. (Appl. No. 5376 of 1917.)

A CRUCIBLE furnace has circumferential flues having inlets and outlets to each crucible tangential and diagonally opposite each other. Carbon monoxide generated from charcoal in a separate fire-box is supplied through branch pipes into each crucible.—W. R. S.

*Furnace; Smelting*—R. B. Llopart, Assignor to Mineral Refining and Chemical Corporation, St. Louis, Mo. U.S. Pat. 1,253,064, Jan. 8, 1918. Date of appl., Jan. 26, 1917.

A DOWN-DRAUGHT smelting-furnace has connected horizontal tiers of vertically spaced heating-flues, vertical flues to connect opposite alternate ends of the horizontal flues, and discharge flues connecting the lowermost tier of flues with the chimney. Connected horizontal muffles are disposed in tiers alternately and transversely through the spaces between the flues. The muffles are spaced apart one above another and connected at alternate ends to form a continuous zig-zag passage from top to bottom. Combustion chambers are disposed vertically adjacent to and controlling the flues. An ore-port is provided at the top of the furnace for feeding ore into the uppermost muffles; vertical ore-ways connect alternate opposite ends of the muffles, the latter having stoke-doors through which the ore is worked downwards. A flue

admits reaction gases into the lowermost muffles, the gases passing upwards through the muffles in the opposite direction to that of the ore and heat; a vent port at the top of the furnace allows the escape of the reaction gases.—W. R. S.

*Zinc vapours; Condensers for*—Norsk Elektrisk Metalindustri Aktieselskap, Sarpsborg, Norway. Eng. Pat. 105,559, Apr. 5, 1917. (Appl. No. 4955 of 1917.) Under Int. Conv., Apr. 8, 1916.

THE zinc vapour and gases from the distilling chamber pass by a horizontal pipe into the condensing vessel, the top of which just fits over the pipe outlet. This outlet is formed as a short downward branch from the horizontal vapour conduit and is provided with a vertical transverse partition, so that an outlet for the gases from the condenser into the extension of the vapour conduit is formed. The condenser is oscillated angularly about the pipe outlet so that the zinc which condenses as a powder is absorbed into the body of molten zinc at the bottom, the coating of oxide on the particles being detached by the movement.

—W. F. F.

*Slag; Method for utilising the waste heat of*—wherein the slag is granulated by means of air. C. Semmler, Wiesbaden, Germany. Eng. Pat. 112,792, Jan. 6, 1917. (Appl. No. 285 of 1917.)

MOLTEN slag is fed into the middle zone of a vertical tower, is granulated by a jet of compressed air, and passes downward over a series of oppositely inclined plates to a moving horizontal band or grid at the bottom. Air is circulated by a fan upward through the shaft to cool the slag and heat the air, and the hot air passes through the tubes of a boiler in the upper end of the shaft. The cooled air passes downward through a parallel shaft, through and over the granulated slag on the grid at the bottom of the two shafts, and the cycle is then repeated. The air used for granulating the slag is preheated by passing it through a coil in the downward shaft for the hot air, serving at the same time further to cool the latter. The circulating air is thus added to the air from the jet and both pass through the boiler tubes. In a modification, the air heated by the molten slag is used for the heating of Cowper stoves or to preheat the combustion air therefor.—W. F. F.

*Gold and silver; Obtainment of*—from their ores. A. A. Lockwood, London. Eng. Pat. 112,876, Apr. 18, 1917. (Appl. No. 5453 of 1917.)

THE pulped ore is agitated in a cyaniding vessel and then led to one end of a long shallow electrolysis vessel containing a series of parallel, vertical, longitudinal electrode plates. The pulp is kept in suspension by a jet of air and is led from the other end of the electrolysis vessel back to the cyaniding vessel. It is withdrawn from the cyaniding vessel through an open-ended vertical compartment provided with internal baffles to retain coarse particles of ore. The cathodes are preferably steel plates coated with carbon, and the anodes iron plates containing 30–40% Si ("tantiron").—W. F. F.

*Electrode used in the electrolytic process of refining metals.* F. L. Antisell, Perth Amboy, N.J. U.S. Pat. 1,250,757, Dec. 18, 1917. Date of appl., Feb. 8, 1913.

A ROD or bar, for suspending an electrode in an electrolyte, is provided on the under side with V-shaped projections, facing inwards and converging at their lower ends. The bar is also provided with a notch or recess at one end on its under side.—B. N.



*Nickel from copper; Process of electrolytically separating* — G. A. Guess, Oakville, Ontario, Canada. U.S. Pat. 1,251,511, Jan. 1, 1918. Date of appl., Aug. 14, 1917.

NICKEL sulphate solution is electrolysed using a copper-nickel anode, and a substantially insoluble reagent, such as calcium carbonate, which forms with copper an insoluble carbonate, and a sulphate, the presence of which is not detrimental in the electrolyte, is maintained in contact with the anode. The cathode is screened from contact with the carbonate.—B. N.

*Electroplating apparatus.* L. Potthoff, Flushing, N.Y. U.S. Pat. 1,251,568, Jan. 1, 1918. Date of appl., Dec. 14, 1916.

A TUMBLING barrel for the articles to be plated is supported in an electrolyte in a tank, the barrel being made in separable halves clamped together and holding between them a stationary cathode rod with cathode arms. The opposite heads of the barrel are provided with cathode plates having peripheral flanges and central bushings, the cathode rod being supported in the bushings but not free to rotate. Several peripheral cathode tracks of conducting material, continuous within the periphery of the barrel when assembled, support the articles to be plated, and rotate with the barrel to vary the points of contact between the articles and the tracks, the current being conducted from the tracks by the cathode rod. A movable chain engages with pins on the barrel to rotate the latter, and the barrel can be moved longitudinally to disengage the pins from the chain.—B. N.

*Electroplating apparatus.* L. Potthoff, Montclair, N.J., and G. W. Schweinsberg, Brooklyn, N.Y. U.S. Pat. 1,251,569, Jan. 1, 1918. Date of appl., May 12, 1917.

THE tank containing the electrolyte is provided with an endless conveyor for moving the articles through the electrolyte, and comprising links having vertical and horizontal bearing surfaces, which slide on a single rail track, supporting the conveyor. A guide strip engages the links and retains them in place on the track, and hooks, rigid with the conveyor, support the articles to be plated, so that the centres of gravity of the articles are in a vertical line below the track. A series of anodes are arranged in the tank parallel to the line of movement of the articles, and the electrolyte is agitated by a longitudinal pivoted bar near the bottom of the track substantially parallel to the anodes. Means are provided for vertically oscillating the bar backwards and forwards beneath the anode surfaces to cause movement of the electrolyte between the articles and the anodes, and across the line of feed of the articles, so as to renew the electrolyte and penetrate the recesses of the articles.—B. N.

*[Tungsten] wires; Method of lubricating refractory during drawing.* K. Farkas, Assignor to A. B. Lisle, Providence, R.I. U.S. Pat. 1,253,362, Jan. 15, 1918. Date of appl., May 6, 1916.

THE process consists in applying an aqueous paste of magnesium oxide to the surface of the wire, heating it, and passing it through a heated diamond die.—W. R. S.

*Ore concentration; Process of and apparatus for* — O. D. Welsch, Kimberly, Nev. U.S. Pat. 1,253,653, Jan. 15, 1918. Date of appl., May 26, 1917.

THE method consists in agitating ore pulp within a confined space with compressed air, and then causing air under less pressure to pass downwards through and with the agitated pulp into the main body of pulp to form air bubbles in it and a froth

upon its surface. The process is carried out in a spitzkasten having froth and gangue discharges: a compressed air pipe extends into the spitzkasten below the level of the pulp or nearly to its lower end. The pipe discharges into an air lift chamber open at its upper and lower ends, the lower end extending below the lower end of the air pipe; its upper end has a deflector extending outwards and downwards. The air lift chamber is surrounded by an air chamber closed at its upper end, and extending into the space around and below the level of the upper end of the air lift chamber. A valve controls the pressure in the air chamber.—W. R. S.

*Metals; Process of making — in electric furnaces.* E. Humbert, Welland, Ontario, Canada. U.S. Pat. 1,252,443, Jan. 8, 1918. Date of appl., Apr. 26, 1917.

A CHARGE, partly at least in the solid condition, and containing an excess of carbon, is introduced into the furnace, and an arc is struck between an electrode and the solid portion, whilst a current of air is passed into the solid portion only, to oxidise the excess of carbon before the charge is melted. The operation is continued until the solid portion is melted and the molten charge refined to the desired extent.—B. N.

*Furnace; Electric ore* — J. A. Ward, Assignor to E. S. Campbell, W. A. Ridgeway, and H. Sorensen, Spokane, Wash. U.S. Pat. 1,252,635, Jan. 8, 1918. Date of appl., Apr. 12, 1917.

A FURNACE of the shaft type is surrounded by an electric heating coil, and a series of conducting pencils pass through the furnace wall so as to be in contact with the ore within it. An annular conducting band surrounds the crucible of the furnace and is in electrical contact with the furnace contents. A switch is provided for opening a circuit through the coil of wire, and for closing a circuit from the annular conducting band through the ore contents to the conducting pencils, the closing being effected slightly before the opening of the wire coil circuit. The current flowing through the heating coil is controlled by introducing in series into the circuit a number of adjustable arc-forming contacts or electrodes exterior to the heater. Means are provided for automatically adjusting the contacts to effect a regulation of the temperature in the coil of wire, and for utilising the heat generated by the arc or arcs formed between the contacts in aiding the process of ore reduction.—B. N.

*Electro-deposition of metals.* A. G. Betts, Asheville, N.C. U.S. Pat. 1,252,654, Jan. 8, 1918. Date of appl., May 15, 1916.

AN apparatus for the electro-deposition of metals comprises a lead-lined receptacle containing an anode and cathode spaced from the receptacle, with positive and negative terminals connected to the electrodes, and an independent connection, offering a relatively high resistance to the flow of the current, between the lead lining and the negative terminal. A salt of a metal of greater solubility in the acid of the salt than lead, is electrolysed, whilst maintaining at the lead surface a negative potential high enough substantially to prevent the formation of a lead salt.—B. N.

*Fume condenser.* T. B. Stewart, Portola, Cal. U.S. Pat. 1,252,731, Jan. 8, 1918. Date of appl., Jan. 6, 1917.

A HORIZONTAL tank is provided with a chimney at one end and with upper and lower horizontal partitions. At the end of the upper partition opposite the chimney, a cap extends upwards and the gaseous products from the smelter are introduced into the cap near the top. Inclined baffle-



plates project from the inner wall of the chimney and have water-spraying devices at their inner ends. The water is circulated through the tank and around the partitions, and the fumes pass from the cap through the water between the partitions to the chimney.—T. H. B.

*Alloys; Process of making* —. H. L. Doherty, New York. U.S. Pat. 1,252,887, Jan. 8, 1918. Date of appl., Aug. 27, 1917.

ALLOYS of metals of widely different melting points are made by heating the metals, at a temperature sufficient to melt the less fusible metal, in a casing, under sufficient pressure to prevent volatilisation of the more volatile metal. The alloy is cast under pressure.—T. H. B.

*Ore concentration*. R. B. Martin, New York. Eng. Pats. 103,990 and 103,991, Jan. 27, 1917. (Appl. Nos. 1393 and 1394 of 1917.) Under Int. Conv., Jan. 28, 1916.

SEE U.S. Pats. 1,236,856 and 1,236,857 of 1917; this J., 1917, 1052.

*Metals; Electrolytic recovery of — from their solutions*. U. C. Tainton, Johannesburg, Transvaal. U.S. Pat. 1,251,302, Dec. 25, 1917. Date of appl., July 6, 1915.

SEE Eng. Pat. 11,336 of 1915; this J., 1916, 969.

*Electric furnace*. U.S. Pat. 1,252,633. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Furnace; A high temperature electric resistance* —. W. Rosenhain and E. A. Coad-Pryor. Faraday Soc. [Advance proof.] 7 pages.

THE graphite or carbon resistor is in the form of a tube, made up of superposed rings, with conical top and bottom surfaces so that they fit accurately one upon the other; the rings are turned from a solid tube or rod of graphite. The thickness of the rings may be varied so as to increase or diminish the number of rings for a given height of tube, and thus vary the number of zones of contact for varying the resistance and the temperature. The ends of the tube are connected to metallic water-cooled electrodes, and the tube is surrounded by finely-divided carbon, or vegetable black, and magnesia blocks enclosed in a metal case. In a furnace of this type with rings  $2\frac{1}{4}$  in. internal diameter, about 100 heats at temperatures ranging from  $1500^{\circ}$  to  $2400^{\circ}$  C. were obtained in a period of six weeks, and the only repairs then necessary consisted in replacing the top six rings by new ones. In a furnace of  $2\frac{3}{4}$  in. internal diameter a temperature of  $1500^{\circ}$  C. can be attained in 30 mins. with a consumption of 8 kilovolt-amperes, and  $1700^{\circ}$  C. with a consumption of 10 kilovolt-amperes (400 amperes, alternating current, at 25 volts). In a furnace of  $3\frac{1}{4}$  in. diam., pure iron can be melted with a power consumption of 10 kilovolt-amperes taking a current of 550 amperes. In a still larger furnace of 7 in. internal diameter a temperature of  $1650^{\circ}$  C. was attained with a power consumption of 23 kilovolt-amperes (2000 amperes); the lower efficiency is due to the smaller ratio of length to diameter of furnace. —B. N.

### PATENTS.

*Electrolytic apparatus*. L. W. Chubb, Edgewood Park, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,250,141, Dec. 18, 1917. Date of appl., Jan. 10, 1917.

AN electrolytic cell contains a series of superposed horizontal plates of film-forming metal, spaced apart, alternate plates being connected with each other so as to form two distinct sets. The plates

are provided with openings of two different sizes, the larger openings in each plate being so disposed as to register with the smaller openings in the adjacent plates. Rods extend through the registering openings in the plates, and spacing members of film-forming metal surround the rods between the plates, and, being of less diameter than the larger openings in the plates, they extend through these openings and thus allow the plates comprising each alternate set to be rigidly clamped together in spaced relationship.—B. N.

*Electrolytic apparatus*. J. Coulson, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,250,146, Dec. 18, 1917. Date of appl., Jan. 6, 1917.

TWO sets of vertical plates of film-forming metal are employed, and each set is supported and rigidly clamped by horizontal spacing members, the members clamping one set of plates passing through openings in the second set. The spacing members are provided with screw-threaded and tapped openings, whereby several are connected together to form a substantially continuous rod. Means are provided for removing the plates from the container, and for preventing movements of the spacing member rods towards the ends or sides.—B. N.

*Secondary storage battery and method of making the same*. C. S. Palmer, Newtonville, Mass. U.S. Pat. 1,251,412, Dec. 25, 1917. Date of appl., July 23, 1915.

THE electrode is composed of a grid comprising lead supporting plates, approximately 0.12 in. in thickness, with laminations forming lead shelves approximately 0.02 in. in thickness, and spaced apart approximately 0.04 in. Active material containing the "PbO" radical fills the spaces between the shelves. The electrode is immersed in an electrolyte consisting essentially of a concentrated solution of sodium bisulphate over 1.1 sp.gr., containing approximately 4 to 8% of additional sulphuric acid. The material is subjected alternately to the charging and discharging action of an electric current.—B. N.

*Furnace; Electric* —. C. H. Vom Bauer, Douglaston, N.Y. U.S. Pat. 1,252,633, Jan. 8, 1918. Date of appl., Aug. 6, 1917.

THE furnace is provided with a solid bottom and an oval-shaped refractory side wall, of such a form that the end portions in plan are parts of arcs of circles of equal radii but different centres, the middle portion forming the arcs of a circle of a radius  $\sqrt{2}$  times the radius of each end arc. The centres of all the arcs are in line, and three electrodes, located approximately at the centres of the arcs, are connected to a two-phase three-wire current supply. The electrodes are thus spaced, so that, at the slag line, the heat gradient is the same for each electrode at the point of the side wall which is nearest to such electrode.—B. N.

*Electric furnace control apparatus*. J. A. Seede, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,253,732, Jan. 15, 1918. Date of appl., June 21, 1917.

THE control apparatus comprises arc-supporting electrodes, connected to conductors supplying energy, and also connected operatively to motors. Means are provided for controlling the motor connected to one electrode, so as to regulate the length of the arc in response to variations in current in the supply circuit, and to move the electrode in a direction whereby a desired current value is maintained. The second motor responds to voltage changes, and moves the electrode in a direction whereby a desired voltage drop is main-

tained, means being also provided for braking the motors when the desired regulation has been attained.—B. N.

*Art of electrical precipitation of particles from fluid streams.* U.S. Pat. 1,252,101. See I.

*Potash extraction [from felspar].* U.S. Pat. 1,253,560. See VII.

*Electrode used in the electrolytic process of refining metals.* U.S. Pat. 1,250,757. See X.

*Process of electrolytically separating nickel from copper.* U.S. Pat. 1,251,511. See X.

*Process of making alkali perborates.* U.S. Pat. 1,253,060. See VII.

## XII.—FATS; OILS; WAXES.

*Sulphonation of fixed oils.* L. G. Radcliffe and S. Medofski. J. Soc. Dyers and Col., 1918, 34, 22—35.

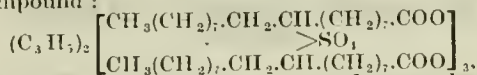
The authors give a summary of the literature of the subject and a bibliography, followed by the results of their own experiments. Various oils were sulphonated, the fatty acids liberated from the sulphonation products as described by Radcliffe and Palmer (this J., 1915, 643), and the characters of these fatty acids compared with those of the fatty acids from the original oils. Sulphonation was effected by adding concentrated sulphuric acid (35, 50, or 75 grms.), drop by drop, to 100 grms. of the oil contained in a jar cooled by ice, the mixture being agitated continually and the temperature not allowed to rise above 15° C. No sulphur dioxide was evolved. After all the acid had been added, the mixture was stirred for a further 2 hours, left at rest for some hours, washed with a saturated solution of sodium sulphate, and heated in a water-oven till clear. The heating in a water-oven caused charring of the products from linseed and rape oils and was omitted in these cases. In the case of sesame, cottonseed, and olive oils, the chief effects of sulphonation on the fatty acids were a rise of the solidifying point, and diminution of the iodine value and neutralisation value; the acetyl value was considerably increased, indicating the formation of hydroxy-compounds, the increase being greater the higher the proportion of olein or oleic acid present. With rape and whale oils similar results were obtained, except that there was no marked increase of the acetyl value, indicating that with these oils there was no notable formation of hydroxy compounds but production of lactones (or anhydrides) or polymerides. The fatty acids from sulphonated linseed oil agree fairly closely in character with those from the original oil, i.e., there is no considerable formation of stable new compounds, either lactones or hydroxy-compounds. With castor oil, also, no new hydroxy compounds are formed, but the lower acetyl value and iodine value of the fatty acids from the sulphonated oil and the difference between the saponification value and neutralisation value of these acids indicate the formation of polyricinoleic acids (see Jaillard, this J., 1892, 355; 1893, 528; 1894, 820). The combining powers of various oils and fatty acids with sulphuric acid or their "sulphonation numbers," were determined by treating 5 grms. of the oil or fatty acid with 25—35% of concentrated sulphuric acid, added drop by drop, at 0°—5° C., allowing to stand for 2 hours, dissolving the product in neutral alcohol, and titrating with alkali. The difference between the amount of alkali required and that which would have been required if no combination had taken place, is a measure of the combining power of the oil with sulphuric acid. The following

values (mgrms. KOH per gm. of oil or fatty acid) have been obtained: oleic acid, 67.75; olive oil, 63.42; rape oil, 53.76; sesame oil, 32.76; whale oil, 26.40; linseed oil, 21.4; palmitic acid, 8.21; stearic acid, 7.63. With the exception of rape oil the order corresponds with that in which the acetyl value of the fatty acids has increased after sulphonation. The figures show that the ease with which oils react with sulphuric acid depends not upon the "degree of unsaturation," but upon the percentage of olein or oleic acid present. Saturated fatty acids are not affected by sulphuric acid. In the case of highly unsaturated oils, such as linseed oil, the action of sulphuric acid is probably either to form unstable addition products (sulpho compounds) or to cause polymerisation, the polymers being easily decomposed. With all oils sulphuric acid acts to some extent as a hydrolysing agent, the liberated glycerol remaining in the mixture, either in the free state or as sulpho-glycerol compounds. In the case of oils containing a large percentage of olein, as for example, olive oil, the action of sulphuric acid is confined (beyond the hydrolysis of the triglycerides) to the olein, which is converted into saturated compounds, viz.,  $\theta$ -tri-olein-hydrogen-sulphate,

$C_3H_5[CH_3(CH_2)_7 \cdot CH_2 \cdot CH(HSO_4) \cdot (CH_2)_7 \cdot COO]_3$ ,  
and  $i$ -tri-olein-hydrogen sulphate,

$C_3H_5[CH_3(CH_2)_7 \cdot CH(HSO_4) \cdot CH_2(CH_2)_7 \cdot COO]_3$ .  
These compounds are unstable and are easily saponified, yielding sulphuric acid and hydroxy-acids. The  $\theta$ -hydrogen-sulphate yields  $\theta$ -hydroxystearic acid,  $CH_3(CH_2)_7 \cdot CH(OH) \cdot (CH_2)_7 \cdot COOH$ , which undergoes immediate transformation into sterolactone, and the  $i$ -hydrogen-sulphate yields  $i$ -hydroxystearic acid.

$CH_3(CH_2)_7 \cdot CH(OH) \cdot (CH_2)_7 \cdot COOH$ , which is stable. According to this view Turkey-red oil derived from olive oil, when neutralised with sodium carbonate, would contain the sodium salts of  $\theta$ - and  $i$ -hydroxystearic acids, unchanged saturated and unsaturated glycerides, and the sodium salts of oleic and palmitic acids, together with the sulpho-compounds formed. According to Geitel and Jaillard (this J., 1888, 218, and *loc. cit.*) the chief saturated product is a neutral sulpho-compound:



but the corresponding  $i$ -compound may also be formed. These products are more unstable even than the hydrogen-sulphates mentioned above and like them yield the corresponding  $\theta$ - and  $i$ -hydroxystearic acids.

Some preliminary experiments on the sulphonation of Chinese wood oil (tung oil), the preparation of pure linolenic acid, and the separation of the sulpho-compounds produced on sulphonating olive oil are described.

*Fats and oils; Tentative standard methods for sampling and analysis of commercial—other than those of the coconut, butter, and linsed groups.* J. Amer. Leather Chem. Assoc., 1918, 13, 14—28.

The methods described have been worked out by a committee of the American Chemical Society, and are put forward as tentative standards for industrial chemists pending their adoption by the Society. Careful directions are given for sampling under all circumstances. No sample should be accepted for analysis which does not amount to 3 lb. at least. Moisture and volatile matter are determined by drying to constant weight in a vacuum oven specially designed to give uniform temperature throughout. The pressure in the oven must be less than 100 mm. of mercury, and the temperature 15°—20° C. above the boiling point of water at that temperature, i.e., if the pressure in the oven is 70 mm., the temperature



must lie between 60° and 65° C. Distillation methods were carefully considered before the above was adopted. A more rapid method is suggested for routine work. Insoluble impurities are determined by treating the dried material with kerosene, using petroleum ether for a final washing of the residue. By this means all metallic soaps are dissolved and are determined as soluble mineral matter on burning off the kerosene from the filtrate. For the titer test, the fat is saponified by a 25% solution of caustic potash in glycerin. In the determination of unsaponifiable matter, petroleum ether has been adopted for extracting, after careful consideration of the suitability of ethyl ether. The extraction is carried out in a stoppered measuring cylinder, and the ether layer siphoned off. A blank determination is made with pure dry stearin. Methods involving the evaporation of the fat with the alkaline solution and subsequent extraction of the dry residue were found unsatisfactory.—F. C. T.

*Use of chaulmoogra oil as a specific for leprosy.*  
Brill and Williams. See XX.

#### PATENTS.

*Catalyser and process of making same.* F. W. Weber, Perth Amboy, N.J., Assignor to The Reessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,252,973, Jan. 8, 1918. Date of appl., May 3, 1915.

A LIQUID catalyser is obtained by preparing a fine paste of ox-liver, mixing with it infusorial earth, adding almond meal to the mixture, and extracting with a solvent consisting of approximately 70% water and 30% glycerin containing a preservative, e.g., sodium benzoate.—A. DE W.

*Lanolin; Process of obtaining wax-like substances from*—. I. Lifschütz, Hamburg, Germany. U.S. Pat. 1,252,591, Jan. 8, 1918. Date of appl., Nov. 3, 1914.

SEE Ger. Pats. 286,244 and 286,245; this J., 1915, 1153.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Resins; Exudation*—. IX. M. Bamberger and H. von Klimburg. Monatsh. Chem., 1917, 38, 457.

THE resin obtained from the stone pine (*Pinus cembra*) possesses an odour recalling vanillin, becomes reddened on exposure to light, and has m. pt. about 70° C., acid value 127, and iodine value 112, but the figure for the last is as low as 78.4 if the resin is previously purified by dissolving in alcohol and then pouring into water acidified with hydrochloric acid; the methoxyl content is lower than in other natural resins, amounting only to 13 parts per 1000. Boiling water extracts from the resin caffeic acid and also small quantities of ferulic acid and vanillin, whilst the residual molten resin on fusion with potassium hydroxide yields *p*-hydroxybenzoic acid, catechol, protocatechuic acid, acetic acid, and a trace of butyric acid. As with the resins investigated earlier, stone pine resin, after extraction with water, can be separated into an  $\alpha$ -resin soluble and a  $\beta$ -resin insoluble in ether, the  $\alpha$ -resin, which has a much lower methoxyl content than the  $\beta$ -resin, predominating. Attempts to produce a resinol analogous to pinoresinol or lariciresinol (Bamberger and Landsiedl, this J., 1895, 45) were unsuccessful. Lariciresinol when heated with zinc dust in a stream of hydrogen yields a distillate containing toluene, xylene, naphthalene, methyl-naphthalene, a trace of guaiacol, and probably

also cumene. Lariciresinol also gives a resinous ozonide but the only decomposition product identified after shaking with water was hydrogen peroxide. Pinoresinol is already known to contain two hydroxyl groups; by heating with alcohol and a little sulphuric acid it can be converted into an amorphous anhydro-compound,  $C_{15}H_{14}O_3$ , which resists the action of methyl sulphate in alkaline solution but on treatment with acetyl chloride yields diacetylpinoresinol.—D. F. T.

#### PATENTS.

*Phenolic condensation product; Method of producing a*—. J. P. A. McCoy, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,253,261, Jan. 15, 1918. Date of appl., Mar. 20, 1913. Renewed June 28, 1916.

A SHAPED condensation product is formed by reacting upon a phenolic substance with a substance containing an active replaceable methylene group, e.g., formaldehyde, in the presence of carbon dioxide, arresting the reaction when the product has become infusible and insoluble but is yet capable of softening under the influence of heat, shaping the product and applying heat to it.

—A. DE W.

*Phenolic condensation product.* J. P. A. McCoy, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,253,262, Jan. 15, 1918. Date of appl., Dec. 4, 1915.

A SHAPED resinous condensation product is obtained by reacting upon a phenolic substance with a material containing an active replaceable methylene group, i.e., hexamethylenetetramine, dissolving the resulting product in a solvent containing hexamethylenetetramine, passing carbon dioxide through the solution, separating the resulting resinous material from the solvent, and finally moulding it with heat.—A. DE W.

*Phenolic condensation product; Process of preparing a*—. J. P. A. McCoy, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,253,404, Jan. 15, 1918. Date of appl., Feb. 5, 1914. Renewed Nov. 30, 1917.

RESINOUS products are prepared by treating a phenolic substance with a condensing agent mixed with a reactive reduction product of such condensing agent, e.g., a mixture of carbon dioxide and formaldehyde from partially reduced carbon dioxide.—A. DE W.

*Turpentine oils; Process of refining and deodorising raw*—. H. Dedichen and O. Halse, Assignors to Union Co., Christiania. U.S. Pat. 1,253,793, Jan. 15, 1918. Date of appl., Jan. 31, 1917.

RAW turpentine oil is first subjected to chemically active rays in the presence of a gaseous oxidising agent, and is then treated with iron chloride, whereby a refined turpentine oil is obtained consisting of "a pure mixture of  $\alpha$  and  $\beta$  pinene and comprising dextro-rotary  $\beta$  pinene."—A. DE W.

*Paint compositions.* W. E. Wright, Cleveland, Ohio, U.S.A. Eng. Pat. 109,255, July 26, 1917. (Appl. No. 10,775 of 1917.) Under Int. Conv., Aug. 26, 1916.

SEE U.S. Pat. 1,226,934 of 1917; this J., 1917, 894.

*Phenols and formaldehyde; Process for obtaining hard products of condensation of*—. K. Tarassoff, Moscow, and P. Shestakoff, Petrograd, Russia. U.S. Pat. 1,252,507, Jan. 8, 1918. Date of appl., Feb. 8, 1916.

SEE Eng. Pat. 104,887 of 1916; this J., 1917, 558.

## XIV.—INDIA-RUBBER ; GUTTA-PERCHA.

*Reclaimed rubbers ; Method of valuation of —.* J. B. Périgrin. Caout. et Gutta-percha, 1918, 15, 9436.

THE approximate rubber content of samples of reclaimed rubber was ascertained by difference after determining the ash, sulphur, and loss by successive extractions with acetone, alcoholic sodium hydroxide, and pyridine respectively, and the cost of the actual rubber present was calculated from the selling price of the material. In a number of samples examined, this cost worked out at figures varying from about 1s. 6d. to about 3s. 4d. per lb.—E. W. L.

## XV.—LEATHER ; BONE ; HORN ; GLUE.

*[Tanning] extract analysis ; Comparative —.* J. M. Seltzer. J. Amer. Leather Chem. Assoc., 1918, 13, 9—11.

TANNING extract was sampled by the manufacturer and the tanner, and each sample was analysed by the tannery chemist and the manufacturer's chemist. 100 such analyses are tabulated for comparison. The averages for the whole series show a greatest difference of only 0.07% tannin.—F. C. T.

*Leather ; Determination of ash in —.* O. Riethof and C. T. Gayley. J. Amer. Leather Chem. Assoc., 1918, 13, 7—9.

THE authors suggest that the insoluble ash should be determined by incinerating the dried, extracted leather, and the soluble ash preferably by evaporating the aqueous leather extract and igniting the residue. The sum of these determinations gives the total ash. By adopting such a uniform procedure the present variations and errors can be largely avoided.—F. C. T.

## XVI.—SOILS ; FERTILISERS.

*Soil reaction ; Studies in — as indicated by the hydrogen electrode.* J. K. Plummer. J. Agric. Res., 1918, 12, 19—31.

MEASUREMENTS of the reactions of soils in suspension in water, and of the film water from soils, obtained by the oil pressure method in a Morgan apparatus (this J., 1917, 972), have been made by means of a hydrogen electrode. It was found that the film water from a soil has the same reaction as the free water, differing only in intensity. Ammonium sulphate, applied to soils, materially increases the hydrogen ion concentration, and potassium sulphate does so to a less extent, whilst acid phosphates do not appear to affect the hydrogen ion concentration of field soils. Sodium nitrate slightly reduces the acidity of soils, whilst lime materially increases the hydroxyl ion concentration. The acidity developed from ammonium sulphate is more intense in the film water than in the free water of soils.—W. G.

*Manures ; Decomposition of green and stable — in soil.* R. S. Potter and R. S. Snyder. J. Agric. Res., 1917, 11, 677—698.

SOIL organic matter decomposes more rapidly in a soil to which calcium carbonate has been applied, than in untreated soil. Similarly the decomposition of the total organic matter of a soil, after the application of stable manure or green manure in the form of oats or clover, is more rapid under the influence of calcium carbonate than without it. Stable manure seems to increase the rate of decom-

position of green manure to a slight extent. Both of these manures tend to conserve the lime in the soil.—W. G.

*Fertilisers ; Action of various —, especially manganese sulphate, on the growth of oats.* L. Hiltner and G. Korff. Biedermann's Zentr., 1918, 47, 15—19.

OATS were grown in various types of soils in 8 litre pots and manured with 25 grms. of manganese sulphate, copper sulphate, or iron sulphate, or with 50 grms. of powdered sulphur. Each pot of soil also received 25 grms. of guano. In every case, an increase in the yield of oats was noted in the manured as compared with control unmanured soils, especially in those soils treated with manganese sulphate. The effect of the manganese was most marked when applied to soils containing much humus ; in peaty soils, the yield of oat-grains was 70 times as great in the presence as in the absence of manganese. (See also J. Chem. Soc., Mar., 1918.)—H. W. B.

*"Zeolitic" silicic acids ; New method for the estimation of — in soils.* G. Guedroit. Rev. Exp. Agron. Petrograd, 1916, 17, 400—407. Bull. Agric. Intell., 1917, 8, 1190—1191.

FOR the estimation of the "zeolitic" silicic acids liberated during the treatment of soils with hot, concentrated hydrochloric acid, the following procedure is recommended. Five grams of soil is subjected to the acid treatment and then heated in a platinum dish at a temperature not exceeding 62.5° C. until the organic matter is all destroyed. The residue is heated on a water-bath for half an hour with 100 c.c. of 5% potassium hydroxide solution and filtered, the insoluble portion being washed ten times with a 1% solution of potassium hydroxide. The filtrate is acidified with hydrochloric acid and evaporated to dryness, the residue being heated at 125°—150° C. for 1—1½ hours. The silicic acid may then be separated and estimated in the usual manner.—W. G.

*Nitrogen-fixing bacteria ; Physiology and biology of —.* V. L. Ormiansky. Arch. des Sciences Biol. de Petrograd, 1916, 19, 209—227. Bull. Agric. Intell., 1917, 8, 1190.

A CRITICAL discussion of important recent work on *Clostridium* (see this J., 1917, 40) and an account of new experiments on the biology and physiology of that organism. Growth is more abundant and the fermentation of sugar more energetic at about 30° C. but nitrogen-fixation is less than at the ordinary temperature. *Clostridium* may be heated to 75° C. without destruction, and thus other non-sporing species may be eliminated by prolonged pasteurisation at that temperature. Spores of *Clostridium* may be preserved in the dry state in air for 20 years without losing their power of nitrogen-fixation. With cultures in the presence of carbohydrates, the fermentation of ten carbohydrates (dextrose, levulose, sucrose, galactose, maltose, raffinose, dextrin, inulin, glycerol, mannitol) was observed, the most vigorous growth occurring with dextrose, raffinose, inulin, and mannitol. Sugar concentration has a great influence on the nitrogen-fixation by *Clostridium* ; the greater the concentration the less the nitrogen-fixation per gram of sugar. The manner in which the nitrogenous food is supplied also has an effect on the fixation, too large an increase in nitrogen-content of the medium causing a decrease of fixation and finally stopping it. With a *Clostridium* from Volhynie, however, nitrogen-fixation still took place when the ratio of nitrogen to sugar in the medium was 16 : 1000. The author agrees with Winogradsky that *Clostridium* is a typical butyric ferment.—W. G.



*Phosphoric acid; Determination of* — particularly in superphosphate. G. Vortmann. Z. anal. Chem., 1917, 56, 465—487.

IN the precipitation of phosphoric acid as ammonium magnesium phosphate, the presence of ammonium sulphide and oxalate does not interfere with the results obtained; ammonium molybdate is also without influence. Large quantities of aluminium salts interfere with the precipitation unless an excess of "magnesia mixture" is used. If the phosphoric acid is first separated by precipitation with molybdic acid, this precipitation may be made in either nitric, hydrochloric, or sulphuric acid solution. The addition of pyridine to molybdic acid solution increases the sensitiveness of the latter; 0.01 mgrm.  $P_2O_5$  in 10 c.c. of solution may be detected, whilst the limit for the usual molybdic acid reagent is about 0.1 mgrm.  $P_2O_5$  in 10 c.c. The mercury oxide method for the determination of phosphoric acid in superphosphate is accurate; in this method, the phosphoric acid is precipitated, by means of yellow mercury oxide, as a basic mercury phosphate, the latter is decomposed by sodium sulphide, and the phosphoric acid then determined by the magnesia method. Precipitation of the phosphoric acid as calcium triphosphate is unreliable. (See also J. Chem. Soc., Mar., 1918.)—W. P. S.

*Borax as destructive of vegetation.* W. H. Roberts, A. Smetham, and J. A. Voelcker. Analyst, 1918, 43, 58—59.

IN a law case, in which it was alleged that damage had been done to a sewage farm through the turning on to it of waste-liquor from borax works, the evidence of the authors for the plaintiffs showed that the presence of borax in the soil was the one and only cause of the destruction of the crops. Samples of the damaged soil, dried at 100° C., contained 0.04, 0.032, 0.036, and 0.163% of borax, whilst the soil from other parts of the farm on which an altogether different effluent flowed was free from borax. Experiments carried out by J. A. Voelcker at the Woburn Experimental Station (J. Roy. Agric. Soc. Eng., 1915, 76) showed that the presence of 0.043% of borax in a soil was destructive to wheat and barley, and other experiments indicated the possibility of borates accumulating in the soil.—W. P. S.

## XVII.—SUGARS; STARCHES; GUMS.

*Moulds; A case of raw sugar deterioration caused by* —, W. J. T. Amons. Archief Suikerind. Ned.-Indië, 1917, 25, 1225—1231.

FROM a sample of deteriorated raw sugar taken from a store, various moulds were isolated, amongst which were *Aspergillus niger*, *A. fumigatus*, *A. flavus*, *A. clavatus*, *Penicillium glaucum*, *P. purpurogenum* (Stoll), a few yeasts and bacteria being also present. It was found possible to induce the deterioration of raw sugar under suitable hygroscopic conditions by means of *Penicillium glaucum* alone in the absence of bacteria. The author believes that probably no one micro-organism is solely responsible for sugar deterioration, and that at one time moulds, at another bacteria, may be the cause, depending upon the conditions prevailing at the time of infection.—J. P. O.

[*Sugar*] *factories using the defecation process; Filtration of muddy juice in Java* —, A. Schweizer and G. Loos. Archief Suikerind. Ned.-Indië, 1917, 25, 1561—1563.

CERTAIN factories using the ordinary defecation process had Kelly presses installed, and it was found, contrary to what had been experienced in

defecation-sulphitation houses, that the capacity of the filters was not as great as expected, the difficulty being principally in sweetening off, but also in dropping the mud from the frames and in cleaning the cloths. Efforts were made to improve matters by the addition of materials such as fine carbon to the mud, but unavailing. Suspecting that the absence of crystalline calcium sulphite might account for the difficult filtration, liming and sulphitation of the muddy juices was carried out, using 10—14 litres of 15 B. lime cream to 1000 litres of juice and sulphiting to neutrality to phenolphthalein. This treatment was entirely satisfactory, results analogous to those obtained in defecation-sulphitation factories being thus obtained.—J. P. O.

*Sugar refining; Use of vegetable decolorising carbon ("Norit"), as compared with animal charcoal (boneblack) in* —, J. Sauer. Intern. Sugar J., 1918, 20, 24—29.

REPLYING to criticisms upon the use in sugar refining of decolorising carbon, particularly the vegetable material known as "Norit," it is claimed that volume for volume 3 parts is capable of exerting the same decolorising effect as 100 parts of animal charcoal. Not only colouring matter, but gums, pectins, and other non-sugar bodies, as well as mineral matter, are adsorbed from solution by the carbon, and can be found in it after use. In the factory decolorisation is carried out after clarification with lime and filtration, and preferably in a solution that is distinctly acid, using sulphurous or phosphoric acid, while boiling may be carried on for 15 minutes or even longer without inversion occurring. It is claimed that in the revivification of decolorising carbon according to the "Norit" patents (this J., 1913, 85; 1914, 853), the adsorptive power decreases to a less extent after each re-burning than in the case of animal charcoal, so that it is more economical in use. Decolorising carbon is now being employed in cane and beet factories and refineries in different parts, and also in other establishments, those, for example, making jam, chocolate, biscuits, or lemonade, for which a white sugar is required.—J. P. O.

*Cane sugar manufacture [in Hawaii]; Separation of non-fluid impurities from juices, syrups, and molasses in* —, E. Kopke. Intern. Sugar J., 1918, 20, 29—32.

MILL juice carries with it as mechanical impurities principally small particles of bagasse and earth, and for their removal screens of perforated brass with mechanically operated slat scrapers are now exclusively used in Hawaii, though a revolving screen arranged above the intermediate bagasse conveyor is to be installed. Very fine particles of bagasse passing through the ordinary perforations are intercepted by wire cloth of 100—200 mesh. Limed and heated juices are allowed to subside in tanks of either intermittent or continuous type. Filters, the medium in which may be cloth, sand, bagasse, or "Excelsior," are also used, but less every year, notwithstanding the excellent results, as they are costly in respect of labour, quantity of medium, and loss of sugar. Scum and mud collected from the settling tanks of limed and heated juices is separated from any remaining juice in filter-presses, and besides those of the plate-and-frame type, the Kelly and Sweetland presses are now being used in Hawaii. Overhead supply tanks delivering under pressure remain the best arrangement for passing the scums and mud to the presses; a centrifugal pump is in most cases quite as satisfactory; but reciprocating pumps giving a pulsating delivery are objectionable, and should only be used in conjunction with a supply tank. Syrup and molasses



contain principally gummy substances and calcium salts as mechanical impurities, for the removal of which settling is out of the question, while filtration when the gums are high is also impossible. Very encouraging results have, however, been obtained during the past three campaigns with a clarifying centrifugal (this J., 1917, 398), which machine is capable of operating continuously. Analysis of some of the mud taken from this apparatus after clarifying a molasses gave the following percentage results: organic matter, 51.85; silica, 4.85; iron and alumina, 2.25; lime, 21.49; magnesia, 3.94; phosphoric acid, 4.17; sulphuric acid, 9.88; carbon dioxide, 0.62. Combining these constituents, the residue is shown to contain: calcium phosphate, 9; calcium sulphate, 7; and compounds of lime and organic acids, about 22 per cent. It would, therefore, resemble evaporator scale, except in the large proportion of organic matter.

—J. P. O.

*Sucrose content; Some methods for the determination of the — of [cane] molasses.* T. van der Linden. *Archief Suikerind. Ned.-Indië*, 1917, 25, 1240—1272.

A COMPARATIVE examination of the several methods that have been proposed to obviate the error in the double polarisation method due to the presence of optically-active impurities, the rotations of which are also affected by the acid used for hydrolysis, has led to the following conclusions. Uniformly concordant results were obtained with the processes of Tervoooren (*Archief Suikerind. Ned.-Indië*, 1904, 321) and Steuerwald (this J., 1913, 1025), in the latter of which hydrolysis is not carried on at 68°—70° C., but at the prevailing temperature; with Java molasses, however, the liquid obtained in both methods, after clarifying with basic lead acetate, was so dark as to make the direct reading hardly possible, even in a 100 mm. tube. On the other hand, Pellet's sulphurous acid method (*Bull. Assoc. Chim. Sucr.*, 8, 623; this J., 1897, 474) gave an excellent clarification, while the results were equal to or only slightly lower than those found by the methods of Tervoooren and Steuerwald. Stanek's method (this J., 1914, 705) proved unsatisfactory with Java molasses, difficulty being experienced in clarifying with bromine as prescribed, whilst the results were distinctly lower than those found by the methods of Tervoooren or Steuerwald. Saillard's method (this J., 1913, 836) was found too tedious for routine work, and was not applied, nor was the invertase method described by Ogilvie (this J., 1911, 62), owing to the difficulty of obtaining and preserving yeast in the tropics. Deerr's method (this J., 1915, 503), though theoretically exact and of value in special investigations, is too lengthy and involved for routine factory work. It gave the lowest results of all. Pellet and Lemeland's process of polarising after the destruction of the reducing sugars by alkali (*Bull. Assoc. Chim. Sucr.*, 1915, 33, 89), as well as its modification by Cross and Taggart, is not considered trustworthy, there being a considerable difference between the polarisations before and after acidifying with acetic acid, while the values it yields, though regular in themselves, are not in accord with those given by the double polarisation method of Steuerwald. In treating reducing sugars with alkali under the conditions of the method, the *levo*-rotation is not totally removed, and a slight oxidation of sucrose occurs, especially when hydrogen peroxide is used. In general, the several methods examined are more complicated than the "cold inversion" process of Steuerwald now in use in Java, and none can be recommended in preference to it. A combination of Pellet's sul-

phurous acid method and Steuerwald's method, however, might advantageously be employed, as in this way the difficulty in obtaining light-tinted liquids experienced in working with certain molasses, particularly those from carbonatation factories, would be overcome.—J. P. O.

*Molasses; Dry substance and sucrose determinations in cane —.* L. G. L. Steuerwald. *Archief. Suikerind. Ned.-Indië*, 1917, 25, 1417—1425.

SAMPLES of well-mixed and previously analysed molasses were sent from the experiment station to the sugar factories in Java with the request that the dry substance should be determined in them by the Josse method of drying on a coil of bibulous paper and the sucrose by the Tervoooren double polarisation process. As a whole the results returned for the dry substance content were very unsatisfactory, varying between wide limits, the conclusion being that the Josse method is unsuitable for factory routine work. On the other hand, the figures for the sucrose content concurred well with those found at the experiment station.

—J. P. O.

*Formation of lactose from starch by the "loosened" ferments of sucrose serum.* Röhmman. *See XVIII.*

## XVIII.—FERMENTATION INDUSTRIES.

[Yeast;] *Maltase-activity [of —] in relation to fermentation and storage. The maltase activity of different yeasts. Influence of oxygen on the maltase-activity of yeast in beer.* F. Schönfeld and H. Krumhaar. *Woch. Brau.*, 1917, 34, 60—62, 157—159, 165—166, 189—190.

I. YEAST maltase, like zymase, is an intracellular enzyme and can only be extracted after the yeast has been dried or the cells ruptured by mechanical means. It does not appear to escape from cells which die under normal conditions, *e.g.*, during the storage of beer. In the fermentation of all-malt worts the hexoses are eliminated at a relatively early stage (see this J., 1916, 1230), and as the destruction of maltose requires the presence of maltase as well as zymase, a deficiency of either of these enzymes may cause the later stages of fermentation to become sluggish. II. To compare the maltase-activity of different yeasts the authors introduced equal quantities (5 or 2%) into 5% or 2% maltose solutions with 8% of toluene, and maintained the liquids at 6°—7° C. The amounts of dextrose formed after successive intervals of 1 or 2 days were determined by conversion into glucosazone. The amount of toluene used completely suppressed the action of the zymase of the yeast, and the rate of formation of dextrose was therefore regarded as a measure of the maltase-activity of the yeasts. The results for a number of brewery yeasts indicated a general parallelism between maltase-activity and attenuating power, and the latter is considered to be dependent on the former. III. From further experiments it is concluded that the maltase-activity of yeast is stimulated by aeration, and may become permanently weakened by prolonged deprivation of air. Since the after-fermentation of all-malt beers proceeds mainly at the expense of the maltose remaining after the primary fermentation, it is largely controlled by the maltase-activity of the yeast present during storage, and will therefore be stimulated by the introduction of air into the beer, *e.g.*, by means of rousing or the use of chips, which contain a considerable amount of air in their pores.—J. H. L.



*Feast ; Reproduction of* —. T. Bokorny. Woch. Brau., 1917, 34, 269—271.

IN the fermentation of sugar solutions the weight of yeast produced, expressed as a percentage of the weight of sugar decomposed, varies greatly according to the conditions ; different investigators have found values as low as 0.8% and as high as 14%. The author obtained a still higher result, 16%, in the fermentation of sucrose solution containing only urea as source of nitrogen (cp. this J., 1917, 1284), the liquid being exposed to the air in a very shallow layer, only 0.1 inch thick. An important factor which influences the relative amounts of sugar fermented and assimilated is the rate of fermentation. When the amount of yeast present is large, so that fermentation proceeds rapidly and is soon complete, little or even none of the sugar may be assimilated, i.e., no fresh yeast may be formed. If the sugar is added to a yeast suspension in small quantities at intervals, a larger proportion of it will be assimilated than if it is added all at once at the outset. The difficulties which have been experienced in the production of beers of very low gravity, owing to insufficient yeast crops and degeneration, may possibly be avoided by employing a smaller amount of pitching yeast, so that in consequence of slower fermentation a larger proportion of the sugar may be assimilated by the yeast.—J. H. L.

*Worts ; Proximate constituents of sweet and hopped* — at successive stages of the mashing process. A. R. Ling. J. Inst. Brew., 1918, 24, 15—21.

IT has been held by practical brewers that if the extraction of the contents of the mash tun is carried to completion, the last runnings are liable to contain undesirable substances which may cause trouble at a later stage, and chemists have attributed this to the extraction of excessive amounts of nitrogenous matters. Some analytical data are now presented which do not confirm the latter view. In a brewery where the mash-tun wort is boiled in three "lengths," three samples of the wort drawn off from the tun were taken at different stages and analysed. Their respective specific gravities were 1095.8, 1070.2, and 1008.5, and their contents of protein ( $N \times 6.25$ ) expressed as percentages of the extract present were 4.89, 5.45, and 8.97% respectively. It appears, therefore, that the matter extracted by the later spargings contains a considerably higher proportion of nitrogen than the extract in the first wort, but in view of the small amount of total extract in the last runnings the extra amount of nitrogenous matter introduced into the wort is almost negligible. The extract in the third sample was found to contain a rather larger proportion of "apparent dextrin," and a smaller proportion of "apparent maltose" than in the two other cases, though there was little difference between the specific rotatory powers of the three extracts. Analysis of the three successive copper worts, after boiling, showed substantially similar relations to those observed with the mash-tun worts.—J. H. L.

*Beers ; Use of malt flour in the production of English* —. F. Hyde. Amer. Brewers' Review, 1917, 31, 359.

IN the preparation of English beers, using a highly attenuating yeast, the author has found certain advantages in the use of a dressing of malt dust made into small pellets and added to the wort during the primary fermentation when the yeast head is forming. About 1 lb. is used per 20 barrels of wort ; a small amount of sugar krausen is added at racking, the beer is fined and sent out 2 or 3 days later and usually consumed within 10 days. The practical advantages of the dressing

are better and more solid yeast crops, more complete attenuation, lessened liability of the beer to chill-haze, and enhanced head-retaining power. On the other hand there is a certain risk of infection since the malt dust contains bacteria, but this has been over-estimated. The alcohol-content of the beer is increased as a result of the dressing.

—J. H. L.

*Alcoholic fermentation ; Intermediary reactions in* —. H. Euler, H. Ohlsen, and D. Johansson. Biochem. Zeits., 1917, 84, 402—406.

A SWEDISH beer yeast dried at 40° C. and extracted with water gives an extract which contains a phosphatase capable of forming directly a phosphate ester from *lævulose* but not from dextrose. The latter sugar can, however, be esterified if it is first treated with fresh yeast. A yeast weakened by standing for six weeks in a solution containing sodium chloride and dextrose but no nitrogen, can also cause ester formation from phosphate and *levulose* but not from dextrose. The results confirm Harden's view that the hexose phosphate is a derivative of *lævulose* or a closely allied sugar, and that other sugars must be converted into this before esterification with the phosphate takes place.—S. B. S.

*Lactose ; The formation of* — from starch by the "loosened" ferments of sucrose serum. F. Röhmman. Biochem. Zeits., 1917, 84, 399—401.

THE parenteral injection of sucrose into rabbits gives rise to special ferments in the serum ("*her-vorgelockte Fermente*"). The extract of the liver of a rabbit thus treated acts on soluble starch, causing degradation, and producing, amongst other products, lactose.—S. B. S.

*Urease ; Preparation of* — from bacteria. M. Jacoby. Biochem. Zeits., 1917, 84, 354—357. (See also this J., 1917, 663, 901, 1106.)

THE bacteria are removed from their cultures on agar and dried on porous porcelain. The preparation thus obtained is permanent and acts in the presence of toluene.—S. B. S.

*Enzymes ; Formation of* —. V. M. Jacoby. Biochem. Zeits., 1917, 84, 358.

LEUCINE inhibits rather than accelerates the action of urease (see preceding abstract) and this indicates, taken in conjunction with the author's previous work, that the amino-acid is necessary for the formation of the enzyme, but not for its action.—S. B. S.

*Physiology and biology of nitrogen-fixing bacteria.* Omeliansky. See XVI.

*Yeast as foodstuff.* Petit. See XIXa.

"Nitrogen syrup." Jalowetz. See XIXa.

*Determination of lactic acid in presence of other organic acids.* Szeberényi. See XX.

#### PATENTS.

*Method of rendering alcohol more readily ignitable when used as fuel in internal combustion engines.* Eng. Pat. 112,741. See IIa.

*Process of treating by-product liquors from wood pulp sulphite plants.* U.S. Pats. 1,253,853 and 1,253,854. See V.

## XIXA.—FOODS.

*Flour; Examination of "85%"* — H. Delehay. *Ann. Falsif.*, 1917, 10, 554—556.

To determine whether a flour consists of 85% of the wheat grain the author suggests a method in which flour is passed through two sieves, Nos. 30 and 60 (Continental gauge); the portions retained by these two sieves are mixed, weighed, and the crude fibre content determined. Flours which conform with the 85% standard yield about 10% of coarse substance (on the two sieves), with a crude fibre content of 4%. In other flours the amount of coarse material falls as low as 4.5%, with a crude fibre content of 1%. The results obtained should be confirmed by a microscopical and chemical examination of the flour.—W. P. S.

*Flour; Ratio of soluble nitrogen to total nitrogen in* — E. Rousseaux and M. Sirot. *Ann. Falsif.*, 1917, 10, 556—560.

THE average ratio of soluble to total nitrogen in various flours is as follows:—Wheat, 6; maize, 5.5; rye, 4.4; bean, 8.2; barley, 8.2; rice, 23.6. When this ratio in the case of wheat flour is either above or below 6, the bread-making quality of the flour is unsatisfactory.—W. P. S.

*Bread and flour; Poisonous* — *Detection and determination of sapotoxins.* L. Stœcklin. *Ann. Falsif.*, 1917, 10, 561—572.

A SAMPLE of wheat examined by the author contained 19% of foreign seeds, including 10% of corn-cockle (*Agrostemma githago*). The toxic properties of the sapotoxins contained in the latter would explain the numerous cases of poisoning observed recently in France; it is possible that such samples of wheat are by no means rare. The hæmolytic action of sapotoxins is relied on for their detection, their quantity being determined by comparison with standards.—W. P. S.

*"Sharps" and bran; Composition of* — and *the effect thereon of the Food Controller's Orders.* H. V. Cox. *Analyst*, 1918, 43, 53—56.

THE composition of "sharps" (or middlings) and bran has been materially affected by recent orders of the Food Controller regarding the milling of wheat. To get the additional percentage of flour, a second grinding and sifting is necessary, and the "sharps" and bran suffer by loss of starchy material. The average yield of milling products of wheat at the present time is:—Government grade flour, 80; "sharps," 14; bran, 6%. The following table shows the alteration in the starch, pentosan, and crude fibre content of "sharps" and bran:—

	"Sharps."				Bran.	
	Pre-war		Present		Pre-war average.	Present average.
	Average.	Maximum.	Average.	Maximum.		
Crude fibre.....	% 6	% 7.5	% 8	% 10	% 9	% 13
Pentosan.....	12	18	17	20	20	24
Starch.....	32	Min. 25	25	Min. 18	15	14

Oat husk contains crude fibre 33, pentosans 40.4, and starch 3.27%.—W. P. S.

*Wheat bran and its adulterants.* E. Collin. *Ann. Falsif.*, 1917, 10, 539—551.

A DESCRIPTION is given of the microscopical appearance of wheat bran and of substances used to

adulterate it; among the latter are husks of other cereals, rice husk, earlnut meal and husk, vegetable ivory, sawdust, castor oil seeds, maize husk, etc. The characteristic features of these are illustrated.—W. P. S.

*Diphenylamine-sulphuric acid; Use of* — *for colorimetric determinations [e.g., of nitrates in milk].* J. Tillmans. *Z. anal. Chem.*, 1917, 56, 509—511.

IN the method described previously by the author (this J., 1911, 44) for the determination of nitrates in milk, any variation in the intensity of the colorations obtained may be overcome by treating the test solution and the standards in exactly the same way.—W. P. S.

*Margarine; Manufacture of* — *in the United Kingdom.* W. G. Watson. *J. Roy. Soc. Arts*, 1918, 66, 212—220.

THE average weekly quantities of margarine manufactured in Great Britain increased from 1611 tons in 1913 to 3564 tons in 1917, whilst the imports of margarine increased from 1460 tons in 1913 to 2647 tons in 1916, and the weekly imports of butter decreased from 3980 tons in 1913 to 2092 tons in 1916. The churning process of 5 to 10 years ago has been replaced by a continuous process in which a motor-driven emulsifier, requiring little attention, is used. Arrangements are made to receive the milk in the factory within 6 to 8 hours after milking. The milk is separated (the cream being used for butter-making) and the skim milk is pasteurised, cooled nearly to freezing point, and stored in tanks under cold-storage conditions. The ripening process, by means of pure cultures of lactic organisms, is carried out in propagators continuously, the fresh milk being run in at the top as the ripened milk is drawn off at the bottom. Live steam has been found the most effective means of sterilising the plant. At Southall the air in the works is also purified, and brought to a constant temperature and humidity, by being drawn through a chamber, provided with means for heating or cooling, where it comes in contact with a fine rain produced by hundreds of water-jets. Thence it is distributed through the work-rooms by means of air-ducts. By aid of these precautions the keeping qualities of the margarine have been improved. (See also this J., 1918, 54 R.)—C. A. M.

*Yeast as foodstuff.* P. Petit. *Brasserie et Malterie*, 1917, 7, 257—260.

YEAST extracts intended for human consumption should possess a pleasant flavour, resembling that of meat extract, without sourness or bitterness;

they should keep well in closed bottles and if possible be dark in colour. The following method of preparation is described. Carefully sifted and well washed yeast is treated first with a 0.1—0.15% solution of sodium or ammonium carbonate and then with a similar solution of half this concentration. Thus freed from bitter resins, the



pressed yeast is liquefied by admixture with 10% of sodium chloride and a little water, the mixture being maintained meanwhile at 40°–45° C., in a shallow pan. After liquefaction flavouring vegetables such as celery, onions, or leeks may be added, and the mixture is heated at 70°–75° C., exposed to the air in a shallow pan, until the required "meaty" flavour and dark colour have developed. The temperature may then be raised to the boiling point for a moment. After the insoluble matter has been removed by filtration or subsidence, the clear extract is concentrated in steam-jacketed pans and filled into sterile bottles, and if necessary pasteurised. Products of high nutritive value may be obtained by mixing concentrated, unhopped malt wort with extracts made from yeast which has been liquefied with sugar instead of salt. A less known type of product is flaked yeast extract. To prepare this the purified yeast is liquefied with a small quantity of salt and the subsequent heating is limited to 30°–35° C. to avoid darkening the colour. The filtered extract is concentrated at a low temperature, *in vacuo*, and converted into flakes by passage between heated rollers. The dried product keeps almost indefinitely and is said to have a nutritive value three times as great as that of meat.

—J. H. L.

"Syrup; Nitrogen —." E. Jalowetz. *Braun und Malzind.*, 1916, 17, 247. *Z. ges. Brauw.*, 1917, 40, 118.

WHEN concentrated sucrose solutions are digested with washed brewers' yeast for 3–4 hours at 53°–54° C., partial inversion of the sugar takes place, and by subsequent concentration syrups containing 2% or more of protein may be obtained resembling malt extracts in appearance and flavour.—J. H. L.

*Antineuritic properties of the infusorial earth extract of the hydrolysed extract of rice polishings.* H. C. Brill. *Philippine J. Sci.*, 1917, 12a, 199–206.

ONLY a portion of the vitamin content of the hydrolysed extract of rice polishings is extracted by infusorial earth, used in the proportion of 50 grms. of earth to 500 c.c. of extract. The extract appears to lose its antineuritic properties as it ages.—W. G.

*Gossypol, the toxic substance in cottonseed.* W. A. Withers and F. E. Carruth. *J. Agric. Res.*, 1918, 12, 83–102.

FURTHER feeding experiments (compare this J., 1915, 1267) have been conducted with rats, rabbits, and pigs. Cottonseed meal is much less toxic than raw cottonseed, probably owing to oxidation of the gossypol during the cooking in the preparation of the meal. Feeding experiments with small pigs in pens showed that cottonseed meal is definitely injurious but that the ether-extracted raw seed does not appear to cause cottonseed meal poisoning. Gossypol is toxic to pigs. In addition to this toxic effect a diet of cottonseed meal and corn (maize) meal has nutritive limitations which may lead to failure of pigs to thrive in pens, this result being, however, quite distinct from cottonseed meal poisoning. This poisoning may be postponed or averted in the case of pigs by outdoor exercise, access to forage and soil, and improved diets.—W. G.

*Malt meal; Rearing of young pigs on —.* Richardson. *Biedermann's Zentr.*, 1918, 47, 43–47.

YOUNG pigs grew rapidly on a diet in which a large proportion of the milk was replaced by "cerealis," a malt meal containing 12.6% protein, 2.7% fat, 27.2% maltose, 27.6% starch, 3.8% fibre, 3.0% ash, and 9% moisture. Although

"cerealis" is not now obtainable, similar results may be expected to follow the use of auto-digested malt for this purpose.—H. W. B.

*Casein; Effect of time of digestion on the hydrolysis of — in the presence of starch.* J. S. McHargue. *J. Agric. Res.*, 1918, 12, 1–7.

THE Van Slyke method for protein analysis (this J., 1911, 1135), when applied to a mixture of casein and starch in the proportion of 1 to 5, the hydrolysis being carried on for 12–15 hours with 20% hydrochloric acid, gives results for the amino-acid groups comparable with those obtained for casein alone. Prolonging the time of hydrolysis with the casein-starch mixture causes a redistribution of the nitrogen in the histidine and cystine groups. The insoluble residue from the casein-starch hydrolysis contains nitrogen, which is in an inert form and should not be included in the humin determination.—W. G.

*Use of different alkalis in quantitative analysis.* Palet. See XXIII.

#### PATENTS.

*Milk; Drying —.* C. H. Campbell, Assignor to Borden's Condensed Milk Co., New York. U.S. Pat. 1,250,127, Dec. 18, 1917. Date of appl., Dec. 29, 1913.

MILK is concentrated, then converted into an aerated, plastic, adhesive mass which is spread on a flexible wire net and dried by passing it between a pair of hot-air boxes.—W. P. S.

*Liquids [milk]; Apparatus for treating —.* C. H. Campbell, Assignor to Borden's Condensed Milk Co., New York, N.Y. U.S. Pat. 1,253,317, Jan. 15, 1918. Date of appl., Jan. 7, 1916.

THE apparatus consists of a trough-shaped vessel containing a horizontal shaft on which agitators are mounted. Air is admitted by a series of pipes placed alternately on each side of the trough between the agitators, and reaching towards the bottom of the trough. In a form of the apparatus specially designed for the treatment of milk, the shaft is hollow and is divided lengthwise into a supply and an exhaust compartment, with an opening into each at opposite ends of the shaft. The agitators are hollow discs tapering towards their peripheries. From the supply compartment of the shaft, a pipe opens into each disc towards the apex, and opposite to it is placed an outlet pipe leading into the exhaust compartment. Scrapers are provided for the discs during rotation. A liquid at a suitable temperature may be passed through the discs.—J. H. J.

*Grain and other coarse granular materials; Process for drying —.* E. Passburg, Berlin. U.S. Pat. 1,250,496, Dec. 18, 1917. Date of appl., Nov. 9, 1916.

THE grain is stirred and heated to 60°–70° C., then transferred to a non-heated vessel, and dried therein first under slightly and then under greatly reduced pressure.—W. P. S.

*Egg preservative.* M. J. Davis, Brooklyn, N.Y., Assignor to M. Micolino, New York. U.S. Pat. 1,250,567, Dec. 18, 1917. Date of appl., June 2, 1915. (See also Eng. Pat. 105,840 of 1916; this J., 1917, 665.)

TUNG oil or a mixture of tung oil and resin is used for coating the eggs.—W. P. S.

*Meats; Duplex process for smoke-curing —.* A. C. Legg, Birmingham, Ala. U.S. Pat. 1,250,934, Dec. 18, 1917. Date of appl., Dec. 14, 1915.

MEAT is dried in an upward current of hot dry

smoke until the fat begins to shrink, and then in a down draught of cool smoke from which "the products of active combustion" are excluded.

—W. P. S.

*Fish: Process of preserving—and products thereof.* R. M. Thompson, Tacoma, Wash. U.S. Pat. 1,252,865, Jan. 8, 1918. Date of appl., Nov. 26, 1917.

Fish of the shark family is cooked in a retort and the exuded juice withdrawn; a vacuum is then produced in the retort and the fish drained. Next an acid fluid is introduced and allowed to impregnate the fish, after which a partial vacuum is again produced in order to remove some of the moisture. The meat is then separated from the bone and canned. The product may be flavoured with the flavours of other meats.—J. H. J.

*Lubrication of milk centrifugals and the like.* H. Holte, and Aktieselskapet Separator, Skien, Norway. Eng. Pat. 111,662, Nov. 9, 1917. (Appl. No. 16,427 of 1917.) Under Int. Conv., Nov. 28, 1916.

*Preparation of colouring bodies for use in food, pharmaceutical and other preparations.* Eng. Pat. 112,886. See IV.

*Method of manufacturing radioactive mineral water salts.* U.S. Pat. 1,253,830. See VII.

## XIXB.—WATER PURIFICATION ; SANITATION.

*Use of different alkalis in quantitative analysis.* Palet. See XXIII.

*Acidity determinations in water, leach liquors, mine waters, etc.* Rankin. See XXIII.

### PATENTS.

*Water: Means for rendering—radio-active.* G. E. Heyl and T. T. Baker, London. Eng. Pat. 112,865, Mar. 30, 1917. (Appl. No. 4651 of 1917.)

A SMALL quantity of a solid radio-active compound, such as radium-barium sulphate of about 1 : 1,000,000 concentration, which has been ground to a very fine powder under water, is contained within a capsule of a material soluble in water, such as gelatin and the like. When placed in water, the capsule dissolves, releasing an infinite number of excessively small radio-active particles.

—L. A. C.

*Waters: Method for treating ferruginous—.* L. S. Hughes and C. H. Wolcott, Chicago, Ill. U.S. Pat. 1,254,009, Jan. 15, 1918. Date of appl., Sep. 8, 1914.

A FERRUGINOUS water is treated with a soluble hypochlorite to convert ferrous salts into ferric salts, and then neutralisation is commenced with a compound which will form an insoluble salt with the acid content of the water, and completed with magnesite. The precipitate is recovered.—J. H. J.

*Water and other liquids or the like: Filtering of—.* D. Dunbar, Glasgow. U.S. Pat. 1,253,674, Jan. 15, 1918. Date of appl., Aug. 19, 1914.

SEE Eng. Pat. 19,142 of 1913; this J., 1914, 980.

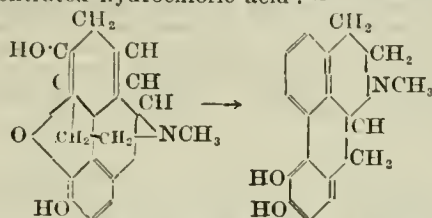
*Water: Process for the elimination of manganese and iron from—.* V. Kobelt, Berlin-Schöneberg, Germany. U.S. Pat. 1,253,840, Jan. 15, 1918. Date of appl., Jan. 21, 1915.

SEE Eng. Pat. 405 of 1915; this J., 1916, 326.

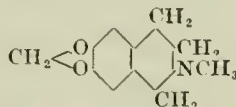
## XX.—ORGANIC PRODUCTS ; MEDICINAL SUBSTANCES ; ESSENTIAL OILS.

*Morphine alkaloids: Constitution of the—.* Synthesis of isoquinoline derivatives. II. A. Kaufmann and N. Dürst. Ber., 1917, 50, 1630—1637. (Compare J. Chem. Soc., 1916, i., 502.)

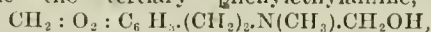
ACCORDING to the commonly-accepted constitution of the morphine alkaloids, these bases do not contain the isoquinoline ring but a special nitrogen-ring of their own, which, however, may be ruptured and the free chain closed up again into an isoquinoline nucleus. This is illustrated by the change from morphine to apomorphine, brought about by heating with zinc chloride solution or concentrated hydrochloric acid:—



The free chain, existing for a moment after the rupture of the "morphine ring," might be  $\text{N}(\text{CH}_3)\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{OH}$ , and accordingly, the authors have attempted to bring about the condensation of piperonyl- $\beta$ -hydroxyethylmethylamine,  $\text{CH}_2:\text{O}_2:\text{C}_6\text{H}_3\cdot\text{CH}_2\cdot\text{N}(\text{CH}_3)\cdot\text{CH}_2\cdot\text{CH}_2\cdot\text{OH}$ , which contains this side-chain, to dihydrohydrastinine,



Unlike the tertiary phenylethylamine,



however, piperonyl- $\beta$ -hydroxyethylmethylamine (a tertiary benzylamine) does not yield an isoquinoline derivative. This is probably due to the fact that tertiary benzylamines readily decompose into benzyl alcohol and secondary amines. Piperonyl- $\beta$ -hydroxyethylmethylamine is prepared by condensing piperonylmethylamine with ethylene oxide.—J. C. W.

*Butyric acid: Detection and determination of—.* G. Denigès. Ann. Chim. Analyt., 1918, 23, 27—31.

FIVE c.c. of butyric acid solution is mixed with 5 c.c. of hydrogen peroxide solution (0.1 vol. strength for 0.01 % butyric acid solution or 5 vols. for a 0.5 % butyric acid solution) and 1 c.c. of ferrous ammonium sulphate solution (5 grms. of ferrous ammonium sulphate and 10 c.c. of 10 % sulphuric acid per 100 c.c.), and the mixture is heated at 70° C. for 5 mins., 6 drops of sodium hydroxide solution are then added, the mixture is cooled rapidly, filtered, and 5 c.c. of the filtrate is treated with 3 drops of sodium hydroxide solution and 3 drops of 5 % sodium nitroprusside solution. After shaking, the mixture is rendered acid with acetic acid. The intensity of the red coloration (due to acetoacetic acid) which develops is proportional to the quantity of butyric acid present and the amount of the acid may be ascertained by comparison with standards. When present in complex mixtures, the acid must first be separated by distillation or extraction.—W.P.S.

*Lactic acid: Determination of—in the presence of other organic acids.* P. Szeberényi. Z. anal. Chem., 1918, 56, 505—506.

ON oxidation with chromic acid, lactic acid yields acetic acid, carbon dioxide, and water, whilst



other organic acids, such as tartaric, citric, malic, and oxalic acids give only carbon dioxide and water. After volatile substances (volatile acids, alcohol, esters, etc.) have been distilled off, a portion of the residual solution corresponding with about 40 c.c. of *N*/1 sodium hydroxide solution is treated with 30 c.c. of 50% chromic acid solution and 20 c.c. of dilute sulphuric acid, the mixture is diluted to 100 c.c., boiled under a reflux condenser for 15 mins., cooled, and the acetic acid formed is separated by distillation, and titrated. About 3% of the lactic acid present is destroyed completely by the oxidation and the results calculated from the acetic acid found, are correspondingly low. A small portion of the lactic acid distils over with the volatile acids. If only acetic and formic acids are also present, it is possible to determine this quantity of lactic acid by titrating the distillate and determining the molecular equivalents of the acids in the usual way; the formic acid, must, of course, be determined separately. (See also *J. Chem. Soc., Mar., 1918.*) —W. P. S.

*Chaulmoogra oil; Use of* — as a specific for leprosy. H. C. Brill and R. R. Williams. *Philippine J. Sci., 1917, 12A, 207—220.* (See also this *J.*, 1904, 669; 1907, 420.)

TEN samples of chaulmoogra oil from various sources were examined and found to have the following physical characters:—

	Maximum.	Minimum.	Average.
Sp. gr. at 30° C. ....	0.9535	0.9429	0.9471
[ $\alpha$ ] <sub>D</sub> in chloroform ..	+58.20°	+45.69°	+50.81°
Acid value (c.c. of <i>N</i> /10 alkali) ....	21.48	1.55	6.79
Saponification value ..	210.5	189.1	200.4
Refractive index ....	1.4774	1.4720	1.4751
Iodine value .....	110.4	97.6	102.4

The mixed free acids from two samples had [ $\alpha$ ]<sub>D</sub><sup>20</sup> = +54.00° and +47.40° (in chloroform); iodine value, 101.3 and 99.7; acid value 35.31 and 34.95 c.c. *N*/10 alkali; m.pt. 38°—39° and 39°—40° C. The chaulmoogric and hydnocarpic acids were isolated and their physical constants were as follows:—For chaulmoogric acid, saponification value, 200.5—202.4; [ $\alpha$ ]<sub>D</sub><sup>20</sup> +59.05° and +58.10° (in chloroform); iodine value 89.5—90.7; for hydnocarpic acid, saponification value, 222.7—218.2; [ $\alpha$ ]<sub>D</sub><sup>20</sup> +67.70° and +67.60° (in chloroform); iodine value, 100.2—99.9. A sample of oil from the seeds of *Hydnocarpus anthelmintica* had sp. gr. at 30° C. 0.9487; [ $\alpha$ ]<sub>D</sub><sup>20</sup> +49.50° (in chloroform) acid value, 0.6 c.c. of *N*/10 alkali; saponification value, 206.2; refractive index, 1.4725; iodine value, 90.8. The use of various fractions of chaulmoogra oil in leprosy is discussed.—W. G.

*Essential oils; Determination of alcohols in* —. T. T. Cocking. *Perfum. and Essen. Oil Record, 1918, 9, 37—38.*

A FORMULA is given by which the amount of free alcohol may be determined in the presence of any ester or mixture of esters provided that these are not affected by acetylation. Let *a* be the saponif. value of the original oil, *b* the saponif. value of the oil after acetylation, and *y* the molecular wt. of the alcohol (if monohydric), then percentage of free alcohol is 
$$\frac{(b-a)y}{0.42016(1335.5-b)}$$
 —W. P. S.

*Chloral hydrate, chloral syrup, etc.; Analysis of* —. M. François. *Ann. Falsif., 1917, 10, 575—581.*

A METHOD for determining chloral hydrate depends on the reaction of this substance with sodium

hydroxide:  $\text{CCl}_3\text{CHO} \cdot \text{H}_2\text{O} + \text{NaOH} = \text{CHCl}_3 + \text{HCOONa} + \text{H}_2\text{O}$ . Provided that the solution is dilute and the time of contact short, secondary reaction between the alkali and chloroform does not take place to any appreciable extent. About 2 grms. of the chloral hydrate is dissolved in 200 c.c. of water, a slight excess of *N*/1 sodium hydroxide is added, and, after 1 min., the mixture is titrated with *N*/1 sulphuric acid, using phenolphthalein as indicator. Each c.c. of *N*/1 sodium hydroxide solution used is equivalent to 0.1655 grm. of chloral hydrate. With pure chloral hydrate the results obtained by the method vary from 99.7 to 100.1%.—W. P. S.

*Carbamides; Constitution of* —. V. Mechanism of the decomposition of urea when heated in solution with alkalis and with acids respectively. Hydrolysis of metallic cyanates. E. A. Werner. *Chem. Soc. Trans., 1918, 113, 84—99.*

THE decomposition of urea on heating with acids or alkalis in aqueous solution is primarily dependent on its dissociation into ammonia and cyanic acid. Its so-called "hydrolysis" is only the hydrolysis of cyanic acid or of an alkali cyanate, and is therefore not a change which gives any support to a "carbamide" structure. Owing to the proportion of free urea being greater in presence of sodium hydroxide than in presence of hydrochloric acid, under comparable conditions the velocity of decomposition is greater in the former case. The ionisation suggested by Masson (*Z. physik. Chem., 1910, 70, 290*) fails to explain the conversion of ammonium thiocyanate into urea or the formation of urea during the hydrolysis of cyanic acid or metallic cyanates, and is not in agreement with the facts. The acceleration of the conversion of ammonium cyanate into urea and the retarding of the decomposition of urea in solution by alcohol are readily explained by and are in complete agreement with the dissociation theory. A sterilised solution of urea does not undergo any "hydrolysis" or reversion to ammonium cyanate at the ordinary temperature; the latter can only arise from the dissociation of the urea as a preliminary stage.—F. W. A.

*Antineuritic properties of the infusorial earth extract of the hydrolysed extract of rice polishings.* Brill. See XIXA.

*Use of different alkalis in quantitative analysis.* Palet. See XXIII.

#### PATENTS.

*Larrea Mexicana or Hedionda; Process of making and extracting the medicinal properties of the* —. C. R. Blundell, Assignor to W. J. Bryan, jun., Tucson, Ariz. U.S. Pat. 1,250,132, Dec. 18, 1917. Date of appl., July 17, 1917.

THE ground pulp of *Larrea Mexicana* is saturated with paraffin oil (sp. gr. 0.9), submitted to direct solar rays for 12 hours, heated to 212° F. (100° C.) for 2 hours, and cooled to 80° F. (27° C.). The mixture is then pressed, and freed from resinous solids by centrifuging.—J. H. J.

*Tuberculosis, leprosy, and other diseases; Substance for treatment of* — and process of mixing said substance. B. S. Paschall, Seattle, Wash. U.S. Pat. 1,250,345, Dec. 18, 1917. Date of appl., May 1, 1917.

THE wax elaborated by tubercle and other acid-fast bacilli is saponified, and an alcohol, a hydrocarbon, and a fatty acid are separated from the products obtained. These three products are then esterified and the esters mixed together. The mixture obtained is claimed to have a therapeutic action upon a host harbouring acid-fast bacilli when administered parenterically.—J. H. J.

*Chlorhydrins; Manufacture of* —. K. P. McElroy, Assignor to Chemical Development Co., Washington. U.S. Pat. 1,253,615, Jan. 15, 1918. Date of appl., June 22, 1915.

A BATH containing a chloride in solution is treated with oil-gas and a current passed through an anode and cathode in the bath, and through a secondary cathode separated from the bath by a diaphragm. Limited oxidation takes place at the anode, the presence of water preventing the formation of olefine chlorides to any considerable extent. The process applies also to the formation of other halogen-hydrins. A solvent for cellulose esters is composed of the chlorhydrins corresponding to the gaseous olefines of oil-gas, the liquid being heavier than water, boiling in the dry state between 125° and 135° C., in the moist state, below 100° C., and being miscible with several times its volume of water.—L. A. C.

*Chlorhydrins; Process of making* —. K. P. McElroy, Assignor to Chemical Development Co., Washington. U.S. Pat. 1,253,616, Jan. 15, 1918. Date of appl., June 17, 1915.

A MIXTURE of steam and oil gas is blown through a horizontal chamber, maintained at about 100° C.; the chamber is provided with baffles, to cause the mixture to take a circuitous path, and successive quantities of chlorine gas are also passed in. At the bottom of the chamber is a quantity of boiling water which absorbs the hydrochloric acid formed, and which is maintained at a low concentration. The hot vapours are condensed to obtain the chlorhydrin.—L. A. C.

*Oxidising hydrocarbons; Process of and apparatus for* —. K. P. McElroy, Assignor to Chemical Development Co., Washington. U.S. Pat. 1,253,617, Jan. 15, 1918. Date of appl., Aug. 2, 1912.

A GASEOUS olefinic hydrocarbon (e.g., ethylene) is subjected to limited anodic oxidation, the hydrocarbon being maintained in excess in the sphere of reaction.—L. A. C.

*Anæsthetic bodies.* Parke, Davis and Co., Assignees of L. Thorp, Detroit, Mich., and E. A. Wildman, Indianapolis, Ind., U.S.A. Eng. Pat. 105,745, Feb. 27, 1917. (Appl. No. 2921 of 1917.) Under Int. Conv., Apr. 17, 1916.

SEE U.S. Pat. 1,193,650 of 1916; this J., 1916, 979.

*Alcohols; Process for manufacturing* —. B. S. Lacy, Sewaren, N.J., Assignor to The Roessler and Hasslacher Chemical Co. U.S. Pat. 1,253,055, Jan. 8, 1918. Date of appl., Oct. 4, 1913.

SEE Eng. Pat. 20,550 of 1914; this J., 1915, 575.

*Preparation of colouring bodies for use in food, pharmaceutical or other preparations.* Eng. Pat. 112,886. See IV.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photography; Colloid chemistry and* —. Lüp-pö-Cramer. Kolloid-Zeits., 1917, 21, 154—155.

PHOTO-MICROGRAPHS of silver chloride crystals, which have been exposed to sunlight, exhibit a very large number of dark points which are considered to be due to aggregates of silver particles.

The pressure developed by the corresponding chlorine leads to disturbances in the crystalline structure which result in disintegration.—H. M. D.

### PATENT.

*Sensitised [photographic] plates; Drier for* —. L. McCue, Assignor to F. L. Harwood, R. W. Albertson, J. H. Gilson, and N. M. Whipple, Chicago, Ill. U.S. Pat. 1,253,990, Jan. 15, 1918. Date of appl., Mar. 19, 1914.

THE device includes a drying chamber, the bottom of which is heated from below and is covered with sand for even distribution of the heat, and an arrangement for keeping the plates in the drying chamber in motion.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Cellulose; Nitration of cotton* —, and influence of prolonged bleaching on its transformation into dinitrocellulose. R. Namias. Monit. Scient., 1918, 8, 5—6.

IN a series of experiments, 5 grms. of cotton of the best quality was nitrated in 200 grms. of a bath containing 60% of sulphuric acid, 24% of nitric acid, and 16% of water, for periods of 2, 4, 12, and 24 hours at 18° C. The nitrogen content of the four products was practically identical, viz., 11.80% to 11.92%, and all were completely soluble in ether-alcohol. The fibre is appreciably weakened if nitration is continued much beyond two hours, or if higher temperatures are employed, and the nitrocellulose is less suitable for making collodion, or for coating incandescent mantles, etc. All the samples burnt instantaneously without leaving a residue. Cotton which had received excessive chlorine treatment showed no difference under the microscope and gave no evidence of tendering, but when nitrated under the same conditions, the percentage of nitrogen in the products varied from 9.86% to 10.37%, and the solubility in ether-alcohol was incomplete. A residue was left on burning, indicating the presence of unnitrated cotton. The author suggests that this may be due to the presence of oxycellulose in the over-bleached cotton.—F. Sp.

### PATENTS.

*Explosive compositions; Production of* —. Commercial Research Co., Long Island City, N.Y., Assignees of H. Hibbert, Pittsburgh, Pa., U.S.A. Eng. Pat. 106,086, Feb. 8, 1917. (Appl. No. 1964 of 1917.) Under Int. Conv., Apr. 17, 1916.

SEE U.S. Pat. 1,231,351 of 1917; this J., 1917, 943.

*Explosive substance.* C. R. Jahn, Schleichbusch, Assignor to Sprengstoff A.-G. Carbonit, Hamburg, Germany. U.S. Pat. 1,253,691, Jan. 15, 1918. Date of appl., Dec. 15, 1913.

SEE Eng. Pat. 18,333 of 1914; this J., 1915, 935.

## XXIII.—ANALYSIS.

*Alkalis in quantitative analysis; Use of different* —. L. P. J. Palet. Ann. Chim. Analyt., 1918, 23, 26—27.

ATTENTION is directed to the necessity of using one and the same alkali for certain determinations in order to obtain concordant results. For instance, in the determination of caffeine in maté,



with calcium oxide as the alkali 0.790% of caffeine was found, with magnesium oxide 0.780%, and with ammonia 0.090%. The difference in the action of alkalis is still more marked in the case of aqueous solutions containing albumin, asparagine, peptone, or urea, *e.g.*, in the case of sewage effluents.—W. P. S.

*Sulphuric acid and barium; Gravimetric determination of — as barium sulphate.* Z. Karaoglanow. Z. anal. Chem., 1917, 56, 487—498.

THE presence of nitric acid or a large quantity of hydrochloric acid causes the results obtained in this determination to be too low; potassium salts and ferric chloride have the opposite effect, the results being too high. The conditions of experiment being equal, the errors produced by the presence of the above substances are always less in the determination of barium than in that of sulphuric acid. (See also J. Chem. Soc., Mar., 1918.)—W. P. S.

*Potassium and sodium as chlorides; Determination of — through the use of the refractometer.* B. A. Shippy and G. H. Burrows. J. Amer. Chem. Soc., 1918, 40, 185—187.

A METHOD for the determination of potassium and sodium as chlorides in the presence of each other depends on the fact that the refractive index of sodium chloride solution is higher than that of a corresponding potassium chloride solution; the difference increases with increase in salt concentration. The refractive indices at 25° C. of 20% potassium chloride and sodium chloride solutions are 1.35992 and 1.36829, respectively. To determine the proportion of potassium and sodium in a mixture of the two chlorides, a 20% solution of the latter is prepared and the refractive index observed; the amounts of the two salts are then found by simple calculation. The method does not possess a high degree of accuracy. (See also J. Chem. Soc., Mar., 1918.)—W. P. S.

*Potassium; Gravimetric determination of — by sodium cobaltinitrite.* C. V. Garola and V. Braun. Ann. Falsif., 1917, 10, 572—575.

THE solution of the potassium salt, containing about 0.25 gm. of potassium chloride and free from other bases except sodium, is evaporated to 25 c.c. and treated with 25 c.c. of sodium cobaltinitrite solution (cobalt nitrate, 28.6 grms., glacial acetic acid, 50 c.c., sodium nitrite, 180 grms., water to 1000 c.c.). After 18 hrs., the precipitate is collected, washed with 10% acetic acid, then once with 95% alcohol, dried at 100° C. and weighed. The weight is multiplied by 0.2074 to obtain the quantity of K<sub>2</sub>O present. The method is accurate and may be applied to fertilisers, soils, wines, etc.—W. P. S.

*Acidity determination in waters, leach liquors, mine waters, etc.* J. J. Rankin. Met. and Chem. Eng., 1918, 18, 96.

THE accurate titration with sodium carbonate of acidic liquors containing sulphates of ferric iron, copper, aluminium, and manganese is impossible owing to the ill-defined end point. This difficulty can be avoided by a reduction of ferric to ferrous sulphate with potassium iodide, the iodine liberated being removed by addition of the proper quantity of sodium thiosulphate. Cupric sulphate is reduced to cuprous iodide which is precipitated, and the iodine liberated is discharged in the same way. In practice a convenient quantity of the liquor is diluted to 250 c.c. and an excess of potassium iodide added. Sodium thiosulphate

(10%) is added until the iodine colour is nearly discharged, and the solution made up to a definite volume. An aliquot part is taken and more sodium thiosulphate added until the colour is finally discharged, when the titration with standard soda can be made, using methyl orange as indicator. Chlorides and nitrates do not interfere nor does aluminium sulphate if sufficient ferrous iron is present.—H. J. H.

*Copper group from the arsenic group; Separation of the — with special reference to the identification of arsenic.* M. C. Sneed. J. Amer. Chem. Soc., 1918, 40, 187—191.

THE mixed sulphides are digested with a reagent prepared by saturating a 12% sodium hydroxide solution with hydrogen sulphide and adding to each litre 400 c.c. of 40% sodium hydroxide solution. Mercuric, arsenic, antimony, and tin sulphides dissolve, whilst lead, bismuth, copper, and cadmium sulphides remain insoluble. The latter are separated by filtration. The filtrate is treated with a quantity of ammonium carbonate sufficient to precipitate the mercury completely, and, after this has been removed, the solution is acidified with hydrochloric acid. The precipitated arsenic, antimony, and tin sulphides are collected, treated with 20 c.c. of concentrated hydrochloric acid, and the mixture is warmed until vigorous evolution of hydrogen sulphide ceases. Arsenic sulphide is not dissolved by this treatment; after the solution containing the tin and antimony sulphides has been separated, the arsenic sulphide is dissolved in ammonia and the solution neutralised with nitric acid; a yellow precipitate forms which is dissolved by boiling with nitric acid, and the solution is treated at 85° C. with an excess of ammonium molybdate. With a large amount of arsenic, a yellow precipitate forms at once, but with traces half an hour's heating may be required to obtain a precipitate. (See also J. Chem. Soc., Mar., 1918.)—W. P. S.

*Zinc; Determination of — by Schaffner's method.* V. Hassreidter. Z. anal. Chem., 1917, 56, 506—509.

THE preliminary separation of the iron, whether by one or more precipitations by ammonia as prescribed in the various modifications of this method, should be made under conditions which prevent as far as possible the occlusion of zinc in the precipitate; the quantity of ammonia used must not be too small. The iron precipitate should be tested for zinc, and a control precipitation with known amounts of iron and zinc should be made at the same time and under the same conditions. (See also J. Chem. Soc., Mar., 1918.)—W. P. S.

*Gas analysis; Applications of —. IV. The Haldane gas analyser.* Y. Henderson. J. Biol. Chem., 1918, 33, 31—38.

MODIFICATIONS of Haldane's apparatus for the estimation of carbon dioxide in air and in blood are described, by means of which the apparatus may be more readily taken apart and cleaned. (See also J. Chem. Soc., Mar., 1918.)—H. W. B.

*Nitrogen; Microchemical estimation of —.* B. Sjollem and C. W. G. Hatterschy. Biochem. Zeits., 1917, 84, 359—370.

A CRITICAL examination of the methods of Bang and of Folin and Denis (this J., 1916, 1133), with some suggested minor alterations in the details. (See also J. Chem. Soc., Mar., 1918.)—S. B. S.

*Determination of total carbon in fuels and its importance in the study of fuel economy.* Damour and De la Morinière. See IIA.

*Detection of selenium in sulphuric acid.* Palet. See VII.

*Determination of silicon in ferrosilicon.* Nicolardot and Koenig. See X.

*Filtration of silica [in analysis].* Nicolardot and Koenig. See X.

*Tentative standard methods for sampling and analysis of commercial fats and oils other than those of the coconut, butter, and linseed groups.* See XII.

*Determination of ash in leather.* Riethof and Gayley. See XV.

*New method for estimation of "zeolitic" silicic acid in soils.* Guedroitz. See XVI.

*Determination of phosphoric acid, particularly in superphosphate.* Vortmann. See XVI.

*Some methods for determination of sucrose content of [cane] molasses.* Van der Linden. See XVII.

*Estimation of phosphorus, hypophosphoric, and phosphoric acid in mixtures.* Van Name and Huff. See VII.

*Dry substance and sucrose determinations in cane molasses.* Steuerwald. See XVII.

*Examination of "85%" flour.* Delchaye. See XIXA.

*Poisonous bread and flour. Detection and determination of sapotorins.* Stœcklin. See XIXA.

*Use of diphenylamine-sulphuric acid for colorimetric determinations [e.g., of nitrates in milk].* Tillmans. See XIXA.

*Effect of time of digestion on the hydrolysis of casein in presence of starch.* McHargue. See XIXA.

*Detection and determination of butyric acid.* Denigès. See XX.

*Determination of lactic acid in presence of other organic acids.* Szeberényi. See XX.

*Determination of alcohols in essential oils.* Cocking. See XX.

*Analysis of chloral hydrate, chloral syrup, etc.* François. See XX.

#### PATENTS.

*Gas-testing apparatus.* H. R. Webster, Horsforth. U.S. Pat. 1,252,975, Jan. 8, 1918. Date of appl., June 29, 1917.

See Eng. Pat. 107,811 of 1916; this J., 1917, 980.

*Method of and means for indicating the condition of steel, iron, or other magnetisable metal during heat treatment.* Eng. Pat. 112,894. See X.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Akt-Ges. Brown, Boveri, et Cie. Operating surface condensing-plant by steam-jet air pumps. 4346. Mar. 12. (Switzerland, June 25, 1917.)

Bingham, and Huntington, Heberlein, and Co. Apparatus for electrical separation of suspended particles from gaseous bodies. 3842. Mar. 5.

Brckett, and Brckett and Co. Screening or filtering apparatus. 3781. Mar. 4.

Broadley. Machines for grinding ores, minerals, stones, etc. 4303. Mar. 12.

Dibdin. Vaporisation of liquids or generation of gases by action of liquids on other matter. 4526. Mar. 14.

Dunsford and Linley. Refrigerating-apparatus. 4561. Mar. 14.

Harrison. 3823. See VIII.

Harvey, and Harvey Gas Furnace Co. 3857. See II.

Harvey, and Morgan Crucible Co. 4682. See X. Hewett. Drying fruit, liquids, chemicals, etc. 3877. Mar. 5.

Howe. Method of obtaining motive power. 4394. Mar. 13.

Hutelins. Means for drying gases. 4443. Mar. 13.

Hutelins. Drying, etc., apparatus. 4444. Mar. 13.

Kilburn (Sulzer Frères). Cooling hot residues from distillation processes. 3997. Mar. 7.

Quinan. Means for elevating liquids. 3930. Mar. 6.

Quinan. Production of chemical reactions. 4343. Mar. 12.

Saunders. Utilisation of heat of gaseous fluids. 4261. 4262. Mar. 11.

Slocum. Process of treating material with gas or vapour. 4452. Mar. 13.

Slocum. Apparatus for treating material with gas or vapour. 4453. Mar. 13.

Stockel. Refrigerating-machines. 4322. Mar. 12.

Stutz. Apparatus for treating material with gas or vapour. 4456. Mar. 13.

Talbot. Means for purifying air. 4196. Mar. 9.

Tuckley. Grinding-machines. 3855. Mar. 5.

Turrettini. Generating heat by chemical reaction. 4041. Mar. 7. (Switzerland, July 17, 1917.)

Williams. Motive power plant. 4563. Mar. 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,366 (1916). Marlow, and Minton, Hollins, and Co. See VIII.

3492 (1917). Bamberg. Grinding-machines. (113,677.) Mar. 13.

4255 (1917). Simon-Carves, Ltd., and Brown. See II.

4686 (1917). Hilger, Ltd., and Merton. See XXIII.

16,744 (1917). Thuman (Crisfield). See II.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Beilby. Carbonisation of coal, shale, peat, wood, etc. 4287. Mar. 12.

Bouillon. Treating peat. 4622. Mar. 15.



Brooke, and Dempster and Sons. Horizontal and inclined retort settings. 4504. Mar. 14.  
 Dempster and Sons, and Toogood. Heating horizontal retorts for gas production. 4203. Mar. 11.  
 Dibdin. 4526. *See I.*  
 Dobson and Pickard. Gas-producers. 4588. Mar. 15.  
 Dobson and Pickard. Combustible gases. 4589. Mar. 15.  
 Easton. Retorts, coke-ovens, etc. 4525. Mar. 14.  
 Garbarini, Gautier, and Maclaure. Arc lamps. 3913. Mar. 6. (France, Oct. 13, 1917.)  
 Hannay. Apparatus for obtaining mixtures of constant composition by evaporation of volatile combustibles by air or gases. 3759. Mar. 4.  
 Harvey, and Harvey Gas Furnace Co. Regenerative gas furnaces. 3857. Mar. 5.  
 Hopkins. Manufacture of composition fuel. 4249. 4626. Mar. 11 and 15.  
 Hutchins. 4443. *See I.*  
 Kilburn (Sulzer Frères). 3997. *See I.*  
 Maxon. Combustion of gas. 4543. Mar. 14.  
 Miller. Combustion of solid fuel in boiler, etc., furnaces. 4259. Mar. 11.  
 Morris. System of supplying explosive mixture to internal-combustion engines for vehicles by self-contained producer-gas plants. 3755. Mar. 4.  
 Rideal and Taylor. 4532. *See VII.*  
 Saunders. 4261. 4262. *See I.*  
 Spicer (Wells). Production and treatment of gas. 4673. Mar. 16.  
 Turrettini. 4041. *See I.*  
 Walker. 3733. *See VII.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,574 (1916). Riedel. Method for preventing the choking-up of gas plant. (101,219.) Mar. 20.  
 17,161 (1916). Hamburger, Lely, and Naanil. Vennoots. Philips' Gloeilampfabrieken. Electric incandescent lamps. (113,626.) Mar. 13.  
 2983 (1917). Rose. Process of manufacturing gaseous fuel. (113,657.) Mar. 13.  
 3962 (1917). Wollaston. Gas-generators. (113,856.) Mar. 20.  
 4255 (1917). Simon-Carves, Ltd., and Brown. Gas-fired coke ovens and muffle and like furnaces. (113,696.) Mar. 13.  
 7084 (1917). Alldays and Onions Pneumatic Engineering Co., and Allday. Apparatus or furnaces for gasifying or vaporising liquid fuel. (113,717.) Mar. 13.  
 11,032 (1917). Sugimoto. *See X.*  
 16,744 (1917). Thuman (Crisfield). Gas washer scrubbers. (113,937.) Mar. 20.  
 19,288 (1917). Skinner. Heating-system for gaseous fuels. (113,945.) Mar. 20.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Blyth and Miles. Means for purifying benzol, etc. 4237. Mar. 11.  
 Hoare. Method of manufacturing benzidine sulphate or benzidine base. 4576. Mar. 15.  
 Watford. Process for synthetic manufacture of benzene. 4654. Mar. 16.

#### COMPLETE SPECIFICATION ACCEPTED.

17,398 (1916). Darzens. Process for the separation of cresols. (107,961.) Mar. 20.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Morris. 4289. 4290. *See XIII.*  
 Perkin. Manufacture of indanthrene. 4585. Mar. 15.  
 Perkin. Manufacture of violanthrene (or indanthrene dark blue). 4586. Mar. 15.

#### COMPLETE SPECIFICATION ACCEPTED.

3287 (1917). British Dyes, Ltd., Turner, and Dean. Manufacture of red azo dyestuffs. (113,825.) Mar. 20.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Bastian. 4206. *See XI.*  
 Boving and Co., and Hellström. Manufacture of paper pulp. 4277. Mar. 11.  
 Cochrane. Treatment of fabrics to render them invisible. 4181. Mar. 9.  
 Fleming. Method of treating paper, etc. 4023. Mar. 7.  
 Henshilwood and Ward. Treatment of fibrous substances. 3898. Mar. 6.  
 Jardine and Nelson. Apparatus for preparing bamboo, etc., for pulp extraction. 4151. 4152. Mar. 9.  
 Kelsey. Process for treating sud or magna obtained in scouring or washing wool, hides, flax, jute, etc. 4516. Mar. 14.  
 Meyer. Manufacture of textile materials. 4542. Mar. 14. (U.S., Apr. 7, 1917.)  
 Robbins. Material for covering wings of aircraft. 4646. Mar. 15.  
 Severn, Thomson, and Twycross. 4029. *See XXII.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

9963 (1916). Fennell, Lane, and Garbutt. Manufacture of paper-makers' pulp. (113,624.) Mar. 13.  
 3873 (1917). Volpato. Process of treating straw in the preparation of textile fibres. (113,853.) Mar. 20.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Poulson. Sizing textile fabrics, etc. 3806 and 4055. Mar. 5 and 8.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Brownson, Jones, and Partington. Production of ammonium nitrate. 3792. Mar. 4.  
 Calvert. Production of ammonia. 4351. Mar. 12.  
 Davis. Production of potash. 3994. Mar. 7.  
 Dutt and Dutt. Process for manufacture and production of alumina. 3767. Mar. 4.  
 Knapp and Taylor. Production of nitrogen compounds. 4454. Mar. 13.  
 Knapp and Taylor. Process of producing ammonia and ammonium compounds. 4455. Mar. 13.  
 Mirza. Zinc-oxide furnaces or kilns. 4272. Mar. 11.  
 Morris. Manufacture of acetate of lead, etc. 4291. Mar. 12.  
 Parkes and Pearson. Fixation of nitrogen of the atmosphere. 4623. Mar. 15.  
 Reid. Process of producing carbide. 4445. Mar. 13.  
 Reid. Apparatus for making carbide. 4446. Mar. 13.  
 Reid. Process for making carbide. 4447. Mar. 13.  
 Rideal and Taylor. Purification of hydrogen. 4532. Mar. 14.  
 Slocum. Preparation of crude materials for manufacture of calcium carbide. 4448. Mar. 13.  
 Slocum. Process for preparation of crude materials for manufacture of metal carbides. 4449. Mar. 13.  
 Slocum. Preparation of crude material for manufacture of carbide. 4450. Mar. 13.

Slocum. Process for fixation of nitrogen. 4451. Mar. 13.

Walker. Purifying hydrogen and removing carbon monoxide. 3733. Mar. 4.

Westcott. Preparation of crude materials for manufacture of calcium carbide. 4457. Mar. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3113 (1917). Hutchins. Manufacture of carbon for electro-chemical, metallurgical, and other services. (113,812.) Mar. 20.

16,456 (1917). British Thomson-Houston Co. (General Electric Co.). Processes for making silicic acid. (113,769.) Mar. 13.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Harrison. Sifters for treating potters' slip, powdered substances, etc. 3823. Mar. 5.

Levi. Refractory substances. 4204. Mar. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,366 (1916). Marlow, and Minton, Hollins, and Co. Ovens or kilns for use in the manufacture of tiles, pottery, and other ware, and for other purposes. (113,794.) Mar. 20.

8400 (1917). Gray (General Electric Co.). Apparatus for drawing cane glass and glass tubing. (113,892.) Mar. 20.

9023 (1917). Snodgrass. Manufacture of artificial abrasives. (113,731.) Mar. 13.

### IX.—BUILDING MATERIALS.

#### APPLICATION.

Müller. Compositions for making slabs, etc., and method of making same. 4551. Mar. 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3080 (1917). Ashenhurst. Porous composition of matter for use in heat insulation and for other purposes. (113,810.) Mar. 20.

5749 (1917). Adlam and Daniels. Brick or block for building or constructional purposes. (113,871.) Mar. 20.

15,400 (1917). Gestetner. Composition such as artificial stone. (113,924.) Mar. 20.

16,674 (1917). Volant. Waterproof material for use in constructional purposes as a substitute for leather, for street paving or the like. (111,485.) Mar. 20.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Alexander, Imbery, and Vint. Hardening and tempering steel, etc. 3825. Mar. 5.

Angel. Treatment and reduction of refractory sulphide, etc., ores. 4676. Mar. 16.

Bingham. Apparatus for heating air blast for iron and steel furnaces. 4385. Mar. 13.

Bingham, and Huntington, Heberlein, and Co. Rabbie-arms for mechanical roasting furnaces. 4427. Mar. 13.

Gayley. Sintering ores. 4545. Mar. 14. (U.S., Mar. 30, 1917.)

Harvey, and Morgan Crucible Co. Crucible melting-furnaces. 4682. Mar. 16.

Industrie-en-Mijnbouw-Maatschappij Titan. Smelting titaniferous iron material. 4633. Mar. 15. (Holland, Mar. 15, 1917.)

Jones. Depositing metal by electric arc. 4101. Mar. 8.

Tetley. Metal-melting pots. 4056. Mar. 8.

Vautin. Recovery of metals from slags. 4681. Mar. 16.

Weir. Manufacture of malleable iron castings. 4239. Mar. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

2391 (1917). Keller. Manufacture of steel. (113,635.) Mar. 13.

3113 (1917). Hutchins. *See* VII.

3414 (1917). Beckett. Refining of metals. (113,834.) Mar. 20.

3422 (1917). Insull. Method of and apparatus for welding or sintering tungsten ingots and the like. (113,835.) Mar. 20.

3423 (1917). Insull. Compressed tungsten powder and the like, and method and apparatus for producing the same. (113,836.) Mar. 20.

11,032 (1917). Sugimoto. Gas burners for blast-furnace stoves. (113,740.) Mar. 13.

13,406 (1917). British Thomson-Houston Co. (General Electric Co.). Manufacture of composite bi-metallic articles. (113,758.) Mar. 13.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Antonoff. Electric batteries. 3856. Mar. 5.

Bastian. Electric heating elements and resistances. 4025. Mar. 7.

Bastian. Electrically-heated clothing, etc. 4026. Mar. 7.

Baur and Treadwell. Electric cells or batteries. 4689. Mar. 16.

Baxter. Electric cells and batteries. 3840. Mar. 5.

Bingham, and Huntington, Heberlein, and Co. 3842. *See* I.

Brown and Morton. Electrolytic cell. 4085. Mar. 8.

Exley and Leitner. Electric accumulators. 4529. Mar. 14.

Exley and Leitner. Manufacture of electric accumulators. 4530. Mar. 14.

Gow. Mounting of electrodes of electric furnaces. 4527. Mar. 14.

Hamer. Conversion of basic or heat-energy contained in energy-carrying fluids into electrical energy. 3829. 3830. Mar. 5.

Howard. Control of electric furnaces. 4428. Mar. 13.

Hoyle and Wrightam. Holders for electrodes of electric furnaces. 4834. Mar. 12.

Illemaun and Montgomerie. 3901. *See* XIII.

Jones. 4101. *See* X.

Pearson. Electric accumulators. 3920. Mar. 6.

Poole. Arc-controlled mechanism for electric arc furnaces. 4153. Mar. 9.

Richardson. Electron discharge devices. 4628. Mar. 15.

Turner and Walker. Mechanism for regulating electrodes of electric furnaces. 3883. Mar. 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3113 (1917). Hutchins. *See* VII.

3318 (1917). Brown, Boveri, and Co., and Gray. Control and regulation of electric furnaces. (113,827.) Mar. 20.

3426 (1917). Insull. Electric furnaces. (113,839.) Mar. 20.

9014 (1917). Bibby. Electric transformers, especially suitable for use in connection with electric furnaces. (113,899.) Mar. 20.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Blundell, Spence, and Co., and Mell. 3801. *See* XIII.



Douglas. Detergent soaps, and making same. 4160. Mar. 9.

Lockwood and Samuel. Treatment of fatty acids or their metallic salts and oils, and manufacture of oxidised products therefrom. 4523. Mar. 14.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Benneche. Paint. 4158. Mar. 13.

Blundell, Spence, and Co., and Mell. Substitute for linseed and other drying oils for use in paints, putty, varnishes, etc. 3801. Mar. 5.

Illemann and Montgomerie. Paint. 3900. Mar. 6.

Illemann and Montgomerie. Non-conducting material. 3901. Mar. 6.

Lockwood and Samuel. 4523. *See* XII.

Mirza. 4272. *See* VII.

Morris. Manufacture of lakes. 4289. Mar. 12.

Morris. Manufacture of chrome yellows. 4290. Mar. 12.

Prior and Warren. Compositions for lacquering, etc. 4274. Mar. 11.

Self. Process for treatment of *Phytelephas* palm, etc., seeds for manufacture of plastic compositions. 4227. Mar. 11.

Self. Composition for use in manufacture of plastic materials. 4629. Mar. 15.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

Broomfield. Manufacture of a rubber compound. 3783. Mar. 4.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Abrahamsen. Tannin-casein compound, and process for manufacture of same. 4347. Mar. 12. (Norway, Mar. 12, 1917.)

Kelsey. 4516. *See* V.

Melbourne and Tilston. Apparatus for treatment of hides, etc. 4015. Mar. 7.

#### COMPLETE SPECIFICATION ACCEPTED.

16,674 (1917). Volant. *See* IX.

### XVI.—SOILS; FERTILISERS.

#### COMPLETE SPECIFICATIONS ACCEPTED.

2819 (1917). Crook and Gilbertson. Manufacture of a phosphatic manure. (113,618.) Mar. 13.

4143 (1917). Guinness and Richards. Method of and apparatus for the treatment of soil. (113,860.) Mar., 20.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATIONS.

Bloxam (Elektrizitätswerk Lonza). 4684. *See* XX.

Ricard. Manufacture of acetone and butyl alcohol by fermentation. 4047. Mar. 7. (France, Aug. 8, 1917.)

#### COMPLETE SPECIFICATION ACCEPTED.

17,338 (1916) and 8804 (1917). Meyer. Process for pressing yeast, and apparatus for use therein. (113,628.) Mar. 13.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Daamen and Van der Hagen. Method for preservation of albumin. 4569. Mar. 15. (Holland, Dec. 12, 1917.)

Dick. Method of making composition butter. 4096. Mar. 8.

Dick. Artificial butter. 4097. Mar. 8.

Goodwin. Treatment of semolina. 3776. Mar. 4.

Hartley. Preserving food. 3768. Mar. 4.

Hartley. Food. 3769. Mar. 4.

Hewett. 3877. *See* I.

Hincks. Butter substitute. 4630. Mar. 15.

Marshall. Method of treating manifolds, etc., from cattle and sheep for production of edible products and feeding-material. 4670. Mar. 16.

Smith. Method of and substance for packing and preserving fruits and vegetables. 4127. Mar. 8.

Talbot. 4196. *See* I.

Talbot. Apparatus for purifying and softening water. 4197. Mar. 9.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,338 (1916) and 8804 (1917). Meyer. *See* XVIII. 9296 (1917). Condensed Food Co. Method of preparing pre-cooked food products. (113,900.) Mar. 20.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Abrahamsen. 4347. *See* XV.

Bloxam (Elektrizitätswerk Lonza). Manufacture of ethyl alcohol from acetaldehyde. 4684. Mar. 16.

Defries and Kernot. Process for manufacture of acetylsalicylic acid. 4620. Mar. 15.

Dreyfus. Manufacture of acetic anhydride. 3797. Mar. 4.

Kernot. Process for manufacture of acidyl derivatives. 4621. Mar. 15.

Ricard. 4047. *See* XVIII.

#### COMPLETE SPECIFICATION ACCEPTED.

2908 (1917). Parke, Davis, and Co. Anæsthetic bodies, and method of preparing the same. (105,744.) Mar. 13.

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATION.

Severn, Thomson, and Twycross. Manufacture of nitrated cellulose products. 4029. Mar. 7.

### XXIII.—ANALYSIS.

#### COMPLETE SPECIFICATION ACCEPTED.

4686 (1917). Hilger, Ltd., and Merton. Radiation pyrometers. (113,865.) Mar. 20.

## I.—GENERAL ; PLANT ; MACHINERY.

*Conduct catalysis. IV. False equilibria.* W. D. Bancroft. J. Phys. Chem., 1918, 22, 22—43.

If a catalysed reversible reaction is brought to a standstill as a result of the poisoning of the catalyst by one or other of the products of the reaction, an apparent or false equilibrium is reached. Various recorded observations are discussed in terms of this mode of explanation. (See also J. Chem. Soc., April, 1918.)—H. M. D.

## PATENTS.

*Separating air or gases from liquids ; Process and apparatus for —.* Rasmussen und Ernst Ges. m. b. H., Chemnitz, Saxony. Eng. Pat. 104,183, Feb. 15, 1917. (Appl. No. 2290 of 1917.) Under Int. Conv., Feb. 16, 1916.

The apparatus consists of an upright receptacle into which the liquid to be treated enters under pressure through a pipe terminating in a nozzle directed upwards. Air separates from the liquid and collects in the upper space of the receptacle, whence it is led through a narrow opening into another smaller receptacle, and thence escapes into the atmosphere. Any liquid carried over by the air is collected in the second receptacle. The process can be repeated as often as necessary by using two or more receptacles connected together. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 1437 of 1880, 17,158 of 1887, 20,468 of 1900, 6011 of 1906, and 18,165 of 1914.)—J. H. P.

*Evaporating plant ; Apparatus for producing and maintaining a variable determined level of liquid in —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 105,543, Jan. 23, 1917. (Appl. No. 1156 of 1917.) Under Int. Conv., Apr. 10, 1916.

The liquid in the evaporator is balanced by a column of liquid contained in an outer vertical tube which communicates with the evaporator and is provided with overflow cocks at different levels.—W. H. C.

*Evaporating liquids ; Apparatus for —.* F. Hart, Easton, Pa. U.S. Pat. 1,251,689, Jan. 29, 1918. Date of appl., Nov. 15, 1917.

A NUMBER of elongated tubular evaporators are arranged horizontally in cascade series. The apparatus is provided at the top with liquid inlet and vapour escape openings, and connecting pipes are arranged between the evaporators to maintain a body of liquid in each with a vapour space above the liquid. The liquid is supplied at the upper end and the concentrated liquid withdrawn at the lower end of the series. Means for heating the evaporators are provided.—J. H. P.

*Evaporator. [Dryer.]* R. W. King and H. G. Miller, The Dalles, Oreg. U.S. Pat. 1,251,696, Jan. 22, 1918. Date of appl., Apr. 14, 1917.

THE apparatus comprises a drying chamber with means for causing a circulation of air through it, and a bleaching chamber or conduit having seals at its inlet and outlet. A conveyor travels through the drying and bleaching chambers, and means are provided for dipping the conveyor through the sealing reservoirs. The material to be dried rests on the conveyor, and is carried through the drying chamber in a circuitous path.—J. H. P.

*Drying material in bulk ; Apparatus for —.* A. Hofmann, Zürich, Switzerland. Eng. Pat. 109,267, Aug. 29, 1917. (Appl. No. 12,380 of 1917.) Under Int. Conv., Aug. 23, 1916.

THE apparatus consists of a drying chamber above which is a heating chamber. The heating gases,

after passing through tubes for heating the air used for drying, are introduced into a flue extending along and to the whole height of the drying chamber, into which they may be admitted at any place required by means of a series of valves arranged at different heights, the heating gases being mixed with the air inside the drying chamber.—J. H. P.

*Fractional distillation.* W. G. Adam, London. Eng. Pat. 112,966, Dec. 30, 1916. (Appl. No. 18,671 of 1916.)

THREE or more stills, each provided with a suitable fractionating column, are arranged at different levels, but not superposed, so that the liquid to be distilled may flow through them in succession. The stills are provided with baffles so that the liquid takes a tortuous course, and they are heated to successively higher temperatures by coils through which the heating medium passes in an opposite direction to that taken by the liquid being distilled.—W. H. C.

*Brick towers, chambers, acid storage tanks, and the like ; Construction of —.* W. A. Coxon, Mitcham. Eng. Pat. 113,015, Mar. 1, 1917. (Appl. No. 2999 of 1917.)

AN inner brick wall or core is faced continuously on both sides with acid-resisting tiles, rebated together and kept in place by grooved tie bricks built into the wall at intervals. The whole is cemented together with acid-resisting cement.—W. H. C.

*Steam ; Generation of —. Feed water heating, and heating of liquids generally.* P. St. G. Kirke, London. Eng. Pat. 113,314, Feb. 15, 1917. (Appl. No. 2258 of 1917.)

IN the generation of steam by means of coal gas, producer gas, and the like, the gaseous fuel is mixed with a primary supply of air insufficient to permit back-firing, and as this mixture issues from the primary mixing chamber an additional supply of air to complete that required for combustion is added to and mixed with it. The mixture passes to a combustion chamber through a secondary mixing chamber where the mixing is completed and the velocity of flow increased above that in the primary mixing chamber and combustion chamber. The combustion of the mixture is completed in contact with an incandescent body of refractory material, as in surface combustion. The gaseous fuel is supplied through a nozzle into the primary mixing chamber, and the combustion chamber may be inside or outside the apparatus in which the steam is generated. The cross-section of the secondary mixing chamber may be uniform or increase from inlet to outlet or from outlet to inlet either gradually or abruptly. The air used may be preheated by the waste gases from the combustion chamber.—J. H. P.

*Centrifugal separators and methods of discharging same.* W. J. Gee, London. Eng. Pat. 113,326 Feb. 21, 1917. (Appl. No. 2579 of 1917.)

IN order to discharge material from a centrifugal separator having a non-perforated drum, such as is described in Eng. Pats. 4155 of 1907 and 9365 of 1915 (this J., 1908, 227 ; 1916, 1206), in such a manner that the various grades remain separated from one another and are thrown into corresponding receptacles, transverse perforated rings are provided which prevent the grades being mixed when the drum is lifted off but allow a free flow of liquid between them. The receptacle into which the solids are thrown is sub-divided into tiers corresponding to the position of the grades so that the



latter are collected separately. Each tier may be provided with a separate scraper and delivery point.—J. H. P.

*Extractor: Centrifugal* —. G. W. Lewis, Grinnell, Iowa. U.S. Pat. 1,253,987, Jan. 15, 1918. Date of appl., June 11, 1917.

THE perforated container of the centrifugal machine is supported at its upper central point so that it can be tilted. It is provided with a level to indicate when it is accurately balanced in a horizontal position. When so balanced the rotating device is moved into gear and started.

—W. H. C.

*Furnace bricks: Treatment of* —. A. McD. Duckham, Ashted, England. U.S. Pat. 1,252,415, Jan. 8, 1918. Date of appl., Dec. 5, 1913.

THE bricks are coated or impregnated with a combustible substance (such as paper) impregnated with a material which liberates oxygen when heated and thus provides a space for the expansion of the brick.—A. B. S.

*Refrigerating machine: Centrifugal* —. V. J. Goetz, Philadelphia, Pa. U.S. Pat. 1,253,026, Jan. 8, 1918. Date of appl., June 10, 1915.

THE gaseous portion of the refrigerating medium is aspirated from a low-pressure chamber into a high-pressure chamber wherein it liquefies and whence it is returned to the expansion chamber. The aspirator is operated by centrifugal force on the compressing medium. The bearings of the centrifugal device are lubricated with the compressing medium.—W. H. C.

*Deflocculating solid materials and agents therefor*. E. G. Acheson, Assignor to Acheson Corporation, New York. U.S. Pat. 1,253,556, Jan. 15, 1918. Date of appl., Oct. 29, 1917.

AMORPHOUS solid materials are treated with an ammoniacal extract of the product obtained by roasting starchy materials at a temperature just below that at which carbonisation takes place.

—W. H. C.

*[Fluid] meter*. C. C. Thomas, Baltimore, Md. U.S. Pat. 1,254,374, Jan. 22, 1918. Date of appl., June 27, 1916.

FLUID flowing in a pipe is heated by an electrical heater to which energy is supplied at a constant rate. An electrical resistance thermometer is arranged with its two elements on opposite sides of the heater, so as to measure the temperature difference, which is registered by suitable recording mechanism. The temperature difference is a measure of the rate of flow of the fluid.—W. F. F.

*Compressing an elastic fluid; Method of* —. H. A. Humphrey, London, Assignor to Humphrey Gas Pump Co. U.S. Pat. 1,254,693, Jan. 29, 1918. Date of appl., June 1, 1909. Renewed June 19, 1917.

SEE Eng. Pat. 12,565 of 1908; this J., 1909, 784.

*Emulsifying apparatus*. H. H. Schou, Assignor to Flakes A./S., Copenhagen. U.S. Pat. 1,254,734, Jan. 29, 1918. Date of appl., July 12, 1916.

SEE Eng. Pat. 17,306 of 1915; this J., 1916, 1052.

*Mechanical feeders for gas producers, furnaces, and the like*. Eng. Pat. 113,203. See IIA.

*Means for heating and boiling liquids*. Eng. Pat. 113,346. See XVIII.

*Apparatus for and processes relating to purifying or treating water or sewage or other liquids*. Eng. Pat. 113,105. See XIXb.

*Treatment of liquids with gases*. Eng. Pat. 113,106. See XIXb.

*Devices for indicating and measuring the flow of gases*. Eng. Pat. 113,197. See XIXb.

*Valves employed in systems supplying gases such as chlorine to liquids such as water*. Eng. Pat. 113,198. See XIXb.

*Apparatus for and process of treating liquid with gases or purifying water*. Eng. Pat. 113,288. See XIXb.

*Diffusers for use in diffusing gases in liquids*. Eng. Pat. 113,374. See XIXb.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Toluol recovery and standards for gas quality*. R. S. McBride. J. Ind. Eng. Chem., 1917, 10, 111—114.

THE gas standards for undertakings in the United States with annual makes above 500 million cub. ft. are tabulated. The legal standards vary largely. In some cities none are prescribed, in some there is a calorific power standard, in some a lighting power standard, while in others the dual standard is in operation. The lighting power standard is sometimes as high as 23 candle power. When carburetted water-gas is made, about 10% of the gas oil used can be recovered as light oil, and of this  $\frac{1}{2}$  can be obtained as pure toluene. Coal gas from horizontal retorts will yield about  $\frac{1}{2}$  to  $\frac{3}{4}$  gallon of light oil per 1000 cub. ft., and of this about 10—12% can be recovered as toluene. For each  $\frac{1}{10}$  gallon of light oil recovered there is a fall in the gross calorific value of 10—14 B.Th.U. per cub. ft. and  $2\frac{1}{2}$ —3 candles decrease of illuminating power. This could be avoided more or less completely by re-carburetting with the benzene or solvent naphtha fraction, but a full enrichment is probably undesirable in the national interest. Examples of the effects of stripping the different types of town gas supplied are given. The suspension or abolition of illuminating standards is advocated to facilitate toluol production and on general grounds. When the lighting power standard is above 18 candles, a reduction might be made to 12—14 candles in a transition stage for, say, one year, so as to prevent consumers' appliances from becoming suddenly unusable. For coal gas plants a calorific power standard of 550—570 B.Th.U. per cub. ft. is recommended.

—H. J. H.

*Toluol production: Intensive* —. I. Proposed improvements in the absorbing and stripping process. F. E. Lichtenthaler. Met. and Chem. Eng., 1918, 18, 144—152.

THE three principal sources of toluene in America are coke-oven gas, town gas, and cracked petroleum products. Of these town gas is considered the one most readily available, and washing for toluol is advocated. Washing by tar is recommended in preference to the oil washing process. Simplicity of plant and operation are the features emphasised. A tar washing arrangement is proposed. Tar is condensed, dehydrated by centrifugal machines, and then allowed to descend a tower scrubber of the cap and seal bubbling type through which the gas ascends. The tar enriched with toluene is then put through a steam stripping still with appropriate heat exchange arrangements, whereby light and carbolic oils are removed, and the product is a "dehydrated" tar suitable for road making. The gas is thus washed with its own tar in countercurrent without circula-

tion. As an alternative to the steam still a direct fired pot still is proposed, consisting of a horizontal cylindrical boiler, along which the tar is caused to flow in a zig-zag path by means of baffles. Live steam is introduced in the last three chambers to complete the stripping.—H. J. H.

*Benzol: A simple method of manufacturing crude* —. W. Greaves. *Coke-Oven Managers' Assoc.*, Feb. 23, 1918. *Gas J.*, 1918, 141, 398—399.

BENZOL is extracted from coal gas by the usual method of scrubbing, and the benzolised oil is pumped under pressure through a cast-iron coil heated to 145° C. by hot furnace gases. The hot oil is then sprayed into a small still or dephlegmator, the interior of which is at atmospheric pressure. The benzol and water vapour pass away to a condenser and separator, whilst the debenzolised oil flows to the oil coolers to be again used for benzol absorption. It is easy to produce the ordinary 65% crude benzol, and the advantages of this method are stated as follows:—Small capital cost, no house required, little ground space, and less expensive parts required than in the ordinary method; inferior fuel may be used for heating the coil; simplicity of working, the fire, pump, and thermometer alone requiring attention; durability and quickness of erection.—J. E. C.

*Oil industry: A new British* —. (I.) E. H. C. Craig. (II.) P. M. Perkin. (III.) A. G. V. Berry and A. E. Dunstan. *Inst. Petroleum Tech.*, Feb. 19, 1918. [Advance proof.]

(I.) BRITISH sources from which oil may be obtained by distillation are oil-shales, coal, cannel coal and torbanites, blackband ironstones, lignite, and peat. Of these the only source at present being utilised is oil-shale, which is mined and retorted only in Scotland. From this source not more than about 300,000 tons of oil fuel for the Navy can be obtained in a year's working. Of the other sources of liquid fuel, coal yields too little and is too valuable to be utilised on a large scale, lignites are not yet available for development on a sufficient scale, and peat has proved troublesome and expensive to treat owing to the difficulty of eliminating water. The remaining possible sources of oil, viz., cannel coals, torbanites, and blackband ironstones, are closely associated, and are, in many cases, easily obtainable. Most cannels contain a very large amount of "jetonised" spores, which are believed to yield on distillation waxes and resinous compounds. Torbanites, though externally similar, are essentially different from true cannels. They consist of a coal matrix in which are embedded yellow or brown globules of "kerogen." These globules vary greatly in size, shape, and number, and contain a considerable proportion of inorganic matter in close, and apparently chemical, combination with hydrocarbons of organic origin. They undergo a degeneration of which four stages have been distinguished, and the quality of oil (paraffins and olefines) yielded on distillation varies according to the stage of this degenerative process, the most degenerated types yielding quantities of heavy paraffin waxes, while the fresher material yields a greater proportion of light oils. Every gradation between a typical torbanite and a typical cannel exists. Owing to the high ash percentage in cannel coals (20 or even 30%, and seldom under 10%), large quantities of cannel have been dumped as useless for fuel purposes, together with impure coaly or canneloid bands ("Jacks, Gees, Rattlers, Batts," etc.) which could not under any pretext be sold as coal. All these deposits, however, contain kerogen globules and uncarbonised vegetable matter from which oil can be obtained

on distillation. Blackband ironstones contain iron oxide or hydroxide in very intimate association with carbonaceous material which is kerogenous or only slightly carbonised, and is thus capable of yielding oil by distillation. Hitherto, blackband ironstones have been prepared for the blast furnace by being burnt in the open with a quantity of admixed coal, but it has recently been demonstrated by Hollingworth (Eng. Pat. 18,103 of 1914; this J., 1915, 1255) that the oil contents of these ores can be extracted by distillation, the nitrogen content being obtained as ammonium sulphate, and sufficient non-condensable gas produced to supply the heat required for the retorts. The residue, designated "carbousiron," consists of ironstone with intimately associated fixed carbon. It is now proposed to exploit the various deposits in this country of cannels and bastard cannels, torbanites, and blackband ironstones, by low-temperature carbonisation, for the extraction of oil and all possible by-products. It is considered probable that over the whole country the cannels and torbanitic cannels will yield from 33 to 35 galls. of crude oil per ton. The petrol obtainable will probably vary from 4 to 10, and the oil fuel from 60 to 70% of the crude oil. The average yield of ammonium sulphate may be estimated at 30 to 40 lb. per ton. The amount of solid paraffin wax recoverable will probably be less than is obtained from the Scotch oil-shales, but an advantage which cannels have over the oil-shales is that the retort residues from the poorer grades can be worked up into very fair briquettes, while those from the better grades can be utilised in producers, to give not only all the power-gas required, but the full yield of ammonium sulphate. Only large central works, situated with due regard to supplies and facilities for transport, and capable of dealing with at least 1000 to 2000 tons of cannel per day are projected. At least six such retorting and refining works could be established without any difficulty. The goal aimed at is some 400,000 tons of oil fuel per annum, and some 6 or 7 million gallons of petrol, besides intermediate oils.

(II.) The urgent importance at the present juncture of increasing the country's oil-fuel supplies is emphasised, and the question of the relative advantages of high- and low-temperature carbonisation of coal, etc., discussed. High-temperature treatment is necessary for the production of benzol and other aromatic compounds required for the manufacture of high explosives, but the yield of oil is low. Low-temperature carbonisation on the other hand gives a maximum yield of oil, but aromatic compounds are absent. By passing steam through coal as it is being carbonised at a high temperature in vertical retorts, considerably larger yields of gas and tar are obtained, but the tar contains very little benzol and toluol.

(III.) The methods employed in the testing of cannel coals for yield of oil are described, and the nature and refining of the crude oil discussed. The results of several tests are given. Crude cannel oil has the following properties:—Sp. gr., 0.887 to 1.000; sulphur, 0.4 to 1.1%; setting point, 5° to 30° C.; water, after partial separation, 1 to 4%; calorific value of dry oil, 9.550 to 9.655 cal. per grm. (17,190 to 17,379 B.Th.U. per lb.). The following fractions are obtainable by refining:—Benzene, approximately 3%, sp. gr. about 0.740 to 0.750, with a final boiling point of 170° C. Intermediate oil, approximately 12%, sp. gr. about 0.800, flash point about 80° F.; such an oil would be an excellent light fuel for tractors, etc. Navy fuel oil, approximately 50 to 60%, sp. gr. about 0.870, flash point over 170° F., and setting point below 25° F. Paraffin scale, variable, averaging approximately 3 to 4%. Pitch, hard or soft as required, more bituminous in character than coal-tar pitch, and at the worst, mixed with oil, would produce a works fuel.—T. ST.



*Improvements in apparatus for the manufacture of concentrated ammonia liquor.* Greaves, *See* VII.

# PATENTS.

*Coke-ovens.* D. Campbell and C. Goodyear, Vancouver, B.C., Canada. Eng. Pat. 112,967, Jan. 12, 1917. (Appl. No. 629 of 1917.)

A SPACE is formed beneath the floor of a coking chamber, communicating on the one hand by means of parallel interspaces with the chamber and, on the other, by means of a floor sloping to an aperture covered by a grating, with means for inducing draught. The space is filled with a permeable mass of finely-divided material, such as gravel or rock, and trap channels may be provided to remove condensed liquid products.—J. E. C.

*Coke-oven.* L. Wilputte, New Rochelle, N.Y. U.S. Pat. 1,253,760, Jan. 15, 1918. Date of appl., May 2, 1917.

REGENERATORS are placed below each half of the coking chamber and parallel thereto. Air and gas are supplied to the regenerators alternately, and each regenerator is provided with a separate waste gas outlet. One set of regenerators is connected by conduits to a common flue, the alternate set being similarly connected to a second main flue.

—J. E. C.

*Coke-oven.* J. E. Hubbell, Philadelphia, Pa., Assignor to L. Wilputte, New Rochelle, N.Y. U.S. Pat. 1,254,007, Jan. 15, 1918. Date of appl., June 14, 1917.

THE regenerators are situated under each half of the heating wall of the coke-oven and parallel to the same. Two passages communicating longitudinally with the base of the regenerators are provided, with means for supplying fluid to be preheated to one, and for withdrawing waste gases from the other.—J. E. C.

*Gas producers.* T. R. Wollaston, Manchester. Eng. Pat. 113,025, Mar. 19, 1917. (Appl. No. 3964 of 1917.)

A GAS producer is divided at or about the position of the grate into two or four distinct sectors, each provided with one or more pairs of crusher rolls movable independently. The crushed clinker from these rolls falls into a water-lute and is propelled by water jets to a desirable part of the plant for removal.—J. E. C.

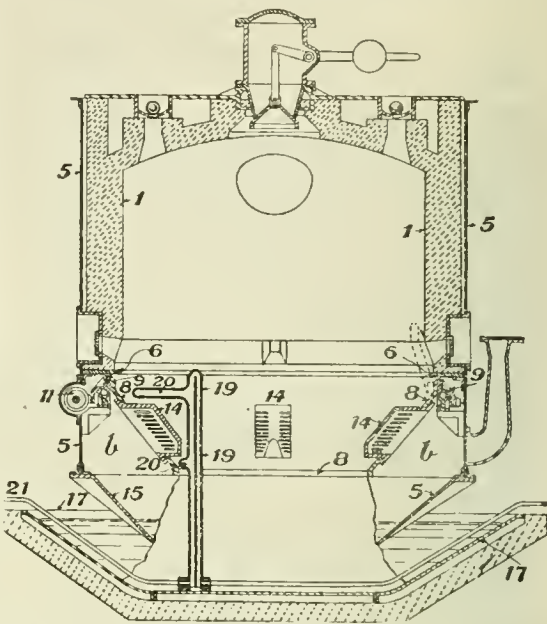
*Gases: Generation of* — [by electrolysis] for use in internal combustion engines. G. C. Colona, Kelso, Scotland. Eng. Pat. 113,048, May 21, 1917. (Appl. No. 7274 of 1917.)

A LIQUID, *e.g.*, water, is decomposed by the action of an electric current passing between two electrodes, one in each section of a divided receptacle. The gases from one section escape to the atmosphere, whilst those from the other section are conducted to the combustion chamber of the engine.—J. E. C.

*Gas producers.* J. Stewart, Glasgow. Eng. Pat. 113,182, Apr. 10, 1917. (Appl. No. 1982 of 1917.)

IN a suction or pressure gas producer, the upper part consists of an outer casing, 5, with a refractory lining, 1, resting on an annular plate, 6, and provided with the usual inlet and outlet openings. The conical base, 8, may be built up of a number of plates attached to the ring, 9, which forms one member of a ball bearing and also carries worm teeth so that it may be slowly rotated by the worm, 11, operated by hand or power. The base, 8, is also provided with two or more grates, 14, with inclined sides and having slots so arranged that air or a mixture of air and steam passing

through them tends to pass over the inner surface of the base, 8. One or more water-cooled pokers, 19, having projecting arms, 20, are supplied with water by the pipe, 21. The lower part, 15, of the casing is conical and dips into the water in the trough, 17. Air mixed with steam produced in the



chamber, *b*, enters the producer at the lower edge of the base piece, 8, and through the grates, 14. In a modification an annular opening is also provided between the ring, 9, and annular plate, 6, forming an additional communication between the chamber, *b*, and the producer chamber.—W. F. F.

*Gas producers, furnaces, and the like: Mechanical feeders for* —. E. W. Harvey, London, and E. N. Wright, Wolverhampton. Eng. Pat. 113,203, May 10, 1917. (Appl. No. 6626 of 1917.)

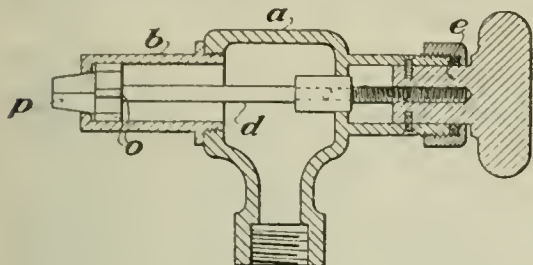
IN an apparatus suitable for feeding fuel mechanically to circular gas producers, furnaces, and the like, the fuel contained in a hopper is distributed by an oscillating circular valve of known form into an open-ended oscillating and revolving shoot which delivers it into the furnace. The distributing shoot is oblong in horizontal cross-section, and is supported near its upper end by horizontal trunnions journaled in the wall of a hollow cylinder surmounting the mouth of the furnace. The cylinder is provided with annular water seals at the top, where it engages with the supporting framework of the distributing valve, and at the bottom, where it engages with the top of the furnace. The hollow cylinder is rotated slowly and the trunnions carrying the distributing shoot are oscillated. The fuel is thus distributed by the shoot during its oscillation over a diameter of the furnace, and the plane of oscillation of the shoot is gradually rotated so as to cover the whole circumference of the furnace.—W. F. F.

*Gas producer.* H. J. Ambruson, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pat. 1,252,612, Jan. 8, 1918. Date of appl., Jan. 25, 1915.

A GAS producer comprises a fuel shell, and blast-distributing means consisting of hollow radial arms in communication with an annular blast-distributing member. The shell and blast-distributing means rotate in unison.—J. E. C.

*Bunsen burners.* Fletcher, Russell and Co., T. W. Fletcher, and W. Banks, Warrington. Eng. Pat. 113,069, Aug. 18, 1917. (Appl. No. 11,879 of 1917.)

A device for regulating the gas supply and adjusting the area of the nipple orifice of a Bunsen burner comprises a casing, *a*, a nipple, *b*, a sliding



non-rotating stem, *d*, a nut, *e*, a pyramidal portion, *p*, adapted to co-act with an orifice of corresponding shape, and supporting wings, *o*.—J. E. C.

*Gas burners for furnaces.* Fletcher, Russell and Co., Ltd., T. W. Fletcher, and J. H. Singleton, Warrington. Eng. Pat. 113,239, Sept. 21, 1917. (Appl. No. 13,583 of 1917.)

THE mixture tube of a gas burner for furnaces is provided with two or more fan-shaped nozzles projecting laterally, arranged in line or "staggered," and spaced close together along the tube. —W. F. F.

*Hydrocarbon oils and spirits; Refining* — J. Nelson, Glasgow. Eng. Pat. 113,131, Feb. 6, 1917. (Appl. No. 1864 of 1917.)

HYDROCARBON oils and spirits obtained by cracking kerosene, solar oil, and other heavy hydrocarbons, are decolorised, deodorised, and the tendency to develop gumminess removed, by treating them with an unsaturated fatty oil such as linseed, cotton, rape, or other oil, with or without phosphoric acid. For example, volatile hydrocarbons boiling below 150° C., obtained by cracking kerosene, are mixed with 20% of raw linseed oil, and the mixture is distilled. The residue in the still may be used for treating a further quantity of spirit. In a modification, a heated chamber containing pumice soaked in linseed oil, or a heated tower down which linseed oil flows, is interposed between the still and condenser. A second purifying chamber containing pumice and phosphoric acid may also be interposed between the linseed oil chamber and the condenser. Further purification may be effected by storing the treated hydrocarbon over water containing 0.5% of caustic soda, or by passing the vapours over soda-lime heated to a temperature above the condensing point of the vapours. Hydrocarbons containing constituents boiling up to 250° C. may also be treated by this process, with or without the use of a dephlegmator to separate the lighter fractions of the cracked oil.—W. F. F.

*Light oils; Art of recovering* — [from gas]. R. B. Parker, Assignor to Somet-Solvay Co., Solvay, N.Y. U.S. Pat. 1,252,481, Jan. 8, 1918. Date of appl., Mar. 6, 1916.

LIGHT oil vapours are absorbed from gas by a solvent oil. The volatile components of this oil are distilled off, the more volatile compounds separated from the heavier distillates including the olefines, and the latter purified with sulphuric acid and re-distilled.—J. E. C.

*Oil; Apparatus for dehydrating* — R. R. Pollak, San Francisco, Cal. U.S. Pat. 1,251,271, Jan. 22, 1918. Date of appl., July 6, 1914.

From a perforated partition in a vessel, oil drips on to a horizontal series of perforated plates and thence flows on to coils of steam pipes. The supply of steam can be controlled by hand and by thermostatic means. Below the pipes is a vertical series of inclined, perforated drip plates, below which are other coils of steam pipes. The vessel is also provided with a cooling chamber and a conduit containing means for heating the air therein, and a condenser leads from above the cooling chamber to a centrifugal blower of regulated speed, whilst another conduit containing two condensers with receiving chambers leads from an upper portion of the vessel to the blower.—L. A. C.

*Gas-producer plants; Generator for* — G. H. Allen, North Fremantle, W. Australia. U.S. Pat. 1,251,107, Jan. 22, 1918. Date of appl., July 27, 1915.

SEE Eng. Pat. 10,881 of 1915; this J., 1916, 1005.

*Process and apparatus for calcining and roasting certain ores [by means of exhaust gases from gas engines].* Eng. Pat. 105,915. See X.

*Process for the manufacture of drying-oils [from kerosene].* U.S. Pat. 1,251,566. See XIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*A new British oil industry.* Craig and others. See IIa.

### PATENTS.

*Charcoal; Production of decolorising* — R. von Ostrejko, Cracow, Austria. Eng. Pat. 106,089, Apr. 3, 1917. (Appl. No. 4813 of 1917.) Under Int. Conv., May 4, 1916.

POWDERED charcoal is heated in a retort, and steam, superheated by the waste gases of the furnace, is admitted at the top of the retort. The gases thereby generated pass from the bottom of the retort to the heating furnace in which they are burnt with a regulated supply of air. After treatment with the steam a portion of the charcoal is removed from the retort proper and further heated in boxes in the hearth of the heating furnace, whilst a corresponding quantity of fresh charcoal is supplied to the retort.—J. E. C.

*Coal; Distillation of* — G. E. Heyl, London. Eng. Pat. 113,012, Feb. 22, 1917. (Appl. No. 2630 of 1917.)

To obtain a greater yield of oil from coal, it is soaked in about 10% of its mass of mineral or coal-tar oil, and after distillation up to about 200° C., an additional 10% is added and the distillation continued up to a temperature of about 400° C.—J. E. C.

*Bituminous coal; Destructive distillation of* — F. C. Blythe, Southsea, Hants. Eng. Pat. 113,079, Oct. 29, 1917. (Appl. No. 15,728 of 1917.)

THE distillation is carried out at a comparatively low temperature, under pressure, on coal to which heavy hydrocarbon oil has been added, whereby the production of hydrocarbon oils is favoured at the expense of permanent gas (see Eng. Pat. 19,750 of 1914, this J., 1915, 1016). When the suitable temperature has been attained the pressure is released and the distillation completed at atmospheric pressure or under a vacuum.

—H. J. II.



*Anti-cathode.* E. W. Kelly, Philadelphia, Pa. U.S. Pat. 1,251,700, Jan. 29, 1918. Date of appl., Sept. 30, 1916.

AN iron-nickel alloy, having a coefficient of linear expansion approximately equal to that of tungsten, is used as the anti-cathode, and a refractory member of tungsten is welded directly to the mass.—B. N.

### III.—TAR AND TAR PRODUCTS.

*Toluol recovery and standards for gas quality.* McBride. See IIA.

*Intensive toluol production. I. Proposed improvements in the absorbing and stripping process.* Lichtenthaler. See IIA.

*A simple method of manufacturing crude benzol.* Greaves. See IIA.

*Influence of different compounds on the destruction of monosaccharides by sodium hydroxide and on the inversion of sucrose by hydrochloric acid. III. Constitutional formulae of the hydroxy-benzoic acids and of sulphonic acid.* Waterman. See XVII.

*Process for deodorisation of the cresols.* Couch. See XIXB.

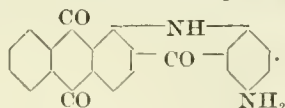
#### PATENT.

*Art of recovering light oils [from gas].* U.S. Pat. 1,252,481. See IIA.

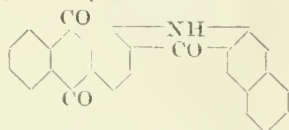
### IV.—COLOURING MATTERS AND DYES.

*Dyes of the 1,2-phthaloylacridone ["anthraquinone-acridone"] series.* F. Ullmann and P. Dootson. Ber., 1918, 51, 9—24.

New acridone dyes have been obtained by condensing 1-aminoanthraquinone with the methyl ester of a substituted *o*-chlorobenzoic acid, hydrolysing the ester so formed, and treating the free *o*-anthraquinonylaminobenzoic acid with concentrated sulphuric acid. The parent 1,2-phthaloylacridone (always designated "anthraquinone-2,1-acridone" by Ullmann) can also be obtained most readily, from methyl *o*-chlorobenzoate, by this method. 6-Amino-1,2-phthaloylacridone



crystallises in bluish-violet tablets, and dyes cotton in a hydrosulphite vat the same shade, whereas the unsubstituted phthaloylacridone gives reddish-violet shades. 6-Methoxy-1,2-phthaloylacridone forms small, violet leaflets, and gives a vat of the same colour. 7-Methoxy-1,2-phthaloylacridone separates as a wine-red mass of needles, and dyes cotton red. 1,2-Phthaloyl-



6,7-benzoacridone forms dark violet needles, gives a green vat, and dyes cotton bluish-violet, whereas the isomeric 5,6-benzo-compound, "Indanthrene Red B.N. extra" of Ger. Pat. 237,236 (this J., 1911, 1153), dyes red shades. (See also J. Chem. Soc., April, 1918.)—J. C. W.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Aeroplune dopes.* Esselen, jun. See XIII.

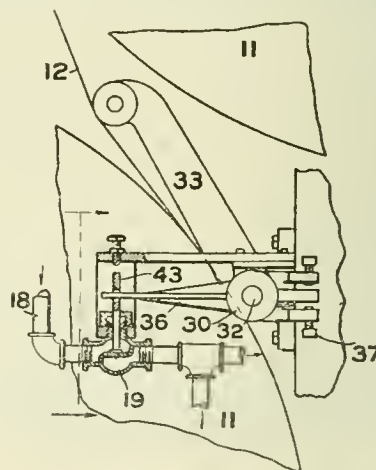
#### PATENTS.

*Flax fibre; Process and apparatus for treating [decorticating]—.* A. Meiro, Moscow. Eng. Pat. 113,285, Jan. 26, 1916. (Appl. No. 1224 of 1916.)

FLAX fibre obtained from dry flax straw or from wet steeped or dew-retted flax is broken by passing between smooth horizontal rollers. Separate bunches of the fibre are then folded in half and the bundles tied tightly with string, pushed into horizontal tubes attached to the vertical shaft of the apparatus and secured on hooks therein. The apparatus consists of a vertical, jacketed cylinder with a central shaft carrying brackets to which the horizontal tubes already mentioned are attached, those tubes near the centre being larger in diameter than those further away. The tubes are provided with small turbines at one end, and the hooks to which the flax is attached are fixed on the vanes and axes of the turbines. When the tubes are filled with flax the vessel is charged with water and crude linseed oil, or with water alone, and the shaft is caused to rotate. The heated liquid passes through the tubes parallel with the flax fibre and causes the turbines together with the attached bundles of flax to rotate within the tubes. The refuse is thus detached, and is separated from the discharged liquid by sieves. Subsequently the cleaned fibre is drained and then dried by circulating air through the tubes.—J. F. B.

*Paper-making and the like; Automatic steam control mechanism for the drying cylinders used in —.* C. Stickle, Indianapolis, Ind., U.S.A. Eng. Pat. 113,240, Sept. 25, 1917. (Appl. No. 13,803 of 1917.)

THE sheet of paper to be dried passes in zig-zag form over a series of heated cylinders, portions of two cylinders, 11, being shown. A spindle, 32,



mounted in a fixed bearing sleeve, 30, carries the two arms, 33, 36, of a bell-crank lever. The arm, 33, is provided with a roller which rests on the sheet of paper, 12, as it passes through the machine, and the arm, 36, passes through an elongated opening in the spindle, 43, of a valve, 19, in the pipe, 18. Stops, 37, on an extension of the arm, 36, are provided to limit the movement of the arm. Pressure water is supplied to the pipe, 18, and operates a reducing valve in the main which supplies steam to the drying cylinders. When the

sheet, 12, is too damp it sags downward and the arm, 33, follows it, tending to close the valve, 19. The pressure water which holds the steam reducing valve in reducing position is thus cut off, and the reducing valve allowed to open to admit more steam to the drying cylinders. When the paper is relatively dry, it contracts, the arm, 33, moves upward, and the valve, 19, opens, admitting pressure water to close the reducing valve, thus diminishing the supply of steam to the drying cylinders.—W. F. F.

*Paper and fabrics [embossing paper]; Process and apparatus for covering — with metal foil.* F. Tscheike, Fürth, Bavaria. Eng. Pat. 113,297, Jan. 16, 1917. (Appl. No. 813 of 1917.)

PAPER is coated with a resinous adhesive which only acquires its maximum adhesiveness at a high temperature or under the action of volatile solvents; it is then passed over a stretching cylinder on to a heated support and the foil is applied to the sticky surface of the paper and attached thereto by a pressure roller or polished steel press-plate, preferably cooled with water. Sheets of foil may be applied by hand with intermittent feeding of the paper, using a horizontal heated support, or rolls of foil may be applied mechanically by continuous feeding against the paper stretched on a heated cylinder and pressed by means of a water-cooled pressure roll also in contact with the cylinder. Embossing paper is made by covering the metal side of the coated paper with a second coating of adhesive of greater adhesive strength than the first, so that, when applied to a foundation by the pressure of a heated die, the foil adheres to the foundation and is released from the paper-backing in the same operation.—J. F. B.

*Size and process of making same [from starch].* U.S. Pat. 1,253,397. See XVII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Bleaching, scouring, and decorticating fabrics or fibrous matter.* A. Poulson, Widnes. U.S. Pat. 1,254,727, Jan. 29, 1918. Date of appl., Feb. 12, 1917. SEE Eng. Pat. 104,048 of 1916; this J., 1917, 383.

*Dyeing.* H. H. Kelsey, Assignor to N. Malcolmson, London. U.S. Pat. 1,254,810, Jan. 29, 1918. Date of appl., June 4, 1917.

SEE Eng. Pat. 107,605 of 1916; this J., 1917, 960.

*Size and process of making same [from starch].* U.S. Pat. 1,253,397. See XVII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Ammonia; Oxidation of — to oxides of nitrogen.* W. G. Adam. Chem. Trade J., 1918, 62, 181—182.

WILTON and Davis studied this reaction in 1908 at the gas works at Beckton, both on the laboratory and works scale, but until 1917 the relative prices of nitre and ammonia prevented its commercial application for the manufacture of nitric acid. Since Aug. 1917, several sulphuric acid plants on the works have been supplied with nitrous gases produced by oxidation of ammonia instead of by the use of nitre pots. Early experiments showed that the catalyst is best used in the form of a wad of platinum gauze,  $\frac{1}{2}$  in. thick. The reaction is started by inserting a plug carrying an electrically heated spiral close up to the gauze.

The ammonia air mixture, which is normally 1:10, is temporarily enriched until the reaction is in active progress, when the heat of the reaction suffices. The time of contact of the mixture with the catalyst was estimated at 0.0046 sec. at 600° C. The oxidation of ammonia must be complete as any remaining unconverted will react with the oxides of nitrogen formed producing free nitrogen. A minimum time of contact is thus necessary, and it must be in one stage so that oxides of nitrogen will not be in contact with unchanged ammonia. Four layers of platinum gauze in the wad sufficed and no advantage was secured by the use of more. The yields in the works were checked by absorption of the oxides of nitrogen in sulphuric acid of 144° T. (sp.gr. 1.72) and came up to those obtained in small scale plant.—H. J. H.

*Nitrogen problem in Switzerland.* P. A. Guye. Schweiz. Chem.-Zeit., 1918, 2, 3—6.

OF the processes of nitrogen fixation discussed in the recent United States report (this J., 1917, 1081—1085), the arc process is considered most suitable for Swiss conditions as water power is plentiful and the process requires practically no imported raw material. The annual national requirements are estimated at 5000 tons of nitric acid and 5000 tons of nitrate fertilisers, which together would require about 20,000 kilowatts or 27,000 H.P., whilst the available water power in the country is about 2 million H.P. The necessity for transporting nitric acid could probably be obviated by collecting the oxides of nitrogen from the arc gases, not by absorption in water but in the form of liquid nitrogen peroxide. This boils at 26° C. and can be transported in sheet iron tanks and converted directly into 96% nitric acid by means of water and air at the centres where the acid is required. The arc process could probably be made to supply the Swiss demand for ammonia, estimated at about 800 tons  $\text{NH}_3$ , for recent laboratory experiments show that gaseous nitric acid can be reduced to ammonia, to the extent of 70%, by means of hydrogen and finely divided nickel, at temperatures between 250° and 300° C. and at atmospheric pressure —J. H. L.

*Ammonia liquor; Improvements in apparatus for the manufacture of concentrated —.* W. Greaves. Coke-Oven Managers' Assoc., Feb. 23, 1918. Gas J., 1918, 141, 398.

SINCE the outbreak of the war there has been a great demand for concentrated ammonia liquor containing a minimum of 25%  $\text{NH}_3$  and a maximum of 0.5%  $\text{H}_2\text{S}$ , as against a pre-war standard of about 16 to 18%  $\text{NH}_3$ . The apparatus required for the former consists of an ammonia still and its accompanying lime still, large enough to deal with the various ammonia liquors sent to them, and to allow the added lime to combine with sufficient hydrogen sulphide to satisfy the final test, at the same time removing as much carbon dioxide and cyanide as possible. In addition a still is required to expel a portion of the hydrogen sulphide, and some means of condensing the excess of steam in the ammoniacal vapours. The author describes a method of converting a sulphate of ammonia plant into a concentrating plant. The change necessitated the provision, in addition to three ammonia stills already installed, of an ammonia still, lime still, and two reflux coolers, together with pumps, piping, etc. The incoming gas liquor passes through a reflux condenser, cooling the vapours from the still, and becoming itself heated to about 71° C. It then passes to one of the old stills where it meets a little live steam on its way down the still, the aim being to drive off a maximum of hydrogen sulphide and carbon dioxide, with a minimum of ammonia. The original liquor



contains 0.75%  $\text{NH}_3$  and 0.23%  $\text{H}_2\text{S}$  and the partly desulphurised liquor contains 0.69%  $\text{NH}_3$  and 0.15%  $\text{H}_2\text{S}$ , so that 35% of the latter is removed at the expense of 8% of the ammonia. This figure can be improved but only by increasing the ammonia content of the waste gases passing to a special scrubber. As the quantity of ammonia passing thence to the ammonia still is limited by the amount of hydrogen sulphide which can be extracted in the lime still, it is a mistake to work with strong gas liquor when making concentrated liquor. At the plant referred to, water was added in the scrubbers to bring the ammonia content down to 0.7%. The waste gases from the desulphuriser consist of hydrogen sulphide, hydrocyanic acid, and carbon dioxide, along with their ammonia compounds. A small steam jet in the outlet pipe assists in overcoming troubles due to deposits of ammonium carbonate. The partly desulphurised liquor passes on to the lime still, and it has been found advantageous to allow the entry from the lime still into the ammonia still to be made higher than in the case of ammonium sulphate manufacture, to allow more time for the lime to act on the ammonium carbonate; in addition more lime is required. The ammonia and steam from this still pass through the first reflux cooler to extract some of the water vapour and thence through a second reflux cooler and final condenser, the finished product showing an average of 28 to 29%  $\text{NH}_3$  and 0.20%  $\text{H}_2\text{S}$ . The temperatures at the control points of the plant are:—Gas liquor entering first reflux condenser, 15° C.; liquor entering desulphuriser, 71° C.; vapours leaving desulphuriser, 77° C.

—J. E. C.

*Potash and other constituents; Extraction of— from sea water bittern.* J. H. Hildebrand. J. Ind. Eng. Chem., 1918, 10, 96—105.

AROUND San Francisco Bay 140,000 tons of salt is produced annually by the solar evaporation of sea water. The salt is accompanied by 3000 tons of potassium chloride and still larger quantities of magnesium salts which are now wasted. The possibilities of recovering this potash from the bitterns remaining after the removal of salt are discussed in the light of van't Hoff's studies of oceanic salt deposits. It appeared that if the evaporation, crystallisation, and filtration of the bittern were carried out at a high temperature, magnesium chloride and sulphate and salt could be separated almost free from potassium salts, while the potassium chloride would become concentrated in the mother liquor. On cooling the mother liquor, carnallite ( $\text{KCl} \cdot \text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) crystallises, and by extraction with a limited quantity of water, magnesium chloride passes into solution and potassium chloride is left. In practice it is recommended to concentrate the mother liquor from solar evaporation pans until its boiling point is 120° C. and its sp. gr. 1.35 at the boiling point. The solid should be separated from the hot liquid by settling in a steam jacketed tank and by centrifuging the precipitate hot. The mother liquor is cooled to allow carnallite to crystallise. From the latter magnesium chloride is extracted, using water equal to about one-half the weight of the carnallite, and potassium chloride crystals are left. The methods of recovering the sulphate and chloride of magnesium and bromine are discussed. Attention is drawn to a similar process proposed by Nishimura (J. Chem. Ind. Tokyo, 1917, 20, 587; this J., 1917, 1046.)

—H. J. H.

*Cement mill dust; Direct heat treatment of— to increase its water-soluble content.* A. R. Merz. J. Ind. Eng. Chem., 1918, 10, 106—109.

THE character of the flue dust from cement kilns using the Cottrell precipitator differs according

to the fuel used. In one works using oil-fired kilns, 92% of the potash in the dust was soluble in water. From two coal-fired plants the proportions of water-soluble potash were only 60% (A) and 41% (B). A further proportion of the potash which could be extracted by 5% hydrochloric acid is classed as "slowly soluble." This has been usually regarded as due to re-combination of potash with the ash of the coal used as fuel. Nestell and Anderson (this J., 1917, 926) state that on extraction for 10 hours with boiling water, it becomes soluble. Porter (Portland Cement Assoc., Chicago, Sep., 1917) claims that the addition of salt to the charge prevents re-combination of the potash. Ross (this J., 1917, 642) has shown that digestion with lime water under pressure is effective. Potash can be set free from silicates by heating them with lime, and as the latter is present in flue dust, simple ignition should have some effect. On heating the dusts with an equal quantity of calcium carbonate for 40 mins. at 1000° C. in a Lawrence Smith crucible, the water-soluble portion of A (see above) rose from 60% to 90%, while in the case of B it fell from 41% to 30%, the re-combination having gone still further. Dust A was grey, while B was black and contained 9.26% of carbon and also sulphide. On igniting the dusts alone under oxidising conditions in a muffle at temperatures varying from 600° to 1100° C. the potash became almost completely soluble in both cases—98% in A and 93% in B. No advantage was attained by prolonging the heating above 20 minutes.—H. J. H.

*Potash; Effect of coal ash on liberation and nature of cement kiln—.* N. S. Potter, jun., and R. D. Cheesman. J. Ind. Eng. Chem., 1918, 10, 109—111.

THE authors dispute the view that the insoluble and slowly soluble portion of the potash in flue dust from coal-fired cement kilns is due solely to re-combination with the coal ash. Some may be due to unchanged coal ash rich in potash. The average  $\text{K}_2\text{O}$  content of coal ash used in a certain cement works is about 5% and as the coal consumption in cement kilns varies from 80 lb. to 250 lb. per barrel of cement, this ash alone can furnish from 0.4 to 1.25 lb. of  $\text{K}_2\text{O}$  per barrel. The ash is so finely divided that little is retained by the charge especially when the dry process is used. Samples taken at every 10 ft. of the length of a kiln showed that some potash was being absorbed by the charge in its progress and probably from the coal ash. The bulk of the ash probably passes forward unchanged, with its potash insoluble, to be collected by the precipitator. Laboratory experiments show that simple heating does not render the potash of the coal ash soluble.—H. J. H.

#### PATENTS.

*Acetic acid; Manufacture of—.* H. Dreyfus, Basle, Switzerland. Eng. Pat. 105,965, Mar. 6, 1917. (Appl. No. 3324 of 1917.) Under Int. Conv., Mar. 6, 1916.

IN the manufacture of acetic acid by oxidation of acetaldehyde with air or oxygen by aid of a catalyst, improved yields are obtained by maintaining the reaction chamber at a temperature above the boiling point of acetic acid, preferably between 130° and 400° C. The mixture of air or oxygen and acetaldehyde is heated on its way to the reaction chamber in chambers through which pass the pipes carrying the hot acetic acid vapour from the reaction chamber.—B. V. S.

*Potassium chloride from natural silicates, rocks, minerals, residues or wastes containing potash; Manufacture of—.* E. A. Ashcroft, London. Eng. Pat. 113,211, June 5, 1917. (Appl. No. 8010 of 1917.)

By heating a mixture of felspar and sodium chloride

in suitable proportions to a temperature between 800 and 1200° C., in the absence of air or moisture, the greater part of the potash is converted into chlorite, its place in the felspar being taken by sodium. The product, after cooling, may be extracted with water and the potassium chloride separated from the excess sodium chloride by crystallisation. It is preferable, however, to use a percolation method, the fused chloride passing downwards over the felspar and being drawn off from the bottom of the container. The greater part of the potassium passes off in the first portions, the extraction of the potassium is more complete for the same excess of sodium chloride, and the chloride left in the residue is chiefly the sodium salt, which may be dissolved out and used for the treatment of more felspar without separating the small portion of potassium chloride it contains. In this method the addition of a little magnesium chloride is an advantage as it increases the fluidity of the fixed chloride. The process is applicable to other substances containing insoluble potassium salts and other chlorides than sodium chloride may be used. (See also this J., 1918, 54 A.)—B. V. S.

*Potassium chloride; Process for purifying* —.

A. B. Cox, Cherry Valley, N.Y., Assignor to General Reduction Gas and By-products Co., New York. U.S. Pat. 1,252,784, Jan. 8, 1918. Date of appl., Apr. 17, 1915.

SODIUM monocarbonate, bicarbonate, and sulphate, borax, and other impurities which will form insoluble calcium compounds, are precipitated from the crude potassium chloride solution by the addition of calcium chloride, and the potassium chloride is recovered from the filtrate by fractional crystallisation.—A. B. S.

*Potassium salts [from greensand]; Process for extracting* —. F. Tschirner, Newark, N.J. U.S. Pats. (A) 1,251,450 and (B) 1,251,451, Jan. 22, 1918. Dates of appl., Apr. 18 and May 3, 1916.

(A) THE product obtained by roasting a mixture of greensand (glauconite) and grey, calcareous marl is ground in water, treated with chlorine gas, and dried. The material is then roasted until the mass becomes yellow, and leached with water to extract the potassium chloride produced. (B) A mixture of greensand marl, 100, "lime sand," 50, and calcium chloride, 30 parts, is heated to 800–820° C. to convert the potassium of the greensand into chloride.—W. E. F. P.

*Potash products; Process of recovering high-grade* —.

J. R. MacMillan, Assignor to Niagara Alkali Co., Niagara Falls, N.Y. U.S. Pat. 1,254,521, Jan. 22, 1918. Date of appl., Oct. 9, 1916.

LIQUID containing potassium and sodium as carbonates and other soluble salts is evaporated to about 40° B., and any salts crystallising out are removed. The solution is cooled and any normal sodium carbonate which separates is removed; the solution is then treated with carbon dioxide at 20–30° C., the precipitated sodium bicarbonate filtered off, and the filtrate cooled to or below 0° C. The potassium bicarbonate which separates out is filtered off. The mother liquor is evaporated to 40° B. and the process repeated.—L. A. C.

*Ferrous alkali silicates; Method of treating* — [for the production of potassium chloride].

Method of treating potash-bearing silicates. W. Glaeser, Fullerton, Pa., Assignor to Potash Extraction Corporation, New York. U.S. Pats. (A) 1,254,676 and (B) 1,254,677, Jan. 29, 1918. Date of appl., Mar. 9, 1917.

(A) A MIXTURE of the silicate and calcium chloride, with or without a "porous material," all in finely divided condition, is heated sufficiently to expel

moisture and combined water without oxidising the ferrous iron present, e.g., at a temperature not exceeding 270° C., and then to about 800° C. in a non-oxidising atmosphere. (B) A mixture of the finely divided silicate and calcium chloride in equal proportions, with or without about 5% of a reducing oxide of iron, is dehydrated at about 500° F. (260° C.) and then heated to above 900° C. in a non-oxidising atmosphere.—W. E. F. P.

*Aluminium hydrate [hydroxide]; Method of precipitating* —. R. S. Sherwin, East St. Louis, Ill., Assignor to Aluminium Co. of America, Pittsburgh, Pa. U.S. Pat. 1,251,296, Dec. 25, 1917. Date of appl., July 16, 1913.

PORTIONS of a solution of an alkali aluminate containing precipitated aluminium hydroxide are withdrawn from the bottom of a tall vessel containing the solution and re-introduced at the top in the form of a fine spray through a column of air. Cooling is thereby effected and the precipitate is intimately mixed with the liquid.

—L. A. C.

*Alums; Process for preparing* — from clay or other similar natural compounds of alumina.

L. L. Jackson, Assignor to O. C. Horney, New York. U.S. Pat. 1,254,229, Jan. 22, 1918. Date of appl., Jan. 11, 1917.

UNCALCINED clay and nitre-cake are heated to a temperature between 150° C. and that required to decompose nitre-cake. Silica in an almost entirely insoluble condition is formed and is separated from the soluble products, sodium sulphate and soda alum.—L. A. C.

*Silicates of magnesium; Process for decomposing* —.

L. L. Jackson, Assignor to O. C. Horney, New York. U.S. Pat. 1,254,230, Jan. 22, 1918. Date of appl., Jan. 19, 1917.

SILICATES of magnesium resistant to acid (e.g., soapstone) are heated with a hydrate of an alkali metal (e.g., sodium hydroxide). The alkali silicate formed is separated, the insoluble residue treated with dilute acid, and the magnesium salt recovered by washing.—L. A. C.

*Chlorine and sulphate of sodium; Process of producing* — by catalysis. H. B. Kipper, New York.

U.S. Pat. 1,255,020, Jan. 29, 1918. Date of appl., July 5, 1916.

IN a continuous process for producing chlorine and sodium sulphate from sodium chloride, a mixture of the latter with a catalyst, e.g., ferric oxide, is subjected to the action of sulphur dioxide and oxygen, at about 600° C., in a horizontal, cylindrical, rotary chamber.—W. E. F. P.

*Zirconia; Manufacture of a new compound of zirconium and its application in the production of pure* —.

The Imperial Trust for the Encouragement of Scientific and Industrial Research, London, and R. T. Glazebrook, W. Rosenhain, and E. H. Rodd, Teddington, Eng. Pat. 112,973, Jan. 29, 1917. (Appl. No. 1440 of 1917.)

A MIXTURE of crude zirconia, fluorspar, and concentrated sulphuric acid is heated until the acid begins to fume. The mass is agitated with boiling water to extract the soluble sulphates and the solution is decanted from any insoluble calcium sulphate. Hydrochloric acid of sp. gr. 1.15 is then added and is followed by concentrated ammonia until a white precipitate of basic zirconium sulphate begins to form. The precipitation is allowed to continue without the further addition of alkali. The basic sulphate may also be obtained by dissolving zirconium hydroxide in dilute hydrochloric acid, concentrating the solution until crystals form, re-crystallising these



from hydrochloric acid, then re-dissolving them and adding sulphuric acid or a sulphate. Substantially pure zirconia is made by igniting the basic sulphate. Zirconium hydroxide may be prepared by suspending the basic sulphate in water, adding strong ammonia, and washing until free from sulphate. Zirconium oxychloride is made by dissolving zirconium hydroxide in dilute hydrochloric acid and concentrating until crystals form abundantly.—A. B. S.

*Sulphuric acid; Method of concentrating*—. J. V. Skoglund, New York. Eng. Pat. 113,551, May 14, 1917. (Appl. No. 6841 of 1917.)

SEE U.S. Pat. 1,232,109 of 1917; this J., 1917, 925.

*Sulphur dioxide and nitrogen; Apparatus for the manufacture of a mixture of*—. L. Descamps, Chateauroux, France. U.S. Pat. 1,254,992, Jan. 29, 1918. Date of appl., July 14, 1916.

SEE Eng. Pat. 100,939 of 1916; this J., 1916, 963.

*Titanium compounds*. H. Spence and H. Wrigley, Assignors to P. Spence and Sons, Ltd., Manchester. U.S. Pat. 1,255,807, Feb. 5, 1918. Date of appl., Dec. 11, 1914.

SEE Eng. Pat. 23,089 of 1914; this J., 1915, 1143.

*Construction of brick towers, chambers, acid storage tanks, and the like*. Eng. Pat. 113,015. See I.

*Acid-proof coating compositions*. Eng. Pat. 107,370. See XIII.

*Method of treating polysulphide solutions*. U.S. Pat. 1,254,908. See XIXB.

## VIII.—GLASS; CERAMICS.

*Lime-alkali glasses; Classification of*—according to the Tscheuschner formula. J. Koerner, Sprechsaal, 1915, 48, 333. J. Soc. Glass Techn., 1917, 1, 140—141.

THE Tscheuschner formula for good glasses of the lime-alkali type:  $z = 3(x^2 + 1)$ , where  $x$  and  $z$  represent the molecular proportions of alkalis and silica respectively per 1 mol. of lime, holds good for pure lime-soda glasses, but for lime-potash glasses the formula  $z = 4(x^2 + 1)$  is more satisfactory. When, as is usually the case, the glass contains both soda and potash, a modified formula is necessary, viz.,  $z = \left(3 + \frac{x_k}{x}\right)(x^2 + 1)$  for glasses in which the preponderating alkali is soda, and  $z = \left(4 - \frac{x_{Na}}{x}\right)(x^2 + 1)$ , where potash preponderates; in these formulae  $x_k$  and  $x_{Na}$  represent the molecules of potash and soda respectively. Eighty different lime-alkali glasses were tested by the method suggested by Mylius (viz., treatment with water at 18° C. for seven days, and then with water at 80° C. for 3 hours, and estimating the dissolved alkali volumetrically). It was found that the most resistant glasses all fell within relatively narrow limits of composition, and notwithstanding some slight variations it is concluded that the results confirm from the practical point of view the value of the modified Tscheuschner formula in determining the ratio of alkalis to silica most suitable for the production of a good glass.

"Glass-gall" in soda-lime glasses. H. Knoblauch, Sprechsaal, 1915, 48, 185. J. Soc. Glass Techn., 1917, 1, 146—147.

"GLASS-GALL" is due to the presence of un-

decomposed salt-cake and appears only in glasses made from batches containing this substance. Salt-cake batches usually contain carbon (ground coal, coke, or anthracite), which acts as a reducing agent and aids the decomposition of the salt-cake. Some salt-cake remains undecomposed if insufficient carbon be used, but excess of carbon does not necessarily prevent the appearance of glass-gall. In presence of excess of carbon the gall may appear brownish-red and begin to foam badly, and the whole body of glass may be "seedy" and difficult to fine. It is best to mix the carbon with the salt-cake (after being slightly damped) before its addition to the other batch materials, and it is essential to have a high furnace temperature in melting, as otherwise some of the carbon may be oxidised before the salt-cake melts and so may not exert its proper reducing action. The presence of gall-stones throughout the body of metal generally indicates that the furnace temperature is too low and the metal too viscous to allow the stones to rise to the surface. Gall-stones sometimes appear in the metal after this has been perfectly clear, probably owing to separation of dissolved undecomposed salt-cake when the temperature falls somewhat during "standing off" or as the metal approaches the working end of a tank. Glasses rich in soda dissolve salt-cake more readily than those of high lime or silica content. Gall-stones in cullet cannot be removed by re-melting with an additional quantity of carbon, as this would produce discoloured glass.

## PATENTS.

*Compartment kiln*. R. II. McElroy, Assignor to International Clay Machinery Co., Dayton, Ohio. U.S. Pat. 1,254,421, Jan. 22, 1918. Date of appl., June 20, 1917.

A GAS-FIRED continuous kiln is provided with water-smoking openings and flues with bag walls at each side of each chamber, with burner-openings behind these bag walls and individual air flues for each burner-opening, the air-flues being beneath and communicating with the perforated soles of the chambers.—A B. S.

*Mortar for firebricks*. U.S. Pat. 1,252,785. See IX.

## IX.—BUILDING MATERIALS.

*Direct heat treatment of cement-mill dust to increase its water-soluble potash content*. Merz. See VII.

*Effect of coal ash on the liberation and nature of cement mill potash*. Potter, jun., and Cheesman. See VII.

## PATENTS.

*Cement and lime kilns or the like; Charging apparatus for*—. A. Steiger and W. W. Steiger, Zürich, Switzerland. Eng. Pat. 109,264, Aug. 24, 1917. (Appl. No. 12,187 of 1917.) Under Int. Conv., Aug. 26, 1916.

AN automatic charging device for kilns consists of a vertical pipe fitted with a perforated cone-valve at its lower end, so as to cause the larger lumps in the charge to roll towards the centre of the furnace and the smaller pieces to the circumference, thereby creating a uniform resistance to the passage of gases through the furnace. The cone is adjustable horizontally and/or vertically. An electric contact device effects the stoppage of the supply of material as soon as the latter reaches a predetermined height in the pipe. A suction

pipe for the waste gases is provided in order to facilitate the passage of air through the middle of the material in the furnace.—A. B. S.

*Mortar for firebricks.* A. B. Cox, Cherry Valley, N.Y., Assignor to General Reduction Gas and By-Products Co., New York. U.S. Pat. 1,252,785, Jan. 8, 1918. Date of appl., Apr. 14, 1917.

A MORTAR for firebricks which will vitrify when heated to a comparatively low temperature, is composed of 75 parts of shale, 25 parts of ground firebrick, 30 parts of sodium silicate, and 75 parts of asbestos.—A. B. S.

*Concrete resistant to moisture; Method of rendering* —. A. E. Horn, New York. U.S. Pat. 1,255,116, Jan. 29, 1918. Date of appl., Oct. 20, 1917.

CONCRETE is made waterproof by adding to it a substance (such as a solution of ammonium resinate) which accelerates the formation of colloidal substances in the cement.—A. B. S.

*Wood; Process for preserving* —. W. A. Cecil, Evansville, Ind. U.S. Pat. 1,253,667, Jan. 15, 1918. Date of appl., Sept. 25, 1916.

THE wood is placed in a retort, which is then filled with the preservative liquid, the latter being kept under pressure and in circulation by passing it to and from another (open) vessel. The pressure of the liquid in the retort is gradually increased and is maintained at a maximum until the wood is properly impregnated. The use of an open supply vessel enables free air to be eliminated from the retort.—A. B. S.

*Wood and other porous materials; Process and apparatus for impregnating* — with preservative fluid. H. S. Loud, New York. U.S. Pat. 1,253,814, Jan. 15, 1918. Date of appl., Feb. 9, 1916.

THE pieces of wood or other articles are placed in three retorts connected together by valve-controlled pipes and are impregnated with the preservative fluid in the following cycle: (1) The articles are drenched with the fluid under atmospheric or reduced pressure without being submerged; (2) the air pressure is gradually increased and the drenching continued, still without submergence; and (3) the articles are submerged and the liquid applied under hydraulic pressure. The liquid is withdrawn from one retort at the completion of the cycle and transferred to the second retort to drench the wood therein, the air displaced from the second retort being used to increase the pressure in the second stage of the process in the third retort. The various stages of the cycle occur in each of the retorts in turn.—A. B. S.

*Wood, etc.; Impregnation of* —. M. Rüping, Berlin, Assignor to Hülsberg und Co., Charlottenburg, Germany. U.S. Pat. 1,255,310, Feb. 5, 1918. Date of appl., Dec. 4, 1913. Renewed Dec. 7, 1917.

SEE Fr. Pat. 466,263 of 1913: this J., 1914, 618.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Gold; Application of charcoal to the precipitation of* — from its solution in cyanide. H. R. Edmonds, Bull. 161, Inst. Min. and Met., Feb. 1918. [Advance copy.] 25 pages.

At the Yuanni Gold Mines (Western Australia), zinc dust was used for precipitating gold from cyanide solution. At first a mixture of oxide

and roasted sulphide ore was treated, but subsequently, when the supply of the former had become exhausted and roasted ore only was treated, difficulties arose in precipitating the gold. Investigations into the suitability of charcoal as a precipitant having proved satisfactory, it was substituted for zinc dust in August 1916. The charcoal, freshly burnt, quenched, and washed, is finely ground in a ball mill while wet. Precipitation is effected by filtration through cakes of charcoal powder in Moore-Edmonds vacuum extractors, which were previously employed in zinc dust precipitation. The operation is conducted in at least two, preferably three, stages, each of the three extractors containing 300 lb. of charcoal. When sufficiently charged with gold, the vacuum is released, the cake emulsified, and the pulp passed through a clean-up press, the cake from which is burnt and the ash melted down. The plant, which handles 350—400 tons of solution per day, gives a better recovery at a smaller cost than when zinc-dust precipitation was used.

—W. R. S.

*Copper ores; Some experiments in heap-leaching* —. G. D. Van Arsdale. Eng. and Min. J., 1918, 105, 225—233.

HEAP-LEACHING of copper ores is not at present practised on a commercial scale in the U.S.A., but the low grade ore deposits in the South-West are being tested by this process. At Tyrone, N.M., an old ore dump of 20,000 tons, assaying 2.71% Cu, is being leached with acid ferrous sulphate liquor; the extraction was 20% in 10 months. At Bisbee, Ariz., a large amount of low-grade ore, which had to be removed from the workings, was made into a heap containing 9500 tons, the ore assaying 1.30% Cu. So far, a recovery of 26.9% has been realised in 7 months. From the small preliminary tests, the extraction by leaching is estimated to be 40% for the first year, 20% for the second, and 15% for the third. It is now planned to treat a heap of about 40,000 tons at Tyrone. The ores experimented upon are sulphides.

—W. R. S.

*Spelter; Recovery of* — and treatment of [zinc] retort residues. E. M. Johnson. Met. and Chem. Eng., 1918, 18, 135—139.

THE classification of spelter is controlled by the iron and lead content, which increases very considerably between the first and last draws of the furnace. Care is necessary to secure a representative sample of a large parcel. Where the sample is taken by drilling plates, iron may be introduced from the bit. This is avoided when the sample is taken by granulating a small ladleful from each draw. Retort residues may carry considerable quantities of copper, precious metals, and lead, and may repay examination and treatment. The cheapest method of dressing for concentrating is simple screening. Jigging tests are reported but the results are not very decisive. Magnetic separation effected a high separation of iron. A certain degree of concentration can be effected by burning the carbonaceous matter out of the residue by a simple heap roasting, which is beneficial to subsequent smelting. A reported test of a smelting operation on retort and other smelter residues mixed with a suitable proportion of fresh ores and flux, showed a financial loss under conditions which were rather unfavourable. The author considers the metallurgical results promising for operations on a larger scale.—H. J. H.

*Stibnite; Blast-furnace smelting of* —, with considerations on the metallurgy of antimony. W. R. Schoeller. Bull. 161, Inst. Min. and Met., Feb., 1918. [Advance copy.] 31 pages.

STIBNITE ore containing 50% Sb was smelted in a



circular 36-in. water-jacketed blast furnace having a shaft 10 ft. high. The charge was made up of ore 100, foul slag 156, tap cinder 80, chalk 40, iron 28, and coke 60 lb. The antimony in the slag varied between 0.5 and 1.5%, that in the matte from 2 to 7%, whilst the metal produced assayed Sb 75 and Fe 20%, the balance being chiefly sulphur. The furnace smelted 4.9 tons of ore per 24 hours: the yield of metal was 60%, slag and matte losses 6.5%, the rest being volatilised as dust and fume. The latter, which was not recovered satisfactorily owing to inadequate condensing plant, was of a purple colour and contained 70% of metal. As the antimony was always ferruginous even when the proportion of iron in the charge was reduced much below the theoretical quantity, it was decided to try the effect of smelting without iron. After adjusting the addition of coke to 8–10% of the charge it was found that, instead of a product high in sulphur, practically pure antimony was obtained (e.g., Sb 97.55, Fe 0.42, S 0.41%), whilst no matte was formed, showing that all the sulphur was oxidised and volatilised. The author concludes that the blast is responsible for the conversion of stibnite into metal, by partly oxidising the molten ore to trioxide, which at once reacts with the unaltered sulphide, yielding metal and sulphur dioxide. That this reaction is possible in a gas current has been previously established (this J., 1915, 6; 1914, 169; 1913, 260). The furnace plant and its manipulation are described; the conditions for obtaining the best results are stated to be: less than 10% of coke, low blast pressure, metal content of charge about 10%, height of smelting column 15 ft., continuous discharge and separation of slag and metal in a heated fore-hearth; the condensing system should include a dust chamber, a sufficient number of iron cooling-pipes, a long iron flue, an exhaust fan, and a bag-house or wet-condensing tower. The conclusion is, that the blast furnace is not adapted to high-grade ores but is very suitable for poorer material (25–40% Sb). The French process should be applied to the poorest ores or smelting-residues (less than 25% Sb). Liquefaction is a useful concentrating process for ores containing 45–60% Sb, giving "crude antimony" and residues, which latter are treated by the French or blast furnace process. "Crude antimony" and ores richer than 60% Sb are advantageously treated by "precipitation" smelting in reverberatory furnaces, the matte from which is roasted to recover antimony.—W. R. S.

#### PATENTS.

*Iron-sponge; Production of*—A. Sinding-Larsen, Vestre-Aker, Norway. Eng. Pat. 113,351, Mar. 14, 1917. (Appl. No. 3721 of 1917.)

In the dry reaction process for the production of iron-sponge from iron ore, a horizontal reaction chamber, *a*, is provided with a reciprocating trolley slightly shorter than the chamber, carrying a hearth, *c*. The hearth has side walls projecting

upwards, enclosing a space, *t*, covered by an iron plate, *i*, which carries the ore, and which is spaced at its sides from the side walls. The ore is fed from a hopper, *b*, on to the plate, *i*, when the outlet is not closed by the elevated portion, *f*. Short triangular prisms, *k*, are fixed on the roof of the chamber, with their edges so arranged that the ore is prevented from moving with the plate, *i*, in one direction, but is allowed to move in the other direction. A movement of the ore towards the outlet, *p*, is thus produced. Hydrogen is supplied by the pipes, *h*, to the space, *t*, in which electrical heaters are arranged. The ore is reduced to iron sponge, and the water vapour diffuses downwards and is condensed in the lower part of the chamber, which is cooled by water pipes, *o*. The ascending hydrogen is preheated by the hot vapour. Dry hydrogen is supplied by the pipe, *r*, to complete the reduction of the iron as it passes out by the passage, *p*.—W. F. F.

[Iron] ores: *Process of treating*—A. S. Dwight, New York. U.S. Pat. 1,254,316, Jan. 22, 1918. Date of appl., June 17, 1911. Renewed June 2, 1917.

PULVERULENT iron ore containing lower oxides than ferrite is mixed with a pulverised carbonaceous material, and fed in a thin layer on to a moving conveyor. The carbonaceous material is ignited at one point in its travel and air is passed downwards through the mixture. The hot combustion gases oxidise the ore to an extent equivalent to ferrite, and the oxidised particles cohere so as to form thin cakes. The layer of material on the conveyor is of such thickness that the carbon is burnt out, leaving the oxide in cellular form, and the temperature is kept below that of "silicification" of the metal.—W. F. F.

*Iron and steel surfaces: Rust-proofing*—Process for rust-proofing iron and steel. W. J. Oeschger, Assignor to Parker Rust Proof Co. of America, Detroit, Mich. U.S. Pats. (A) 1,254,263 and (B) 1,254,264, Jan. 22, 1918. Date of appl., Feb. 21, 1917.

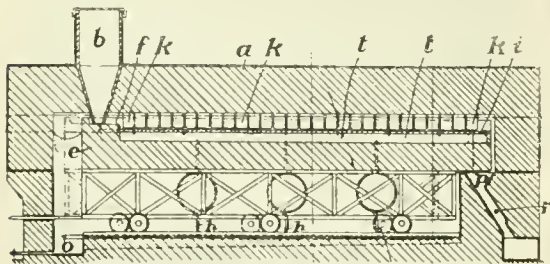
(A) IRON oxide is heated and agitated with phosphoric acid and water until a granular mass is obtained: this is placed in excess of water and heated, and the iron articles immersed therein to form a protective coating. Acid metaphosphates of tungsten, molybdenum, or any of the metals of the third, fourth, and fifth groups, in an excess of water, may be used in a similar manner. (B) The iron or steel is subjected to the fumes from heated acid metaphosphates of iron, tungsten, molybdenum, or any of the metals of the third, fourth, and fifth groups.—T. H. B.

*Coating of [iron or steel] pipes [with zinc]*. E. T. Greenfield, Kiamasha, N.Y. U.S. Pat. 1,254,796, Jan. 29, 1918. Date of appl., Nov. 27, 1915.

AN iron or steel pipe is pickled in dilute sulphuric acid, washed, and a flux containing hydrochloric acid applied to the exterior only. It is then dipped in molten zinc, withdrawn, and the molten metal on the interior of the pipe allowed to flow away. In this way the pipe is coated on the exterior only.—T. H. B.

*Calcining and roasting certain ores; Process and apparatus for*—[by means of exhaust gases from gas engines]. L. E. Tissier, Batna, Algeria. Eng. Pat. 105,915, Apr. 18, 1917. (Appl. No. 5463 of 1917.) Under Int. Conv., Apr. 28, 1916.

EXHAUST gases from engines using poor gas are used for calcining zinc, iron, or manganese carbonates, or gypsum, or for roasting metal sulphides.



A furnace is constructed with two concentric walls of refractory bricks, the intermediate space being packed with wood ashes for heat insulation. The floor of the furnace is a perforated plate surmounting the exhaust chamber of the engine, so that the hot gases pass upward through the material. The material is fed in at the top and withdrawn through side openings. If the heat is insufficient for roasting sulphides or calcareous materials, a small quantity of wood charcoal may be added.—W. F. F.

*Metal [zinc, aluminium]: Method of and apparatus for reducing — to a finely divided state.* Metals Disintegrating Co., Inc., New York, Assignees of E. J. Hall, Passaic, N.J., U.S.A. Eng. Pat. 109,257, Aug. 10, 1917. (Appl. No. 11,522 of 1917.) Under Int. Conv., Aug. 28, 1916.

The solid metal, e.g., zinc or aluminium, is fed into a crucible heated by gas, and provided with an internal conical baffle below the charging opening to minimise the cooling effect of the charge on the molten metal. The molten metal passes downwards through a central vertical pipe to a nozzle, which is surrounded by an annular nozzle to which steam is supplied to atomise the metal. The metal nozzle is constructed of tungsten or other metal having no affinity for the molten metal. The metal is atomised into a larger vertical conduit having a series of compressed-air jets around its periphery to carry away the atomised metal and cool the nozzle. The atomised metal falls into a container which is connected to an exhaust pump.—W. F. F.

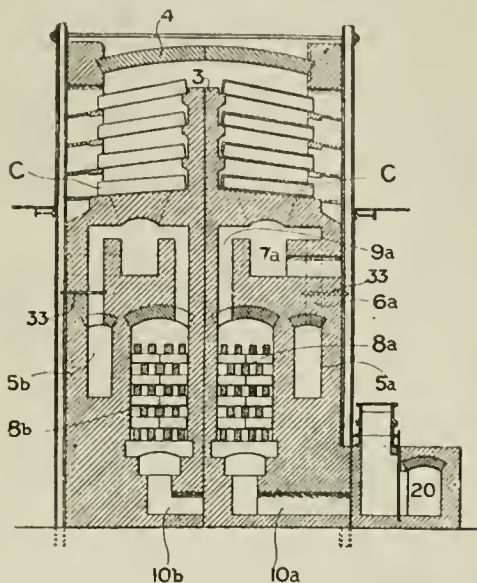
*Zinc: Method of and arrangements for extracting — by the electro-thermal process.* E. S. Berglund, Trollhättan, Sweden. Eng. Pat. 109,435, July 18, 1917. (Appl. No. 10,379 of 1917.) Under Int. Conv., Sept. 5, 1916.

The vapours containing zinc are conducted from two or more electric smelting furnaces to a common condenser, so that one or more furnaces may be put out of action, and the delivery of vapours from these furnaces to the condenser cut off by a suitable arrangement in the gas channel, without disturbing the action of the remaining furnace or furnaces. The vapours, in their passage to the condenser, are passed through a vapour-collecting and heating chamber, in which the temperature, before they enter the condenser, is regulated by electrical heating means to the temperature suitable for condensation to molten zinc.—B. N.

*Furnace, more especially intended for the treatment of zinc.* L. van Gulck, Swansea. Eng. Pat. 113,393, June 23, 1917. (Appl. No. 9053 of 1917.)

Is a reversible gas-heated muffle furnace, particularly applicable for the treatment of zinc, the gas supply in a common conduit may be diverted by a valve into one of the longitudinal passages, 5a, from which it passes through the passage, 6a, to the combustion chamber, 7a. The air supply in a similar common conduit is diverted by a valve into the passage, 10a, from which it passes through the regenerator, 8a, and passage, 9a, to the combustion chamber, 7a. Spaced shelves for the muffles, C, are provided above the combustion chamber so that the hot gases circulate around them. The hot gases then pass over the central wall, 3, over the other set of muffles and through the regenerator, 8b, and passages, 10b, and 20, to the chimney. When the furnace is to be reversed, the air and gas valves are reversed so as to direct the air and gas into passages, 5b and 10b, respectively, and the hot gases then pass through the regenerator, 8a. Sliding shutters, such as 33, are

provided to regulate the supply of gas and/or air, and similar shutters are provided to regulate the opening to the flue passage, 20. A number of double compartments such as described are arranged adjacent to one another in a single



refractory casing; the valve chambers for the air and gas valves are all on one side and the valves are operated simultaneously by interconnected mechanism.—W. F. F.

*Zinc: [Electrolytic] method of smelting —.* I. Partanen, Te luvide, Colo. U.S. Pat. 1,254,531, Jan. 22, 1918. Date of appl., Feb. 2, 1916.

"BLUE POWDER" is treated with chlorine in the presence of moisture, the mixture of zinc chloride and hypochlorite thus produced is fused, and pure zinc is separated from the mixture by electrolysis and withdrawn, whilst the chlorine is again introduced into the furnace into contact with a further supply of "blue powder."—B. N.

*Aluminium: Process for soldering —.* E. H. Moll et Cie., Paris, Assignees of A. H. Alexandre, Levallois-Perret, France. Eng. Pat. 110,758, Oct. 27, 1917. (Appl. No. 15,682 of 1917.) Under Int. Conv., Oct. 27, 1916.

Tin, 55 parts, and zinc, 45 parts, are fused together, 2.5 parts of stearine added, and the mixture stirred and cast into sticks. The softness of the solder may be modified by varying the proportions of tin and zinc. In a modification, the proportions may be tin 75 parts, zinc 45 parts, fine silver 1.65 parts, yellow brass 1.35 parts, and stearine 2.5 parts. The aluminium parts to be soldered are cleaned, heated by a blow-lamp, and then rubbed with a stick of the solder. The tinned surfaces are pressed together, and then strongly heated and allowed to cool.—W. F. F.

*Amalgamators and separators.* E. C. R. Marks, London. From The Diver Mineral Separator Co., Calgary, Canada. Eng. Pat. 113,202, May 7, 1917. (Appl. No. 6484 of 1917.)

In an apparatus for recovering gold, silver, and platinum from sand or other material, the material is fed from a hopper into the upper end of a vertical pipe into which a jet of air, steam, or water is



injected downwards. An air opening is also provided to avoid the formation of a vacuum. The mixture is projected from the bottom of the pipe on to the surface of a quantity of mercury in a cup placed below, and the lighter particles, having a higher velocity, rebound from the surface of the mercury, whilst the heavier particles are retained, the gold and silver being amalgamated, and the platinum sinking to the bottom. To prevent heavy particles of sand from collecting between the mercury and the cup, a ring having outward and inward flanges is fitted into the top of the cup. The lighter particles, also containing metal values, which rebound from the mercury, pass down into a perforated pan, from which they are sifted into another pan. The pans are oscillated slightly at right angles, and a spray of water is directed over the material in them.—W. F. F.

*Metal powder: [Electrolytic] process of making* —. W. C. Moore, Lakewood, Ohio. Assignor to National Carbon Co., Inc. U.S. Pat. 1,254,056, Jan. 22, 1918. Date of appl., July 29, 1915.

METAL powder is prepared by electrolysing a solution of a salt of the metal, so as to form spongy metal on the cathode; the metal sponge is removed and washed in a colloidal solution to free it from electrolyte, whereby a protective coating is precipitated on the metal particles, and protects them from oxidation. The coated particles are then dried *in vacuo*.—B. N.

*Furnace: Electric smelting* —. F. T. Snyder, Oak Park, Ill. U.S. Pat. 1,254,079, Jan. 22, 1918. Date of appl., Mar. 1, 1917.

THE roof of the furnace is carried by a removable frame supported by the base, and can thus easily be removed. Columns of ore and combustible material are fed downwards near the "outer circumference," and the charge is then forced laterally into the melting chamber in contact with the roof, and against the inner edge of a charging port, whereby the latter is sealed against the escape of gas.—B. N.

*Metallurgical [electric] furnace.* W. Buhl, Carnegie, Pa. U.S. Pat. 1,254,308, Jan. 22, 1918. Date of appl., Aug. 20, 1917.

AN electric furnace, with a tilting bowl, has guide-ways arranged to tilt with it. Masts for supporting the electrodes are mounted on the guide-ways to allow horizontal movement relative to the bowl, and the furnace roof is connected with the electrode-supporting masts so that it can be raised from the bowl.—T. H. B.

*Ores: Process for the concentration of* —. J. T. Terry, jun., Mascot, Tenn. U.S. Pat. 1,254,173, Jan. 22, 1918. Date of appl., Mar. 24, 1917.

COMPLEX sulphide ore containing iron sulphides is crushed, and treated with a solution of lime, an ammoniacal solution, and a frothing agent; the iron sulphides become immune to flotation, and rising bubbles carry away the metallic values. —T. H. B.

*[Alloy.] Composition of matter.* F. T. Schuller, Minneapolis, Minn. U.S. Pat. 1,254,854, Jan. 29, 1918. Date of appl., Aug. 6, 1917.

AN alloy of Al 96 to 98%, Sn  $3\frac{1}{2}$  to  $1\frac{11}{16}$ %, and P  $\frac{3}{16}$  to  $\frac{1}{16}$ %.—T. H. B.

*Alloy.* H. S. Cooper. Assignor to The Cooper Research Co., Cleveland, Ohio. U.S. Pat. 1,254,987, Jan. 29, 1918. Date of appl., Oct. 15, 1917.

AN alloy of 99—1% of aluminium and 1—99% but preferably not more than 20%, of beryllium. —W. F. F.

*Steel process.* F. T. Snyder, Oak Park, Ill. U.S. Pat. 1,254,078, Jan. 22, 1918. Date of appl., Jan. 16, 1915.

SEE Eng. Pat. 1213 of 1915; this J., 1916, 123.

*Welding composition.* J. A. Hope, Montreal, Canada. U.S. Pat. 1,254,805, Jan. 29, 1918. Date of appl., Sept. 1, 1916.

SEE Eng. Pat. 102,237 of 1916; this J., 1917, 38.

*Zinc and the like; Process and apparatus for producing* —. F. Tharaldsen, Christiania, Norway. U.S. Pat. 1,255,066, Jan. 29, 1918. Date of appl., Aug. 18, 1916.

SEE Eng. Pat. 108,363 of 1916; this J., 1917, 1014.

*Conduits for electric wires.* U.S. Pat. 1,254,344. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Electric cell; A permanganate* —. A. W. Warrington. Chem. News, 1917, 117, 97—98.

A DILUTE solution of potassium permanganate (3.16 grms. in 250 c.c.), acidulated with 6 c.c. of sulphuric acid, is used as the depolarising agent in a porous vessel containing a carbon plate, and a strong solution of zinc sulphate (14.55 grms. in 750 c.c.) in the outer compartment containing zinc. The voltage obtained with two such two-fluid cells in series was about 4 volts, and after 3 hours, 3.7 volts, whereas two similar one-fluid cells in series gave about 3 volts after a run of 3 hours. (See also J. Chem. Soc., April, 1918.)—B. N.

### PATENTS.

*Electrolytic apparatus.* J. T. King, Toronto, Canada. U.S. Pat. 1,254,045, Jan. 22, 1918. Date of appl., Apr. 19, 1917.

AN electrolytic vessel, which may be rotated on a suitable support, is provided with electrodes supported by two pairs of clamps, one of each pair being connected to slides actuated by springs. The rotating support is connected to a spindle carried in a bearing, the latter being adjustable vertically in a guide. The electrode circuit is provided with a shunt circuit, normally open, which includes contact plates mounted on the guide. A contact piece is carried by the bearing, and is adapted to engage the contact plates and close the shunt circuit when the support is moved a predetermined distance from the electrode clamps.—B. N.

*Furnace: Electric* —. F. T. Snyder, Oak Park, Ill. U.S. Pat. 1,254,077, Jan. 22, 1918. Date of appl., Jan. 19, 1914.

THE electric furnace is mounted centrally on the upper end of an arc-shaped arm, which projects through the "pouring floor," whilst the remainder of the arm is concealed beneath the floor. The pouring spout of the furnace is near the centre of the circle of which the arc is a part, and the arm may be moved along the path of the arc to elevate and tilt the furnace, by means of rotating rollers supporting and gearing with the arm. The furnace is thus tilted without moving the spout appreciably, whilst the position of the arm permits unobstructed access to the spout.—B. N.

*Conduit for electric wires.* D. H. Murphy, Pittsburgh, Pa., Assignor to The American Conduit Manufacturing Co. U.S. Pat. 1,254,341, Jan. 22, 1918. Date of appl., Sept. 19, 1913.

AN iron or steel conduit is provided with an acid-resisting non-corrosive coating on one or more of

its surfaces, which is obtained by electrolytically depositing a thin layer of pure lead, and covering this with a thin layer of pure zinc to fill the minute recesses in the lead, both layers being sufficiently thick to withstand chemical corrosion. A non-corrosive coating is applied over these layers to prevent electrolysis.—B. N.

*Electric-furnace electrodes: Water-jacket for* —. H. J. Kitchen, Luton. U.S. Pat. 1,254,510, Jan. 22, 1918. Date of appl., Mar. 7, 1917.

SEE Eng. Pat. 101,201 of 1916; this J., 1916, 1023.

*Electrolytic apparatus.* G. F. Jaubert, Paris. U.S. Pat. 1,255,096, Jan. 29, 1918. Date of appl., July 26, 1917.

SEE Eng. Pat. 102,974 of 1916; this J., 1917, 602.

*Generation of gases [by electrolysis] for use in internal combustion engines.* Eng. Pat. 113,048. See 11A.

## XII.—FATS; OILS; WAXES.

*Rice polishings; Oil from* —. F. Garelli Annali Chim. Appl., 1917 8, 109—114.

IN commerce rice polishings are mixed with variable proportions of chaff, and used as a fertiliser or feeding stuff. Prior to the war the product was largely exported from Italy, especially to Switzerland and Germany, where it was sold on the basis of the combined fat and proteins. The best qualities contain from 24 to 26%, and inferior qualities from 15 to 16% of fat and proteins. Owing to its porous character the ground material does not keep well, and the contained fat readily becomes rancid. It yields on the average about 6.5% of oil. Oil expressed from freshly separated material had the following characters:—Sp. gr., 0.918; acid value, 13.8; saponification value, 179.4; Hehner value, 94.3; glycerol, 9.03%; and unsaponifiable matter, 0.7%. The fat extracted with petroleum spirit from another sample melted at 25° to 26° C., had acid value 90; and contained 4.95% of glycerol and 3.2% of unsaponifiable matter. The acidity of the oil varied with the kind of rice, the period of production, and the state of preservation. In order to obtain oils low in acidity and rich in glycerol, it is necessary to express the rice polishings as soon as possible after their separation, or, better, to destroy the lipolytic enzymes by heat. The press-cake resembles sesame oil-cake in appearance. A sample contained: moisture, 14.60; nitrogen, 2.73; and fat, 8.68%.—C. A. M.

*Soap solutions: Studies of* —. H. V. Lenher and G. M. Bishop. J. Phys. Chem., 1918, 22, 68—72.

THE emulsification of various oils by the action of soap solution has been examined. Measured quantities of the oil and N/10 sodium oleate solution were mixed with water to give a total volume of 100 c.c. The mixture was heated to 100° C., violently agitated for 5 mins., and then allowed to stand for 5 mins. If oil separated on standing, the experiment was repeated with more sodium oleate solution and less water. By a process of approximation, the volume of soap solution required to bring about complete emulsification was determined. The volumes of soap solution required for the emulsification of 10, 20, 30, 40, 50, and 60 c.c. of oil respectively, under the

specified conditions, were determined in the case of cotton seed, corn (maize), olive, linseed, peanut (arachis), rape seed, castor, sesame, and sperm oils. The results show that the quantity of soap solution increases much less rapidly than the quantity of oil when the volume of this is less than about 40 c.c. For larger quantities of oil, the rate of increase in the required amount of soap solution is much greater.—H. M. D.

*Seeds of Echinocystis oreana [; Oil from* —.] M. R. Daughters. J. Ind. Eng. Chem., 1918, 10, 126—127.

The wild cucumber, *Echinocystis oreana*, is a common plant in waste places from British Columbia to California. Specimens of the seeds collected in three successive years had the following composition—Crude fat, 30.11 to 35.45; proteins, 20.64 to 23.71; starch, 9.20 to 12.05; crude fibre, 20.01 to 22.07; moisture, 3.90 to 4.54; and ash, 2.6 to 2.89%. Oils expressed in the cold and extracted with petroleum spirit had the following characters:—

	Sp. gr. at 25° C.	Refract. index at 25° C.	Solidif. point. °C.	Iodine value.	Saponif. value.
Extracted oil	0.9267	1.4722	+5 to -8	116.5	193.4
Expressed oil	0.9166	1.4701	+5 to -8	117.0	189.1

Freshly-ground seeds yielded an olive-green oil with a taste resembling that of olive oil. On exposure to light the colour faded in a few days to golden yellow. Hydrogenated with a nickel catalyst at 220°—240° C. it yielded a yellowish-white fat which melted at 29°—36° C. and had an iodine value of 76.6. Feeding experiments with mice showed that the original oil and the hydrogenated fat were non-poisonous.—C. A. M.

### PATENTS.

*Hard [hydrogenated] fats; Apparatus for manufacturing* —. K. Kimura, Kobe, Japan. Eng. Pat. 113,232, Aug. 31, 1917. (Appl. No. 12,512 of 1917.)

AN unsaturated fatty oil, a catalyst such as nickel carbonate, and hydrogen gas are agitated in a vertical cylinder enclosed in a steam jacket. A central vertical shaft carries an agitator comprising a framework, star-shaped in horizontal cross-section, each radial portion being covered by metallic netting of nickel, platinum, iron, or silver. The central shaft also carries a supporting spider for the spindles of similar planet agitators which thus revolve around the central agitator. Each planet spindle carries a spur-wheel which engages with an internally toothed ring on the wall of the container. The central agitator causes a centrifugal movement of the liquid outwards, and the rotation of the planet agitators causes an intimate mixing and subdivision. Suitable inlets and outlets are provided for the oil and hydrogen.

—W. F. F.

[*Nickel catalyst suspended in oil.*] *Product of hydrogenation.* C. Ellis, Montclair, N.J. Re-issue 14,429, Jan. 29, 1918, of U.S. Pat. 1,226,620, May 15, 1917. Date of appl., Dec. 18, 1917.

SEE this J., 1917, 723.

*Soap-like detergent and manufacture thereof.* W. Feldenheimer, London. U.S. Pat. 1,254,588, Jan. 22, 1918. Date of appl., Apr. 12, 1917.

SEE Eng. Pat. 106,423 of 1916; this J., 1917, 724.



### XIII.—PAINTS ; PIGMENTS ; VARNISHES ; RESINS.

#### PATENTS.

*Coating compositions : Acid-proof* —. S. Tamari, Tokyo, Japan. Eng. Pat. 107,370, Apr. 13, 1917. (Appl. No. 5250 of 1917.) Under Int. Conv., June 20, 1916.

AN acid-proof composition is obtained by adding gradually, with thorough stirring, about 3 parts of graphite to 1 part of melted sulphur, raising the temperature of the mixture to about 170° C., and cooling. The composition is melted before use.—B. V. S.

*Drying-oils [from kerosene] ; Process for the manufacture of* —. F. C. Thiele, Assignor to Cudahy Refining Co., Coffeyville, Kans. U.S. Pat. 1,254,866, Jan. 29, 1918. Date of appl., Aug. 6, 1913.

KEROSENE containing cyclo-paraffins is treated with a halogen (chlorine), acidulated, and the mixture treated with a metal.—L. A. C.

*[Stencil emulsions and] paper stencils and their manufacture*. E. C. R. Marks, London. From Addressograph Co., Chicago, U.S.A. Eng. Pat. 113,338, Feb. 28, 1917. (Appl. No. 2985 of 1917.)

SEE U.S. Pat. 1,231,551 of 1917 ; this J., 1917, 930. Stencil sheets are produced by treating a fibrous porous sheet, for example, of Yoshino paper, with the emulsion.

### XIV.—INDIA-RUBBER ; GUTTA-PERCHA.

*Rubber ; Vulcanisation of* — by selenium. C. R. Boggs. J. Ind. Eng. Chem., 1918, 10, 117—118.

A STANDARD 30% Para rubber compound was partially vulcanised when heated for 2 hours at 150 C. with black selenium in the equivalent proportion that sulphur would be used. The physical tests showed normal elongation, but only about 50% of the tensile strength of the analogous sulphur compound. All the samples showed but little deterioration on keeping, and after 4 years had the same elongation and about 70% of the original tensile strength. Doubling the proportion of selenium and using an organic accelerator did not give a better product, but caused the samples to become brittle on keeping, as in the case of over-vulcanised or under-vulcanised rubber. On removal from the press this product expanded 25% of its volume, which, however, became normal again when cold. This pointed to deficient vulcanisation. By using amorphous selenium and an organic accelerator some increase of tensile strength without loss of elongation was obtained. The best product obtained showed no appreciable deterioration when put on wire and left for 3 years. Accelerators were found which enabled rubber to be vulcanised by heating it with selenium at the ordinary vulcanising temperature (135° C.) for only about twice the time required with sulphur. The product showed the normal tensile strength (1100 to 1200 lb.) and elongation (2 to 10 or 12 in.) of a similar compound containing sulphur. It was somewhat soft, but did not deteriorate when heated for 4 days in the air at 70° C. It had rather low insulation resistance and dielectric strength. Chemical analysis as used with ordinary vulcanised compounds is not applicable to the selenium compounds, since black selenium is practically insoluble in acetone, and is only sparingly soluble in chloroform and carbon bisulphide. A compound to which 31.7% of rubber

had been added showed that amount of rubber when analysed by the tetrabromide method. Hence it would appear that no correction need be made for combined selenium or that the selenium is readily displaced by bromine. Rubber goods vulcanised with selenium appear to deteriorate much less with age than do sulphur compounds, so that where permanency is required it may be found advantageous to replace sulphur by selenium.—C. A. M.

### XV.—LEATHER ; BONE ; HORN ; GLUE.

#### PATENT.

*Tanning ; Compound suitable for* —. O. Schmidt and A. Andres, Assignors to Badische Anilin und Soda Fabr., Ludwigshafen, Germany. U.S. Pat. 1,254,364, Jan. 22, 1918. Date of appl., Mar. 23, 1914.

SEE Addition of July 31, 1913, to Fr. Pat. 443,730 of 1912 ; this J., 1914, 209.

### XVI.—SOILS ; FERTILISERS.

*Soils ; Correlation between bacterial activity and lime requirement of* —. F. E. Bear. Soil Sci., 1917, 4, 433—462.

THE author has studied the relation between the activities of soil bacteria concerned in nitrogen fixation or transformation and the lime requirement of certain soils, having lime requirements varying from 0 to 4600 lb. of calcium carbonate per 2,000,000 lb. of soil. Ammonification proceeded fairly satisfactorily in most of the soils without the application of lime, but the use of moderate amounts of calcium carbonate increased the rate of ammonification in most cases. The rate of nitrification was almost directly correlated with the amount of calcium carbonate supplied, soils having high lime requirements showing practically no nitrification until calcium carbonate was applied. Nitrogen fixation by non-symbiotic soil organisms was considerably increased by the addition of calcium carbonate, and the application of mono-calcium phosphate was also necessary for maximum nitrogen fixation. A good growth of soya beans was obtained on a soil having a lime requirement of 3000 lb., and well fertilised with acid phosphate (superphosphate) or manure. Nitrogen fixation accompanying the growth of soya beans took place readily in acid soils, but was increased by small applications of calcium carbonate, though decreased by large applications. The results, as a whole, indicate that small applications of calcium carbonate are, as a rule, relatively more effective than large applications as a means of increasing the bacterial activities in acid soils.—W. G.

*Soils ; Moisture equivalent determinations of salt-treated — and their relation to changes in the interior surfaces*. L. T. Sharp and D. D. Waynick. Soil Sci., 1917, 4, 463—469.

DUPLICATE 100-grm. portions of a clay loam soil were treated with 80 c.c. of solutions of different sodium salts. From one of these portions the salt was washed with distilled water before centrifuging, whilst a sample from the other portion was subjected to centrifuging without washing. It was found that there was a marked increase in the moisture equivalent (i.e., the water retained after centrifuging) after the treatment with saline solutions and washing, the extent of the increase

depending on the salt used. The greatest effect was obtained with sodium sulphate, followed in order by sodium hydroxide, sodium carbonate, and sodium chloride. This effect was not obtained when the washing with water was omitted. The authors consider that the salt and water treatment increased the interior surface of the soil by from 2 to 40 %, depending on the salt used.—W. G.

*Soils; Relation of the mechanical analysis to the moisture equivalent of ——. A. Smith. Soil Sci., 1917, 4, 471—476.*

MOISTURE equivalents (see preceding abstract) have been determined for the seven fractions obtained by the mechanical analysis of twelve different soils, and the values obtained were used to calculate the moisture equivalent of a number of soils from the results of their mechanical analyses. The calculated results were, on the whole, only approximations to the results obtained by actual determinations.—W. G.

*Soils; Volumetric determination of sulphates in water extracts of ——. A. W. Christie and J. C. Martin. Soil Sci., 1917, 4, 477—479.*

THE authors advocate the method of Raiziss and Dubin as used for the estimation of total sulphur in urine (see J. Biol. Chem., 1914, 18, 297), the sulphates being precipitated as benzidine sulphate and the precipitate titrated with *N*/20 potassium permanganate. Not less than 15 c.c. and not more than 20 c.c. of water should be used to wash the benzidine sulphate precipitate. The average error by this method for the small amounts of water-soluble sulphates was found to be 3 % of the total.—W. G.

*Nitrogen-assimilating bacteria [of soils]; Influence of nitrates on ——. T. L. Hills. J. Agric. Res., 1918, 12, 183—230.*

SMALL quantities (up to 0.1 %) of potassium, sodium, or calcium nitrate caused a great increase in the number of *Azotobacter* in sterilised soil, but ammonium nitrate was less effective. Higher concentrations did not favour the growth of the organism. The presence of these nitrates caused an increase in the total nitrogen in the soil, but this increase was not commensurate with the increase in the numbers of *Azotobacter*. Slightly different results were obtained on agar films, calcium nitrate in this case causing a decrease in the amount of nitrogen fixed. In the presence of these nitrates marked pigmentation occurred and the volutin bodies in the *Azotobacter* cells increased in number and size. The number of *B. radicola* in sterilised soil was increased by the addition of small quantities of the three nitrates, the increase being less marked than in the case of *Azotobacter*, although the *B. radicola* appeared to be more resistant to higher concentrations of nitrates than *Azotobacter*. Unlike the *Azotobacter*, *B. radicola* does not reduce nitrate to nitrite under aerobic conditions. The presence of large amounts of the three nitrates proved detrimental to the formation of nodules on alfalfa. Nitrates in soil cultures prevented the re-formation of nodules once removed and also caused a decrease in the number of nodules already present.—W. G.

*Root-nematode (Heterodera radicola); Study of the — and its control. W. P. Duruz. Soil Sci., 1917, 4, 481—492.*

NEMATODE activity was found to be diminished by excess of moisture, by high temperature (38.8° C.), and by treatment with formaldehyde, sphagnum moss extract, or sodium cyanide dissolved in water. The last named may be used on green-

house soils at the rate of 200 lb. per acre, dissolved in water so as to give  $\frac{1}{2}$  gallon per sq. foot, the treatment being repeated at the end of one week.—W. G.

#### PATENTS.

*Peat and muck; Process of treating — for the preparation of fertiliser. J. P. Schroeder, Washington, D.C. U.S. Pats. (A) 1,254,365 and (B) 1,254,366, Jan. 22, 1918. Dates of appl., Aug. 13 and 22, 1917. (Dedicated to the public.)*

(A) RAW peat is digested with sulphuric acid, the complex, insoluble proteins of the peat being thereby hydrolysed giving simple compounds, soluble in water and available for plant use. The excess acid is then neutralised by the addition of finely ground rock phosphate. The material is thus obtained in a physical condition suitable for application to the soil. (B) Peat is made slightly alkaline with ammonia and digested under steam pressure, the insoluble proteins being hydrolysed into simple compounds soluble in water. With this material there may then be intimately mixed a small portion of ammonium sulphate and calcium carbonate, which slowly interact and generate ammonia throughout the mass, thus neutralising any tendency to revert to an acid reaction.—W. G.

### XVII.—SUGARS; STARCHES; GUMS.

*Sucrose; Influence of different compounds on the destruction of monosaccharides by sodium hydroxide and on the inversion of — by hydrochloric acid. III. Constitutional formulæ of the hydroxybenzoic acids and of sulphanilic acid. H. I. Waterman. Proc. K. Akad. Wetensch. Amsterdam, 1918, 20, 581—589.*

SALICYLIC acid behaves as a monobasic acid in retarding the destruction of dextrose by sodium hydroxide, whereas *m*- and *p*-hydroxybenzoic acids behave as dibasic acids. Sulphanilic and hippuric acids behave as monobasic acids in this reaction, but exert no influence on the inversion of sucrose by hydrochloric acid. (For the constitutional formulæ see J. Chem. Soc., April, 1918.)—W. G.

#### PATENTS.

*Size and process of making the same [from starch]. A. W. H. Lenders, Cedar Rapids, Iowa. U.S. Pat. 1,253,397, Jan. 15, 1918. Date of appl., July 8, 1916.*

SIZE containing a mixture of thick-boiling with thin-boiling starch or of modified starch with a lubricant, may be prepared by modifying starch with acid to produce a thin-boiling product, which, after addition of raw starch if necessary, is again modified by alkaline treatment.—J. H. L.

*Production of decolorising charcoal. Eng. Pat. 106,080. See IIB.*

### XVIII.—FERMENTATION INDUSTRIES.

*Wines; White "casé" of —. L. Moreau and B. Vinet. Bull. Soc. Agric. France, 1917, 267—271, 292—299. Bull. Agric. Intell., 1917, 8, 1280—1281.*

FROM a study of numerous wines affected by white "casé" the authors conclude that the malady is brought about by the presence of iron and phosphoric acid (cp. Laborde, this J., 1917, 468). Free tartaric acid favours the development of the



disease, de-acidified wines being nearly always immune. Acid potassium tartrate, and malic and succinic acids have far less influence than free tartaric acid. The malady may be prevented in all cases by addition of 1 grm., and in some cases by addition of 0.5 grm., of citric acid per litre, but even the larger amount does not always dissolve the precipitate when it has once formed, though it promotes agglutination and deposition of the suspended ferric phosphate. In a wine affected with white "casé" the tannin present perceptibly aggravates the disease and produces a bluish colour. Such cases, however, should be distinguished from blue "casé," in which the precipitate contains no phosphoric acid or only traces. Aeration tends to promote the development of white "casé," by oxidising ferrous to ferric phosphate, which then separates from solution. The disease can be prevented or cured in two ways, viz., by treating wines of average or less than average acidity with citric acid, and by treating "green" wines with de-acidifying agents. As a preventive measure it is very desirable to practice the de-acidification of musts in years when these have a high acidity.—J. H. L.

*Use of micro-organisms to determine the preservative value of spices.* Bachmann. See XIXA.

*Alcohol from calcium carbide.* Beyer. See XX.

#### PATENTS.

*Fermenting tun; Method of and apparatus for preventing overflow of surface bubbles in a —.* R. G. Abercrombie. Alcoa. Eng. Pat. 113,002, Feb. 9, 1917. (Appl. No. 1976 of 1917.)

*HORIZONTAL, fan-shaped films of fluid, e.g., air, are ejected across the surface of the fermenting liquid, from a number of nozzles, e.g., four, fixed around the top of the tun and in communication with a supply of the compressed fluid.*—J. H. L.

*Heating and boiling liquids [e.g., wort]; Means for —.* P. Robinson. Burton-on-Trent. Eng. Pat. 113,346, Mar. 7, 1917. (Appl. No. 3348 of 1917.)

AN apparatus for fixing in a copper for boiling wort consists of a central hollow hub divided into two compartments one above the other, and having an inlet for the supply of live steam to the lower one and an outlet for condensed water from the upper compartment. Radial tubes project from the central hub and each is divided by a horizontal diaphragm into a lower live steam compartment and an upper condensed water compartment. Upright open tubes are fixed in holes in the diaphragm and open into the live steam compartment, and are surrounded by wider tubes with closed tops which open below into the condensed water compartment.—J. H. P.

*Ferments and process of making the same.* A. D. Barr, McHue, Ark. U.S. Pat. 1,253,334, Jan. 15, 1918. Date of appl., June 3, 1916.

CLAIM is made to a ferment (enzyme) containing an amino-acid, possessing proteolytic power, and capable of decomposing carbohydrates in acid, neutral, or alkaline media with production of acid substances, also to products comprising an enzyme (or a mixture of proteolytic enzymes) together with leucine, e.g., a mixture of equal amounts of pepsin and pancreatin with 5% of leucine. Products capable of decomposing carbohydrates are prepared by triturating a mixture of proteolytic enzymes, adding a small quantity of a substance containing the amino-acid group, and again triturating the mixture.—J. H. L.

*Amino compounds [for foodstuffs, etc.]; Preparation of — by fermentation.* G. Eberle, Stuttgart, Germany. U.S. Pat. 1,254,033, Jan. 22, 1918. Date of appl., Feb. 15, 1915.

SEE Fr. Pat. 476,693 of 1914; this J., 1916, 631.

#### XIXA—FOODS.

*Dialysis; Gliding —. II. [Preparation of fruit syrups.]* H. Thoms. Ber., 1918. 51, 42—45.

DIALYSIS can be greatly accelerated if the liquid is made to glide over the membrane. For this purpose, an apparatus may be constructed of two vessels, clamped together, with a parchment membrane between them, water being placed on one side and the liquid on the other. The apparatus is rotated or shaken in the same plane as the membrane. Such a device has been designed by the author for the preparation of stable fruit syrups, without the destruction of the enzymes, vitamins, or aroma, merely by removing the excess of acids and sugar. (See also J. Chem. Soc., 1917, ii., 561; and April, 1918.)—J. C. W.

*Spices; Use of micro-organisms to determine the preservative value of —.* F. M. Bachmann. J. Ind. Eng. Chem., 1918, 10, 121—123.

BACTERIA, yeasts, and mould-fungi were grown on nutrient media containing definite amounts of different brands of cloves, cinnamon, and all-spice. It was found that there was a considerable difference in the preservative effect of different brands of the same spice, and that the method could be used as a criterion of the preservative value. The results showed that cloves may be as effective a preservative as cinnamon.—C. A. M.

*Haystacks; Spontaneous ignition of —.* E. Jordi. Schweiz. Chem.-Zeit., 1918, 2, 7—10.

SINCE about 1913, haystack fires have been much more frequent than before in Switzerland, probably because modern mechanical appliances enable hay-making and stacking to be completed much more rapidly than was formerly the case and in consequence the hay often has less opportunity to dry. The author refers to the work of various investigators, viz., Miebe, Ranke, Schenk, and Boekhout and de Fries, on the heating of stacks, and concludes therefrom that a rise in temperature to 40° C. may occur owing to respiration of the surviving cells and also to intramolecular respiration; the activities of micro-organisms account for further heating to about 70° C., at which temperature a slow process of dry distillation may set in and eventually heat the stack to 300°—400° C. The final products of this dry distillation are combustible gases and carbon, the latter being pyrophoric owing to the fibrous nature of the original material. Various practical precautions are enumerated; e.g., care should be taken that hay is dry when stacked; stacks should be of as large cross-section as possible, and provided with means for ventilation. If the temperature in the stack should rise to 70° C. the ventilation passages should be closed. Salt may be scattered over hay which has become damaged by exposure to bad weather.—J. H. L.

*Haystacks; New theory of the spontaneous firing of —.* A. Tschirch. Schweiz. Chem.-Zeit., 1918, 2, 10—12.

THE author criticises the commonly accepted theory of spontaneous combustion in stacks, especially the assumption that combustible gas and carbon are produced at relatively low temperatures by "dry distillation." He proposes a

theory based on observations made on the drying of medicinal plants. The first phase of heating is regarded as due to oxidation processes under the influence of the plant oxidases and at the expense of the air present in the stack. This phase results in a small rise of temperature but ceases when the oxygen in the stack has been exhausted. After this the chief rôle is played by reductases, which act energetically between 50° and 70° C. and effect the de-oxidation and eventual carbonisation of such substances as amino-acids and carbohydrates. It is to the accumulation of oxygen withdrawn by the reductases that the danger of ignition is due, for the reduction processes themselves would not produce sufficient heat to ignite the material. The general precautionary measures recommended are, therefore, thorough drying of the hay before stacking (to minimise enzymic action), and thorough aeration of the stacks, which may be built in horizontal layers with air spaces between.

—J. H. L.

*Seeds of Echinocystis oregana.* Daughters. See XII.

#### PATENTS.

*Milk ; Apparatus for evaporating*——. J. H. Mason, Prince Rupert, B.C., Canada. U.S. Pat. 1,254,423, Jan. 22, 1918. Date of appl., Aug. 2, 1916.

THE apparatus comprises a drying chamber and an evaporating reservoir, with a pipe for conveying air from the drying chamber to the evaporating reservoir and a return pipe. The drying chamber contains a refrigerating brine coil for dehydrating the air by freezing the moisture in it. Means are provided for spraying water on to the coil, and for passing heat through it when the drying chamber is disconnected from the evaporating reservoir.—J. H. J.

*Garbage ; Process of converting——into food.* W. H. Faust, Assignor to M. E. Faust, Los Angeles, Cal. U.S. Pat. 1,254,317, Jan. 22, 1918. Date of appl., May 16, 1917.

GARBAGE is cooked and dried for a long period at about 212° F. (100° C.), and then heated to about 400° F. (205° C.) for a short time to destroy micro-organisms. The material is then ground into granular form.—W. F. F.

*Bread and the like ; Process for predetermining the time necessary for the fermentation or rising of dough for*——. A. Watkins, Hereford. U.S. Pat. 1,254,457, Jan. 22, 1918. Date of appl., Aug. 24, 1915.

SEE Eng. Pat. 19,487 of 1914 ; this J., 1915, 243.

*Coffee extract ; Plant for the manufacture of*——. L. Etaix, Paris. U.S. Pat. 1,254,129, Jan. 22, 1918. Date of appl., Aug. 31, 1916.

SEE Eng. Pat. 2722 of 1915 ; this J., 1915, 1268.

### XIXB.—WATER PURIFICATION ; SANITATION.

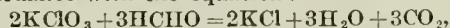
*Cresols ; Process for the deodorisation of the*——. J. F. Couch. Amer. J. Pharm., 1918, 98, 128—130.

EQUAL weights of cresol and concentrated sulphuric acid are thoroughly mixed and heated to 75°—

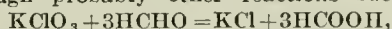
110° C. until a drop of the liquid mixed with water gives only a faint cloudiness. The separation of oily drops shows that the reaction is incomplete unless the cresol contains a large proportion of hydrocarbons. The length of time necessary for the reaction varies between 6 and 14 hours. On completion of the reaction, the product is poured into five volumes of cold water, any oil which separates out being removed. The mixture is neutralised with milk of lime and, after standing for 24 hours, the clear liquid is decanted off. The sediment is washed and the washings added to the decanted liquid. The solution is then evaporated to dryness on the water-bath, the residue dissolved in a little water, and if the odour still persists, the solution must be again evaporated to dryness. When the solution in water is quite odourless, it is diluted with water to twice the volume of the cresol taken, and the calcium precipitated with sulphuric acid, carefully avoiding any excess. After standing for a day, the solution is filtered and the sediment washed ; the filtrate may then be made up to any desired strength. Tests with *B. coli* show that the phenol coefficient of the solution is considerably increased by this treatment, and since no addition of soap is necessary in the solution, it will not become cloudy when mixed with hard waters. It may be used in all cases in place of the *Liquor cresolis compositus* and in the same proportions.—L. A. C.

*Formaldehyde ; A substitute for the permanganate-formalin method of disinfection with*——. C. G. Storm. J. Ind. Eng. Chem., 1918, 10, 123—124.

IF formalin be poured on to crystals of potassium or sodium chlorate there is no reaction until the mixture is heated to about 65° C. The reaction which then occurs is analogous to that which takes place between formaldehyde and potassium permanganate in the cold, and appears to be in accordance with the equation :



although probably other reactions such as :



also occur. The best results for disinfection are obtained by the use of 25 grms. of chlorate to 100 c.c. of formalin ; with these proportions about 23% of the formaldehyde is oxidised, and the remainder volatilised.—C. A. M.

*Quinine derivatives ; Disinfectant action of*——on *diphtheria bacilli*. H. Schaeffer. Biochem. Zeits., 1917, 83, 269—314.

THE antiseptic and disinfectant properties of the alkyl derivatives of hydrocupreine increase, generally, with rise in molecular weight of the alkyl group up to the octyl derivatives, after which they diminish. The antiseptic and disinfecting actions are not always absolutely parallel. (See also J. Chem. Soc., March, 1918.)—S. B. S.

#### PATENTS.

*Water or sewage or other liquids ; Apparatus for and processes relating to purifying or treating*——. C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,105, Nov. 14, 1916. (Appl. No. 16,281 of 1916.)

THE process is specially devised for the treatment of water with chlorine, a supply of which is obtained from two cylinders of the liquefied gas connected to the apparatus with flexible tubing. The water to be treated passes through a Venturi meter, from each side of which a pipe passes to a differential pressure-regulating apparatus contain-



ing either two or three diaphragm chambers. The high and low pressures respectively of the Venturi meter are transmitted by the pipes to each side of the central diaphragm. The chlorine is supplied to a drop pressure control valve (see following abstract), and the high and low pressure gas is supplied to the two outer diaphragms. The apparatus is set so that the pressure difference of the gas supply maintains the required proportion to the pressure difference of the water supply. When this is disturbed, the diaphragms react upon the stem of a needle-valve on the main gas supply pipe, the action of which permits more or less chlorine to pass to the control valve. The chlorine after leaving the control valve passes to a flow indicator and meter of the type described in Eng. Pat. 113,197 (following). From this, the gas passes through a back-pressure valve of the type described in Eng. Pat. 113,198 (following) to a diffuser in the water main. Various modifications of the apparatus are described.—J. H. J.

*Liquids; Treatment of— with gases.* C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,106, Nov. 14, 1916. (Appl. No. 16,282 of 1916.)

THE process refers more especially to the treatment of water with chlorine in a regulated, constant amount. The gas is supplied from compressed gas cylinders and is passed through a differential pressure-reducing valve which is a simplified form of the similar valve described in the preceding abstract. The exit pipe from the valve leads to a control valve which consists of a needle-valve under which the gas has to pass before reaching the exit tube. The stem of the needle is screw-threaded and passes through a gland nut, and engages with a sleeve screwed externally and internally with threads of slightly different pitch. The head of the sleeve is adapted to be turned by a key, a comparatively large movement being required to effect a fine adjustment of the needle-valve. The exit pipe carries a by-pass to the diaphragm of the preceding valve, the action of which regulates the pressure of the chlorine supply to the control valve, so as to maintain the drop in pressure across this valve constant. From the control valve the gas passes through a meter into an absorption apparatus, in which a solution is made for delivery to the bulk of water to be treated.—J. H. J.

*Gases; Devices for indicating and measuring the flow of—* C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,197, Nov. 14, 1916. (Appl. No. 6173 of 1917.)

THE apparatus consists of a pipe connected to the gas supply to be measured, the free end of the pipe being covered with a transparent cap with a constricted orifice. Over this cap is another larger, transparent cap, through which the gas passes to the exit pipe. Narrow tubes pass from the inlet and exit pipes to the two limbs of a manometer, of the pattern in which one limb is placed inside the other. The inlet pipe is connected to the wider limb and the exit pipe to the narrow limb. The latter limb carries an enlarged cup at its upper end to act as a safety reservoir for the indicating liquid, should it be forced up the narrow limb by a sudden increase of pressure. A scale is placed behind the two limbs of the manometer, indicating the pressure difference in the two pipes corresponding to the difference in level of the liquid in the two limbs, or the corresponding rates of flow. In lieu of the cap with the constricted orifice, a cap with a porous diaphragm may be used. The apparatus is specially suited for measuring the flow of chlorine when used for the purification of water.—J. H. J.

*Valves employed in systems supplying gases such as chlorine to liquids such as water.* C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,198, Nov. 14, 1916. (Appl. No. 6174 of 1917.)

THE apparatus consists of a casing and a cover, a diaphragm being clamped between the two. Attached to the lower side of the diaphragm is a needle-valve operating in a seating which communicates with the exit pipe. Above the diaphragm is a spring, regulated by a set screw in the cover of the apparatus. The space below the diaphragm is divided into two parts by a cup surrounding the needle-valve. The gas to be used is admitted to the space below the cup and passes through a strainer into the cup by means of holes around the rim of the cup. When the pressure of the gas in the cup is greater than that of the spring above the diaphragm, the needle-valve is opened and the gas passes into the exit pipe. The cup serves to retain any liquid flowing back through the valve. In lieu of the spring above the diaphragm, a volume of air confined at a definite pressure may be used. This has the advantage that the pressures on each side of the diaphragm are affected equally by any rise of temperature.—J. H. J.

*Purifying water; Apparatus for and process of treating liquids with gases or —* C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,288, Nov. 14, 1916. (Appl. No. 16,283 of 1916.)

THE process is particularly applicable to the preparation of a chlorine solution for use in the purification of water. The supply of chlorine is obtained from a cylinder of the liquefied gas, and is delivered to a differential pressure reducing valve of the type described in Eng. Pat. 113,106 (preceding). The gas at the reduced pressure passes through a back pressure valve of the type described in Eng. Pat. 113,198 (preceding) into an absorption vessel. This is made of glass with an air-tight cover, and in the centre a bell-jar is held in place, containing an inverted siphon with its exit pipe opening through the shoulder of the bell-jar. The absorption vessel is kept nearly full of water, which enters under pressure through a constricted opening in the cover. The exit pipe from the back pressure valve passes through the cover and opens into the top of the bell-jar, and the entering chlorine fills the bell-jar until the siphon comes into action, discharging the gas into the water in the outer part of the vessel. The exit pipe for the solution is placed vertically in the vessel, with its upper end slightly above the level of the top of the bell-jar. The absorption vessel being of glass, the discharges from the bell-jar can be counted.—J. H. J.

*Diffusers for use in diffusing gases in liquids.* [Purification of water.] C. F. Wallace, Richmond, N.Y., and M. F. Tiernan, New Rochelle, N.Y., U.S.A. Eng. Pat. 113,374, Nov. 14, 1916. (Appl. No. 6175 of 1917.)

THE diffuser consists of a shallow closed dish of porcelain, the top plate being made of alundum or other porous material. The bottom of the dish has a small disc let into it, made of material of finer porosity than the top plate. The diffuser is specially designed for use in flowing water in which chlorine is to be dissolved, as described in Eng. Pat. 113,105 (preceding). The diffuser is placed below the water level and the chlorine enters under pressure by a silver pipe passing through the top plate, and escapes through the pores of the material of the top plate. Any water collecting inside the diffuser is expelled through the porous disc in the bottom.—J. H. J.



*Sewage and other liquids : Purification of* —. W. Jones, Stourbridge, Worcestershire, and Jones and Attwood, Ltd., Amblecote, Staffs. Eng. Pat. 113,333, Feb. 26, 1917. (Appl. No. 2761 of 1917.)

AN improved form of tank for use in the activated sludge process consists of a long channel arranged as the outer coils of a spiral, with air diffusers arranged along its length, either in the middle of the channel or at one side or both sides. The floor is made to slope towards the air diffusers. The channel may be restricted in width at each end or at the position of the air diffusers by baffles projecting from the side walls. The channel terminates at the centre in a round tank with a conical bottom, not furnished with air diffusers, in which the sludge settles at the bottom and is returned by an air lift or other means to the crude sewage inlet of the channel. Another form of the channel consists in a to and fro rectangular arrangement with the deposit tank at one end. —J. H. J.

*Activated [sewage] sludge treatment.* W. C. Moor, Fort Worth, Tex., Assignor to Armour and Co., Chicago, Ill. U.S. Pat. 1,254,833, Jan. 29, 1918. Date of appl., July 9, 1917.

ACTIVATED sludge is dehydrated by heating above 30° C. and is then coagulated by addition of sulphuric acid. The gas generated on coagulation causes the coagulum to float; it is separated and dried.—J. H. J.

*Activated [sewage] sludge treatment.* G. B. Mulloy, Assignor to Armour and Co., Chicago, Ill. U.S. Pat. 1,254,836, Jan. 29, 1918. Date of appl., July 9, 1917.

IN treating industrial sewage by the activated sludge process, the sewage is submitted to a preliminary dilution with the effluent from previously treated sewage.—J. H. J.

*Activated [sewage] sludge treatment.* G. L. Noble, Assignor to Armour and Co., Chicago, Ill. U.S. Pat. 1,254,841, Jan. 29, 1918. Date of appl., July 9, 1917.

IN the activated sludge process of sewage treatment, the raw sewage is inoculated with activated sludge from previously treated sewage, which sludge has been withdrawn from the sewage and aerated for about three hours.—J. H. J.

*Polysulphide solutions [insecticides]; Method of treating* —. E. C. Holton, Olmsted Falls, Assignor to The Sherwin-Williams Co., Cleveland, Ohio. U.S. Pat. 1,254,908, Jan. 29, 1918. Date of appl., Dec. 1, 1915.

THE polysulphide solution is prepared of 35°–40° B. strength and 1%–5% of its weight of cane sugar or of an alkaline-earth saccharate is added to it. The solution is then evaporated to dryness and the residue is pulverised. The product contains lime-sulphur and sugar in the ratio of 8 : 1, and can be used as an insecticide.—J. H. J.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Alcohol from calcium carbide; Ethyl* —. O. Beyer, Schweiz. Chem.-Zeit., 1918, 2, 12–13.

It is well known that acetylene can be converted into acetaldehyde by the use of certain catalysts, and the aldehyde can be reduced to alcohol by

hydrogen in presence of finely divided nickel, platinum, or palladium. In Switzerland the production of alcohol from calcium carbide has been undertaken on a manufacturing scale, and owing to the low cost of electrical power the product is expected to compete successfully with spirit produced by fermentation.—J. H. L.

*Influence of different compounds on the destruction of monosaccharides by sodium hydroxide and on the inversion of sucrose by hydrochloric acid.* 111. Constitutional formulae of the hydroxybenzoic acids and of sulphanilic acid. Waterman. See XVII.

## PATENTS.

*Diallylbarbituric acid and alkaloids of the morphine series; Compounds derived from — and process of making same.* P. Hüsey and W. Föhrenbach, Assignors to Soc. Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,255,423, Feb. 5, 1918. Date of appl., Apr. 9, 1917.

SEE Eng. Pat. 107,409 of 1916; this J., 1917, 979. The compounds are easily soluble in hot water, alcohol, methyl alcohol, acetone, and ethyl acetate, and insoluble in petroleum spirit. The compound from diallylbarbituric acid and ethylmorphine is a white, crystalline substance melting at 285° C.

*Anæsthetic bodies.* Parke, Davis, and Co., Assignees of L. Thorp, Detroit, Mich., and E. A. Wildman, Indianapolis, Ind., U.S.A. Eng. Pat. 105,746, Feb. 27, 1917. (Appl. No. 2928 of 1917.) Under Int. Conv., Apr. 17, 1916.

SEE U.S. Pat. 1,193,651 of 1916; this J., 1916, 979.

*Anæsthetic bodies.* Parke, Davis, and Co., Assignees of L. Thorp, Detroit, Mich., U.S.A. Eng. Pat. 106,087, Feb. 27, 1917. (Appl. No. 2896 of 1917.) Under Int. Conv., Apr. 24, 1916.

SEE U.S. Pat. 1,193,634 of 1916; this J., 1916, 979.

*Glutamic acid; Process to separate — from other amino acids.* A. Corti, Dübendorf, Switzerland. U.S. Pat. 1,255,390, Feb. 5, 1918. Date of appl., Dec. 8, 1916.

SEE Eng. Pat. 106,081 of 1916; this J., 1917, 979.

## XXIII.—ANALYSIS.

*Permanganate solutions; Preparation of N/100* —. J. O. Halverson and O. Bergeim. J. Ind. Eng. Chem., 1918, 10, 119–120.

THE preparation of weak standard solutions of potassium permanganate by diluting stronger solutions is inaccurate, owing to the presence of organic matter in the distilled water. By means of the following method N/100 potassium permanganate solutions which will retain their strength indefinitely may be prepared. A solution of 0.4 gm. of pure potassium permanganate crystals in a litre of re-distilled water is digested for 36 hours near its boiling point in a flask which has been rinsed with the same water. After cooling, and standing over night, the supernatant liquid is filtered with the aid of suction through a Buchner funnel lined with ignited asbestos, and kept in the dark in a bottle, free from organic matter. After standing 2 to 3 days, the solution is standardised by adding 10 c.c. of 10% sulphuric acid, rendered faintly pink with permanganate, to 10 c.c. of N/50 oxalic acid solution, placing the vessel in a water bath at 65° C. for a few minutes, and titrating it with the permanganate solution. A correction is obtained by titrating 10 c.c. of the sulphuric acid diluted with the same volume of water.—C. A. M.



*Volumetric determination of sulphates in water extracts of soils.* Christie and Martin. *See* XVI.

*Gliding dialysis. [Preparation of fruit syrups.]* Thoms. *See* XIXA.

*Use of micro-organisms to determine the preservative value of spices.* Bachmann. *See* XIXA.

#### PATENT.

*Bunsen burners.* Eng. Pat. 113,069. *See* IIA.

### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Barbet et Fils et Cie. Tubular evaporating, condensing, and cooling apparatus. 5232. Mar. 25. (Fr., Mar. 31, 1917.)

Bawtree and Hadfield. *See* VIII.

Bouhon. Purifiers and recuperators for smoke, air, or gases. 4911. Mar. 20.

Bregeat. Recovery of volatile solvents used in manufacture. 5240. Mar. 25. (Fr., Nov. 29, 1917.)

British Thomson-Houston Co. (General Electric Co.). Vacuum tubes. 4933. Mar. 20.

Brookfield, Gaunt, and Tylor and Sons. Crucible furnaces. 5095 and 5152. Mar. 22 and 23.

Francart. Kilns, furnaces, ovens, etc. 5027. Mar. 21.

Francart. Kilns, muffle ovens, etc. 5028. Mar. 21.

Francart. Kilns, tunnel ovens, etc. 5081. Mar. 22.

Francart. Heat treatment. 5464. Mar. 28.

Hunter, and Simon-Carves, Ltd. Settling-tanks for clarifying liquids. 4993. Mar. 21.

Kestner. Evaporators. 5107. Mar. 22.

Moore. Apparatus for agitating liquids, powders, granular substances, etc. 4800. Mar. 19.

Quinan. Treatment of gases. 5397. Mar. 27.

Ronse. Manufacture and utilisation of agglomerates. 4776. Mar. 18.

Searle. Distillation of solid and liquid substances. 4738. Mar. 18.

Sutcliffe. Grinding, pulverising and grading materials. 4984. Mar. 21.

##### COMPLETE SPECIFICATIONS ACCEPTED.

5014 (1915). Chenard. Distilling methods and apparatus. Mar. 27.

15,136 (1916). De Baufre. Evaporators. (114,164.) Apr. 4.

17,714 (1916). West and Wild. *See* II.

2017 and 2295 (1917). Wolf. Apparatus for manufacturing, storing, treating, or transporting corrosive liquids or substances. (113,992.) Mar. 27.

3443 (1917). Hutchins. Compression of granular and other substances. (114,002.) Mar. 27.

3645 (1917). Pendlebury. Means for maintaining suspended matter in liquids in a state of suspension. (114,016.) Mar. 27.

3746, 3942, and 12,201 (1917). Hall. Furnaces. (114,177.) Apr. 4.

4074 (1917). Aktieselskabet Myrens Verksted. *See* XIX.

5759 (1917). Hadden (Commercial Research Co.). Rotary furnace structures. (114,228.) Apr. 4.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING. •

##### APPLICATIONS.

Ashcroft. Carbonisation of coal or distillation and cracking of oils, pitches, shales, peat, etc. 4781 and 5502. Mar. 19 and 28.

Ballingall and Dempster. 4802. *See* VII.

Bennion and Wilde. 5349. *See* XXIII.

Carper and Tulloch. Mineral oil mixtures. 5167. Mar. 23.

Dale, Brown, and Co., and Newall. Gas-producers. 5333. Mar. 27.

Gibbons Bros., and Masters. Feeding furnaces of gas-retort settings, coke-ovens, muffles, etc. 4805. Mar. 19.

Helps. Gas manufacture. 5526. Mar. 30.

Hutchins. 5092. *See* XIII.

Kent and Mulliner. Treatment of carbonaceous substances. 5014. Mar. 21.

Lamplough, and Oil Extractors, Ltd. Distillation of coal. 5511. Mar. 28.

Poore. Treatment of wood, peat, etc. 4963. Mar. 20.

Searle. 4738. *See* I.

Soc. Franco-Belge de Fours à Coke. Horizontal coking-ovens. 5328. Mar. 26.

Spicer (Wells). Carbonisation of wood, etc. 4925. Mar. 20.

Tinker. Fuel oils. 4757. Mar. 18.

##### COMPLETE SPECIFICATIONS ACCEPTED.

17,714 (1916). West and Wild. Retorts and methods of working the same. (113,981.) Mar. 27.

3752 (1917). Goodwin and Macrae. Manufacture of fuel from peat. (114,026.) Mar. 27.

4127 (1917). Shedlock. *See* XIII.

5255 (1917). Marks (Soc. Franco-Belge de Fours à Coke.) Regenerators for coke-ovens. (114,222.) Apr. 4.

#### III.—TAR AND TAR PRODUCTS.

##### APPLICATIONS.

Ashcroft. 4781 and 5502. *See* II.

Crabb. Separating and purifying creosote oil. 5080. Mar. 22.

##### COMPLETE SPECIFICATION ACCEPTED.

4436 (1917). Bostaph Engineering Co. Process of producing phenols. (106,274.) Mar. 27.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATION.

Levinstein, Ltd., and Wyler. Sulphur dyestuff. 5519. Mar. 28.

##### COMPLETE SPECIFICATION ACCEPTED.

4057 (1917). Inmray (Soc. Chem. Industry in Basle). Manufacture of metal compounds of hydrazone dyestuffs, and process of dyeing with such compounds. (114,189.) Apr. 4.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Burnley and Howgate. Acid feeders of revolving retort carbonising apparatus for treating fabrics, etc. 5200. Mar. 25.

Cellon, Ltd., Tucker, and Tyrer and Co. Production of dopes. 5300. Mar. 26.

Flatters. 5551. *See* VI.

Gow and St. Armande. Waterproofing solution for aeroplane wings, etc. 4957 and 4958. Mar. 20.

Henshilwood and Ward. Treatment of fibrous substances. 5052. Mar. 22.

Marr. 4721. *See* XV.

Poulson. 4913. *See* VI.

Scatchard. Destruction or extraction of silk from rags, fabrics, or clothing. 5063. Mar. 22.  
Spicer. Production of filter paper, etc. 5484. Mar. 28.

COMPLETE SPECIFICATION ACCEPTED.

11,784 (1917). Wade (Soc. Union Franç. des Papeteries). Regeneration of old paper. (114,257.) Apr. 4.

VI.—BLEACHING; DYEING; PRINTING; FINISHING.

APPLICATIONS.

Flatters. Rendering textile fabrics gas proof and/or waterproof. 5551. Mar. 30.  
Henshilwood and Thornber. Bleaching, dyeing, finishing, and like machines. 5432. Mar. 28.  
Poulson. Process for rendering textile fabrics, etc., waterproof and fireproof. 4913. Mar. 20.

COMPLETE SPECIFICATION ACCEPTED.

4057 (1917). Imray. See IV.

VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

APPLICATIONS.

Ashcroft. Heat treatment of potash rocks with salts for extraction of potash as soluble compounds. 4781 and 5501. Mar. 19 and 28.

Ashcroft. Melting and purifying salts. 4783 and 5503. Mar. 19 and 28.

Bagley. Concentration of nitric acid. 4796. Mar. 19.

Bailey, Denny, and Jeffries. Production of ammonium nitrate. 4877. Mar. 20.

Ballingall and Dempster. Hydrogen-gas plants. 4802. Mar. 19.

Clayton Aniline Co., Marchant, and Schedler. Manufacture of chlorosulphonic acid. 5199. Mar. 25.

Dutt and Dutt. Production of alumina, sodium and potassium carbonates and aluminates, and potassium chloride. 5072. Mar. 22.

Dutt and Dutt. Production of alumina, sodium and potassium carbonates and aluminates, magnesia, and calcium chloride. 5073. Mar. 22.

Harbord. 4854. See X.

Matsushima. 5479. See XI.

Quinan. Production of nitric acid. 5397. Mar. 27.

COMPLETE SPECIFICATIONS ACCEPTED.

2017 and 2295 (1917). Wolf. See I.

3933 (1917). Grossmann. Utilisation of nitre cake. (114,180.) Apr. 4.

6767 (1917). Dawson. Production and separation of ammonium sulphate and Glauber's salt. (114,236.) Apr. 4.

7418 (1917). Tungal and Haughton. Apparatus for use in concentrating sulphuric or other acids. (114,072.) Mar. 27.

VIII.—GLASS; CERAMICS.

APPLICATIONS.

Bawtree and Hadfield. Proofing-materials for treating the walls of porous vessels. 5225. Mar. 25.

Kitson. Furnaces for glass manufacture, etc. 4922. Mar. 20.

IX.—BUILDING MATERIALS.

APPLICATIONS.

Afford. Artificial stone. 4752. Mar. 18.

Davidson. Process of seasoning sawn timber. 5360. Mar. 27.

Friedrich. Producing cold glaze on building materials, particularly cement. 5385. Mar. 27.

Jones (White Heat Products Co.). Manufacture of artificial stones. 4771. Mar. 18.

COMPLETE SPECIFICATION ACCEPTED.

3947 (1917). Thompson (Reilly). Preservation of wood, and production of substances therefor. (114,181.) Apr. 4.

X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

APPLICATIONS.

Abrahamsen and Pedersen. Manufacture of sheet-metal plate with a protective metal coating. 4859. Mar. 19.

Baur and Nagel. Recovery of gold, silver, etc., from sea water. 5104. Mar. 22. (Ger., Jan. 18, 1917.)

Bingham. Blast furnaces. 5261. Mar. 26.

Brayshaw, Brayshaw, and Yates. Gas-fired furnaces for metallurgical and like furnaces. 4797. Mar. 19.

Brookfield and others. 5095 and 5152. See I. Coles. Coating iron and steel for protective purposes. 4971 and 4972. Mar. 21.

Daponté and Newman. Zinc-vapour rust-proofing. 5023. Mar. 21.

Estelle. Electrolytic production of iron. 5398. Mar. 27.

Francart. 5464. See I.

Frankignoul. Blast furnaces. 5117. Mar. 22.

Harbord. Muffled chamber for use as roasting furnace or kiln. 4853. Mar. 19.

Harbord. Roasting complex zinc sulphide ores or concentrates, particularly in conjunction with the manufacture of sulphuric acid. 4854. Mar. 19.

Hepworth. Hardening and tempering wire. 5179. Mar. 23.

MacKay. Treatment of ores and oxides and recovery of lead in metallic form and zinc in the form of an enriched sulphide. 5097. Mar. 22.

Mate and Poulson. Production of malleable iron. 5347. Mar. 27.

Reynolds. Production of steel. 4767. Mar. 18.

Rigaud. Treatment of metallic minerals. 4774. Mar. 18. (Fr., May 12, 1917.)

Schmidt. Smelting-furnace heated by oil or gas. 5227. Mar. 25.

COMPLETE SPECIFICATIONS ACCEPTED.

3722 (1917). Larsen. Treatment of iron-sponge. (114,025.) Mar. 27.

3972 (1917). Hilger, Ltd., and Twyman. Annealing and apparatus used therein. (114,183.) Apr. 4.

4321 (1917). Gill (Jobbins). Utilisation of aluminium skimmings, screenings, slags, and analogous materials. (114,204.) Apr. 4.

4954 (1917). Norsk Elektrisk Metalindustri Aktieselskap. Converting zinc powder into liquid zinc. (105,558.) Mar. 27.

4975 (1917). Woodward, Gatty, Saunt and Co., and Scott. Heat treatment of metals. (114,217.) Apr. 4.

7184 (1917). Perry, and Metalloids, Ltd. Manufacture of iron. (114,237.) Apr. 4.

12,265 (1917). Morterud. Tempering and hardening copper. (114,105.) Mar. 27.

17,045 (1917). Westinghouse Metal Filament Lamp Co. (Westinghouse Lamp Co.). Method of working refractory metals. (114,282.) Apr. 4.

18,215 (1917). Chattaway. Machine for testing the hardness of metals. (114,126.) Mar. 27.

XI.—ELECTRO-CHEMISTRY.

APPLICATIONS.

Ash, and British Thomson-Houston Co. Electric arc furnaces. 4841. Mar. 19.

Estelle. 5398. See X.

Jackson (Burgess Battery Co.). Dry electric batteries. 4866. Mar. 19.



Matsushima. Apparatus for electrolysing salt solution. 5479. Mar. 28. (Japan, Mar. 30, 1917.)

COMPLETE SPECIFICATIONS ACCEPTED.

18,113 (1916). Schuster. Galvanic cells. (113,984.) Mar. 27.  
3581 and 3582 (1917). Schuster. Diaphragms for galvanic cells. (114,010 and 104,011.) Mar. 27.  
3584 (1917). Schuster. Galvanic primary cells. (104,012.) Mar. 27.

XII.—FATS; OILS; WAXES.

APPLICATIONS.

Benn'on and Wilde. *See* XXIII.  
Britton, Griffiths Bros. and Co., Hadfield, and Hadfields, Ltd. Removal of solid fatty acids from liquid fatty acids by chemico-physical means. 5018. Mar. 21.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Cellon, Ltd., Tucker, and Tyrer and Co. 5300. *See* V.  
Hutchins. Manufacture of lampblack. 5092. Mar. 22.

COMPLETE SPECIFICATIONS ACCEPTED.

3815 (1917). Norsk Aktieselskab for Elektrokemisk Industri. Manufacture of pigments from titanium compounds. (104,885.) Mar. 27.  
4127 (1917). Shedlock. Manufacture of lampblack. (114,040.) Mar. 27.  
5960 (1917). De Bruyn. Distemper. (114,230.) Apr. 4.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATIONS.

Dunlop Rubber Co., and Twiss. Vulcanisation of rubber, etc. 4941 and 4944. Mar. 20.  
Moore. Apparatus for drying or heat treatment of rubber, etc. 4801. Mar. 19.

COMPLETE SPECIFICATION ACCEPTED.

1018 (1917). Dunlop Rubber Co., Macbeth, and Cunningham. Manufacture of articles composed of rubber or similar material. (113,987.) Mar. 27.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Bowman and Posnett. Chrome, alum, or vegetable tanning. 5140. Mar. 23.  
McCay. Manufacture of leather. 4837. Mar. 19.  
Marr. Apparatus for drying and conditioning leather, yarns, textile and like materials. 4721. Mar. 18.

COMPLETE SPECIFICATION ACCEPTED.

16,719 (1917). Turner Tanning Machinery Co. Machines for treating hides, skins, and leather. (111,486.) Mar. 27.

XVI.—SOILS; FERTILISERS.

COMPLETE SPECIFICATION ACCEPTED.

15,812 (1917). Becquevort. Production of fertilisers. (114,277.) Apr. 4.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATION.

Henley. Treatment of fermenting wort or wash. 5160. Mar. 23.

COMPLETE SPECIFICATION ACCEPTED.

882 (1917). Wallerstein and Wallerstein. Extract having the flavour and aroma of malt, process of producing the same, and beverage therefrom. (107,367.) Apr. 4.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Ferguson and Gillespie. Food product and beverage. 5151 and 5358. Mar. 23 and 28.  
Greville. Treatment of flour. 4983. Mar. 21.  
Griffiths and Hartley. Apparatus for aerating sewage. 4726. Mar. 18.  
Makin. Foodstuffs, and manufacture thereof. 5060. Mar. 22.  
Rialland. Process for purifying liquids. 5016. Mar. 21. (Fr., Dec. 10, 1917.)  
Siemens-Schuckertwerke. Purifying water in boilers, etc. 5006. Mar. 21. (Ger., Mar. 22, 1917.)  
Spire. Treating and preserving fruits and vegetables. 5033. Mar. 21.  
Wallace. Preserving animal and vegetable substances. 5309. Mar. 26.

COMPLETE SPECIFICATIONS ACCEPTED.

882 (1917). Wallerstein. *See* XVIII.  
3007 (1917). Higginbottom. Flour milling. (113,997.) Mar. 27.  
4070 (1917). Aktieselskabet Myrens Verksted. Drying fish, meat, fruit, and the like by vacuum. (105,072.) Apr. 4.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATION.

Britton and others. 5018. *See* XII.

COMPLETE SPECIFICATIONS ACCEPTED.

17,759 (1916). Ellis (Chem. Fabr. von Heyden A.-G.). Manufacture of derivatives of isovaleric acid. (113,983.) Mar. 27.  
3272 (1917). Dreyfus. Manufacture of acetic aldehyde. (105,064.) Mar. 27.  
4058 (1917). Imray (Soc. Chem. Industry in Basle). Manufacture of a preparation of opium. (114,190.) Apr. 4.  
14,152 (1917). Aylsworth, and Savings Investment and Trust Co. Process of effecting organic chemical reactions. (110,547.) Mar. 27.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Garrett and Greene. Colour photography. 5162. Mar. 23.  
Greene. Colour photography. 5161. Mar. 23.  
Scott. Coating films of celluloid, etc., with sensitive photographic emulsion. 4817. Mar. 19.

XXII.—EXPLOSIVES; MATCHES.

APPLICATION.

Oxley. Matches. 4968. Mar. 21.

XXIII.—ANALYSIS.

APPLICATION.

Bennion and Wilde. Determining presence of water in oils, fats, petroleum, etc. 5349. Mar. 27.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Standardisation of chemical stoneware.* Nielsen and Garrow. See VIII.

**PATENTS.**

*Continuous-rectification apparatus ; Cooling plates for —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 105,549, Mar. 8, 1917. (Appl. No. 3444 of 1917.) Under Int. Conv., Mar. 27, 1916.

A COOLING plate for rectifying column consists of a concave basin with a central chimney, hood, and overflow, and a superposed perforated plate. A cooling coil is immersed in the reflux liquid which collects on the upper side of the perforated plate. The perforations of the plate are confined to the neighbourhood of the cooling coil.—W. H. C.

*Grinding machines.* G. Bamburg, London. Eng. Pat. 113,677, Mar. 9, 1917. (Appl. No. 3492 of 1917.)

A MACHINE for grinding resinous or fatty substances, or other materials which tend to clog the machine, comprises a drum formed by mounting a number of split wooden pulley wheels of large face area close together on a shaft. Rasping plates are fixed to the outer periphery of the drum, and a wooden hopper closely surrounds the drum so that only fine particles can fall between the drum and the hopper. An inclined baffle-plate is mounted on the side of the hopper facing the direction of the revolving rasping drum. It may have a rasping surface or may be smooth, and it is heavily backed with wood to lessen vibration. The material is admitted through a door in the hopper and is forced by the revolving rasps against the fixed baffle. The powder produced passes between the drum and the hopper into a receptacle underneath.—J. H. P.

*Attrition mill.* E. P. Alsted, Truesdell, Wis. U.S. Pat. 1,255,845, Feb. 12, 1918. Date of appl., Sept. 13, 1916.

THE mill comprises opposed rotating plates provided with radial ribs, the working edges of which are undercut and converge towards the axis of the plates for a portion of their length and are parallel to each other at other portions of their length so as to advance the line of cutting engagement of the ribs towards the periphery of the plates for a portion of the length of the ribs and towards the axis along other portions of the ribs. Portions of the radial ribs are bevelled towards the plates. Inner and outer transverse ribs extend across the radial ribs, the inner rib being eccentric to and the outer rib concentric with the axis of the plate. During rotation the inner transverse ribs of opposite plates partly overlap each other to permit the passage of material from pockets inclosed within the ribs to pockets outside.—J. H. P.

*Gases ; Evacuation and compression of —.* E. S. G. Rees, Stafford. U.S. Pat. 1,252,178, Jan. 1, 1918. Date of appl., May 17, 1915.

A ROTARY air pump has its suction connected with the vessel to be exhausted and also with the water space of a sealed receptacle partly filled with water maintained at a comparatively low temperature. The evacuated air is delivered into the sealed receptacle by the pump and is withdrawn therefrom and delivered to the atmosphere by a second rotary air pump, the suction of which is connected with a second supply of water.

—W. H. C.

*Separation of suspended particles from gases ; Apparatus for electrical —.* W. A. Schmidt and G. C. Roberts, Assignors to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,252,183, Jan. 1, 1918. Date of appl., Sept. 9, 1913.

THE gas is passed from a flue through a number of vertical, tubular, collecting electrodes within each of which a flexible ionising electrode is suspended concentrically. The ends of the central electrodes are enlarged where they leave the tubes to reduce the surface field intensity at these points.

—W. H. C.

*Dryer [for fibrous materials].* F. G. Sargent, Westford, Mass., Assignor to C. G. Sargent's Sons Corporation, Graniteville, Mass. U.S. Pat. 1,254,073, Jan. 22, 1918. Date of appl., June 4, 1917.

THE material to be dried is fed intermittently by a reciprocating feed apron on to a reciprocating conveyor within a casing. The material is fed only on the forward stroke of the conveyor. Swinging "kicker arms" are provided above the conveyor to loosen the material.—W. H. C.

*Dry-kiln.* H. Hunter. Assignor to The Hunter Dry Kiln Co., Indianapolis, Ind. U.S. Pat. 1,254,142, Jan. 22, 1918. Date of appl., Feb. 12, 1917.

THE kiln consists of a closed rectangular chamber having a partition, spaced from one side wall, depending from the ceiling to a point near the floor. A second partition is spaced between the first partition and the adjacent wall and extends upwards from the floor to a point near the ceiling. A heating coil is arranged in the space between the second partition and the wall. Air enters through the wall near the bottom, passes upwards over the heater and then descends between the partitions and enters the main compartment of the chamber below the inner partition. Each partition consists of a number of doors, those of the partition nearer the wall being hinged at the bottom and those of the other partition at the top.—W. H. C.

*Drying apparatus.* J. L. Pierce, Assignor to Pacific Commercial Co., Manila, Philippine Islands. U.S. Pat. 1,254,431, Jan. 22, 1918. Date of appl., Dec. 28, 1916.

A NUMBER of superposed conveyors, each provided with a heating unit, are arranged within a casing. Heated air is supplied to the heating units, and products of combustion are also supplied to supplement the heated air without mixing with it.

—W. H. C.

*Dryer.* T. G. Haney, New London, Conn. U.S. Pat. 1,255,162, Feb. 5, 1918. Date of appl., June 18, 1917.

THE material to be dried is passed through the drying chamber on a reticulated conveyor, and is dried by heated air which is passed over the upper run of the conveyor and is then prevented from coming into contact with the air entering the heater.—W. H. C.

*Drying apparatus.* J. B. Adt, Baltimore, Md. U.S. Pat. 1,255,843, Feb. 12, 1918. Date of appl., July 17, 1917.

A ROTARY drying drum has an inlet for the material to be dried and an air outlet at one end and a material outlet and air inlet at the opposite end. The latter end of the drum extends into an air-expansion chamber into which it opens, and a heating jacket surrounds the drum and the opening into the air-expansion chamber. The jacket is carried by the drum and rotates with it. At the end remote from the expansion chamber a header



is provided and this communicates with the jacket. Air is delivered from an air heater into the header.—J. H. P.

*Refrigerating apparatus, and oil separator and condenser mechanism therefor.* Refrigerating apparatus. B. S. McClellan, Chicago, Ill. U.S. Pats. (A) 1,254,518 and (B) 1,254,519, Jan. 22, 1918. Dates of appl., Dec. 23 and 31, 1915. Renewed Apr. 19 and Oct. 6, 1917.

(A) A CONDENSER chamber having a gas inlet communicating with the compression chamber of a compressor and a sealed outlet for refrigerating liquid, is provided with a water cooling coil and is surrounded by an outer cooling chamber through which water is circulated. (B) A compressor having an oil reservoir for lubricating the compressing mechanism, delivers a volatile fluid to a condenser, whence the condensed liquid flows to an expansion valve casing provided with an expansion chamber and thence to an expansion coil, which in turn communicates with the compression chamber of the compressor. Below the expansion chamber is a reservoir in which oil separated from the volatile fluid in the expansion chamber collects and whence it is returned to the oil reservoir of the compressor.—W. H. C.

*Refrigerating apparatus.* O. E. Bornhauser and J. E. Herman, Assignors to The Wagner Refrigeration Co., Sandusky, Ohio. U.S. Pat. 1,255,505, Feb. 5, 1918. Date of appl., Dec. 15, 1917.

A REFRIGERATING apparatus of the intermittent absorption type is provided with a hydraulic diaphragm, controlled by the level of the liquid in the receiver, which serves to regulate the supply of water and gas to the apparatus.—W. H. C.

*Refrigerating apparatus.* O. E. Bornhauser, Assignor to J. E. Herman and C. L. Wagner, Sandusky, Ohio. U.S. Pat. 1,255,286, Feb. 5, 1918. Date of appl., Feb. 19, 1917.

THE apparatus is of the intermittent absorption type and consists of an ammonia generator-absorber with a burner and gas supply, a condenser with a water circulating system for the ammonia gas, and a receiver for the liquid ammonia. A cooling coil included in a by-pass from the condenser system is placed in the liquid container of the generator-absorber. The level of the liquid ammonia in the receiver actuates a device for regulating the gas supply and for throwing the by-pass to the cooling coil out of or into action, according as the generator-absorber is acting as a generator or an absorber. The water supply in the condenser system is regulated by a thermostatic connection actuated by the temperature of the liquid in the generator-absorber. The temperature of the generator-absorber also controls a valve on the gas supply pipe which cuts off the gas when the temperature exceeds a predetermined point, and automatically locks the valve so that it cannot be reopened except by hand.—J. H. J.

*Refrigerating apparatus.* M. F. Ewen, Chicago, Ill. Assignor to Larsen Ice Machine Co. (Inc.), Wilmington, Del. U.S. Pat. 1,255,723, Feb. 5, 1918. Date of appl., Jan. 4, 1915.

A POSITIVELY operated valve is interposed between the condenser-receiver and the expansion chamber of the apparatus. The valve is operated by the pump which transfers the absorbent to the still at predetermined intervals.—W. H. C.

*Centrifugal machine.* U. F. S. Hartman and A. H. La Casse, Crockett, Cal. U.S. Pat. 1,255,007, Jan. 29, 1918. Date of appl., May 22, 1917.

THE frustum-shaped basket of the machine has segmental discharge ports formed in the base and is connected by means of a spider to a hollow

vertical shaft, driven from above, by which it is rotated. The ports are closed when the basket is rotating by a bottom piece supported by an inner shaft passing through the hollow shaft and which is raised or lowered by a lever arm.—W. H. C.

*Boiler feed-water; Method of and apparatus for treating—* H. C. Heaton, Chicago, Ill. Assignor to The Babcock and Wilcox Co., Bayonne, N.J. U.S. Pat. 1,255,164, Feb. 5, 1918. Date of appl., Dec. 8, 1914.

THE feed-water is passed through the low-pressure stage of a multi-stage economiser and then withdrawn along with any air or gases into a tank where the air and gases are separated. The water is then heated by exhaust steam and passed through the high-pressure stage of the economiser into the boiler.—W. H. C.

*Steam-boiler economiser and method of operating the same.* D. S. Jacobus, Jersey City, N.J., Assignor to The Babcock and Wilcox Co., Bayonne, N.J. U.S. Pat. 1,255,170, Feb. 5, 1918. Date of appl., Dec. 8, 1914.

IN using artificially softened or naturally soft water in a boiler, the main supply of water is passed through a section of an economiser: it is then mixed with the hot concentrated liquid from the boiler, and the mixture is passed through another section of the economiser, at a higher pressure, into the boiler.—W. H. C.

*[Agitating] barrel.* J. L. Malm, Assignor to The Midwest Metals Co., Denver, Colo. U.S. Pat. 1,255,196, Feb. 5, 1918. Date of appl., Apr. 26, 1915.

A HOLLOW cylinder rotating on a horizontal axis is closed at one end and connected to a source of fluid pressure at the other end. A series of longitudinal baffles is provided on the inner cylindrical wall, the baffles being connected by filter sheets so as to form a series of chambers to receive the filtrate and then discharge it. A discharge door for solid material is provided in the cylindrical wall midway between the ends, and additional baffles converge spirally from both ends towards the discharge door to collect and facilitate discharge of the filtered material. In a modification, a number of discharge doors are provided, each having a set of converging spiral baffles on each side of it.—W. F. F.

*Eraporator.* G. H. Benjamin, New York. U.S. Pat. 1,255,502, Feb. 5, 1918. Date of appl., Mar. 30, 1917.

A VERTICAL cylindrical casing is provided with a feed pipe, blow-off pipe, and vapour discharge pipe. The heating element projects into the casing and is mounted on a detachable door secured over an opening in the casing, the door and heating element being supported wholly by means outside and independent of the casing.—W. F. F.

*Absorbing a fixed quantity of gas by a liquid; Apparatus for—* G. Braam, Rotterdam, Netherlands. U.S. Pat. 1,256,291, Feb. 12, 1918. Date of appl., Dec. 1, 1917.

AN absorption vat provided with means for agitating the liquid therein is in open communication with a regulating vat. The liquid displaced from the absorption vat by the gas flows into the regulating vat, wherein it is measured and whence it flows back to the absorption vat at a rate dependent on the speed with which the gas is absorbed. The gas is led into the absorption vat at the part which is on top when the vat is at rest.—J. H. P.

*Carboy hampers or crates.* A. W. Cowburn, and W. H. Cowburn and Cowpar, Ltd., Manchester. Eng. Pat. 113,930, Oct. 31, 1917. (Appl. No. 15,866 of 1917.)

[*Nickel*] *catalytic agent.* A. Mittasch, C. Schneider, and H. Morawitz, Assignors to Badische Anilin u. Soda Fabr., Ludwigshafen, Germany. U.S. Pat. 1,256,032, Feb. 12, 1918. Date of appl., June 4, 1914.

SEE Eng. Pat. 8462 of 1914; this J., 1915, 822.

*Metal heating, metallurgical and analogous heating furnaces.* Eng. Pat. 113,591. See X.

*Uninflammable liquids for use in insulating and other similar purposes.* U.S. Pat. 1,255,618. See XI.

## II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Effect of low-temperature oxidation on the hydrogen in — and the change in weight of coal on drying.* S. H. Katz and H. C. Porter. Techn. Paper No. 98, U.S. Bureau of Mines. [15 pages.]

MOST of the work of previous investigators of the oxidation of coal as related to the hydrogen of the coal substance was at the temperature of boiling water and above. The results varied widely, probably due to the absorption of gas by the coal on drying. Mahler has given trustworthy data showing that in low-temperature oxidation hydrogen is evolved from coal in the form of water (see this J., 1910, 744, 863). In the apparatus used by the authors, nitrogen or air passed from a drying train through a large quantity of coal at room temperature, thence through another drying train which caught the water given up by the coal. The weight of water and the change of weight of the coal were then ascertained. When nitrogen was passed through the coal there was invariably a loss in weight, much less, however, than the weight of water evolved from the coal. The small amount of oxygen in the nitrogen is not sufficient to account for the discrepancy, which may be due to absorption of nitrogen by the coal. When air was passed through there was always a gain in weight despite the loss of water. Analyses of the coal before and after the experiments showed an increase in oxygen content and a corresponding decrease in other constituents, but no loss of hydrogen could be shown by these analyses. No evidence is afforded that water is produced by the oxidation of coal at ordinary temperatures.

—J. E. C.

*Gas; Effect of the removal of light oils from —.* L. J. Willien. Amer. Gas Inst., 1917. Gas J., 1918, 141, 442—443.

LIGHT oils may be removed from coal gas by scrubbing, compression, or cooling. The average yield is about 3 gallons per ton of coal carbonised, the following being the approximate composition: benzol, 50 to 60%; toluol, 12 to 13%; solvent naphtha, 10 to 12%; heavy naphtha and wash oil, 13%. The author gives tables showing the effects of removing light oils from coke-oven gas, coal gas, carburetted water-gas, and mixed gas, and the following conclusions are drawn:—The candle power is materially reduced (up to 50% with fairly complete removal). The heating value is reduced from 1.5 to 8%. The naphthalene content is reduced about 75% if the gas is scrubbed with a naphthalene-free oil, whilst partial scrubbing is liable to increase naphthalene stoppages. From

30 to 50% of the carbon bisulphide is removed. There is less accumulation of drip-oil in the distribution system; the gas is more permanent; the scrubbed gas is in better condition for re-enrichment, and the gas is more uniform and less liable to cause stoppage in the service pipes.

—J. E. C.

*Natural gas and its constituents; Compressibility of — with analyses of natural gas from 31 cities in the United States.* G. A. Burrell and I. W. Robertson. U.S. Bureau of Mines, Techn. Paper 158, 1917. Petroleum Technology No. 32. [16 pages.]

THE apparatus used for determining the compressibility of gases consisted of three water-jacketed capillary tubes, the upper ends of which were sealed, the lower ends passing through stuffing-boxes into a steel compression machine containing mercury. By means of a plunger, the mercury could be forced into the glass capillary tubes, thereby compressing the gases contained therein to any desired degree. One tube contained the gas under examination; the other two contained air and served as pressure gauges, the pressure-volume curve for air having been previously determined (Burrell and Robertson, Techn. Paper 131; this J., 1916, 955). The compressibility of pure methane, ethane, propane, and carbon dioxide and of samples of natural gas from a number of cities in the United States was determined and curves were constructed showing the percentage deviation from a perfect gas at pressures varying from 0 to over 40 atmos. The deviations per atmos. for the pure gases were found to be:  $\text{CH}_4$ , 0.228%;  $\text{C}_2\text{H}_6$ , 0.90%;  $\text{C}_3\text{H}_8$ , 1.9%;  $\text{CO}_2$ , 0.67%. To illustrate the method of calculating the compressibility of any natural gas, the following example is given: The natural gas used in Buffalo is composed of 88.1%  $\text{CH}_4$ , 11.5%  $\text{C}_2\text{H}_6$ , and 0.4%  $\text{N}_2$ . The partial pressures of the three constituents at 40 atmospheres are:  $-\text{CH}_4 = 0.881 \times 40 = 35.2 = P_1$ ;  $\text{C}_2\text{H}_6 = 0.115 \times 40 = 4.6 = P_2$ ;  $\text{N}_2 = 0.004 \times 40 = 0.2 = P_3$ . The formula for total deviation is,  $D = aP_1 + bP_2 + cP_3 + dP_4 + \dots$ , where a, b, c, d, etc., are the deviations per atmos. for the constituents. Substituting values in the formula,  $D = (0.228 \times 35.2) + (0.90 \times 4.6) + 8.03 + 4.14 = 12.17\%$ , or the compressibility at 40 atmospheres is 87.83 on the basis of that of an ideal gas being 100. This method gives results closely in accordance with actual determinations and can be applied to calculate the compressibility of any natural gas, provided an ordinary analysis has been made.

—L. A. C.

*Tar; Water in gas —.* M. Coudelou. J. Usines à Gaz. Gas. J., 1918, 141, 201.

IN order to reduce the percentage of water in tar delivered to distillers, the following means have been adopted. In place of the plentiful supply of ammoniacal liquor kept in the hydraulic main to form a hydraulic seal, and introduced under the surface of the tar, only a thin stream of clean water is admitted. The reservoir used to form a hydraulic seal in the passage of the tar from the hydraulic main to the works' tank, which helped to form an emulsion of the tar and water, has been replaced by a vertical length of pipe leading into the tank, the tar entering the tank along an inclined plane, down which it flows in a thin layer from which globules of water easily separate. A similar arrangement is used for filling the wrought-iron cylindrical vessel used for pumping the tar from the works' to the storage tank, in place of the single pipe used before both for filling and emptying the vessel. By the above means, the percentage of water in the tar has been reduced from 15—30% to an average of 5%. —L. A. C.



*Petrol; [Determination of] calorific value of —.*  
B. Blount. Analyst, 1918, 43, 89.

THE bomb calorimeter method is preferable for determining the calorific value of petrol, but it is necessary to enclose the petrol in a combustible envelope the calorific value of which is known; collodion may be used for the purpose.—W. P. S.

*Petrol: Determination of sulphur in —.* B. Blount. Analyst, 1918, 43, 89.

THE Carius method is the most reliable for the purpose. Burning the petrol under a tower down which a solution of ammonia and ammonium carbonate trickles is untrustworthy, as only a portion of the sulphur is retained. The use of a calorimeter bomb rarely gives good results. The sulphur in petrol cannot be oxidised by heating with concentrated nitric acid under a reflux condenser. The quantity of sulphur in petrol is usually less than 0.10%.—W. P. S.

*Comparative values of coke-oven benzols.* Haigh and Lamb. See III.

#### PATENTS.

*Pulverised fuel and air feeder and mixer.* G. W. Clendon, N.Y., Assignor to Locomotive Pulverized Fuel Co. U.S. Pat. 1,254,393, Jan. 22, 1918. Date of appl., July 17, 1914.

AN air conduit with an annular orifice discharges a hollow column of air into a mixing chamber. Pulverised fuel is conveyed through this hollow column by a screw conveyor and the air and fuel are mixed by a series of non-retarding rotating vanes in the mixing chamber.—J. E. C.

*Coke oven.* P. Plantinga, Cleveland, Ohio. U.S. Pat. 1,255,047, Jan. 29, 1918. Date of appl., July 26, 1916.

AN inclined coking chamber is combined with a set of parallel combustion chambers arranged one above the other and adjacent to one side of the coking chamber.—J. E. C.

*Coke-oven.* F. A. Bredel and J. Bergmann, St. Louis, Mo. U.S. Pat. 1,255,579, Feb. 5, 1918. Date of appl., Aug. 22, 1916.

A COKE-OVEN consists of an inclined coal chamber, the bottom wall being straight and the top wall being provided with an offset between the upper and lower ends of the chamber. The gas outlet is in the offset portion.—J. E. C.

*Gaseous fuel; Process of manufacturing —.*  
J. R. Rose, Edgeworth, Pa. U.S. Pat. 1,254,360, Jan. 22, 1918. Date of appl., May 19, 1916.

METHANE is supplied to one end of an elongated flue filled with refractory material and heated to at least 2200° F. (1200° C.). A preheated liquid hydrocarbon is supplied to an intermediate portion, and the mixture is dissociated giving a gas of high heating efficiency and of low carbon content.—J. E. C.

*Gas-producer.* E. A. W. Jefferies, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pat. 1,255,015, Jan. 29, 1918. Date of appl., Dec. 14, 1914.

THE fuel receptacle of the producer is supported by the ash-pan and rotates therewith, the supporting means being so arranged that the surface of the ash-pan from the centre to the periphery and the annular spaces between the wall of the fuel receptacle and the wall and bottom of the ash-pan are entirely unobstructed.

*Gas-cleaner.* J. Ruddiman, Hastings-upon-Hudson, N.Y. U.S. Pat. 1,254,438, Jan. 22, 1918. Date of appl., Apr. 25, 1917.

A GAS cleaner comprises a chamber, with means for maintaining a supply of wash water in the lower portion, a gas inlet and outlet, and a pair of rotary perforated baffles dipping in the wash water and interposed between the inlet and outlet. The gases passing through the perforations of one plate are directed by nozzles against the inter-spaces between the perforations of the adjoining baffle.—J. E. C.

*Gas burners.* Fletcher, Russell and Co., Ltd., T. W. Fletcher, and W. Banks, Warrington. Eng. Pat. 113,355, Mar. 27, 1917. (Appl. No. 4413 of 1917.

IN a gas furnace burner, a single removable and renewable nozzle is fitted on the top of an enlarged rectangular box-shaped chamber connected to the mixing tube. The nozzle may be open at its lower end, or closed and provided with slits or saw-cuts, whilst sliding plates are provided for regulating the opening to the passage of the mixture chamber.—J. E. C.

*Oils and gases; Process and apparatus for separation of —.* P. Jones, Santa Maria, Cal. U.S. Pat. 1,255,018, Jan. 29, 1918. Date of appl., Mar. 30, 1916.

A MIXTURE of oil and gas flowing from a well under pressure is introduced into a receiving chamber and maintained under pressure. The oil separates by gravity and passes away through a restricted outlet, whilst the gas passes away, by an outlet controlled by a valve, at a rate which is varied according to the rate of separation of the oil, so as to maintain the gas under pressure.—J. E. C.

*Petroleum; Treatment and refining of —.* R. Cross, Kansas City, Mo. U.S. Pat. 1,255,138, Feb. 5, 1918. Date of appl., Jan. 8, 1917.

HEAVY petroleum hydrocarbons are heated to a temperature not exceeding 450° C., and the vapours led over a body of the same material in a tank, which serves as a supply tank, on their way to a condenser. A pressure is maintained over both bodies of liquid which is equal to or greater than the normal vapour pressure of the gases in equilibrium with the liquid phase at the temperature of the oil at the entrance to the condenser. Oil is fed to the supply tank at a rate corresponding to the combined withdrawal of vapour from the supply tank and of residual oil from the still.

—L. A. C.

*Hydrocarbon oils; Apparatus for vaporising —.*  
C. B. Forward, Urbana, Ohio. U.S. Pat. 1,255,149, Feb. 5, 1918. Date of appl., Feb. 5, 1916.

A NUMBER of horizontal retorts of different elevations are contained in a furnace chamber, each consisting of an inner tube for oil and an outer tube for steam. The inner tubes extend beyond the furnace walls and are connected one with the other; the outer tubes terminate at the walls. Packing is provided between the ends of the two tubes; the inner tube is free to slide in the packing and the corresponding end of the outer tube is supported on rollers to allow for uneven expansion. The pipes from the jackets of the retort sections discharge into the jacket of a tubular steam-jacketed oil heater, the oil and steam from which pass into the corresponding parts of the upper retort sections.—L. A. C.

*Pressure-still [for producing light oils from gas oils].*  
J. J. Curran, Augusta, Kans. U.S. Pat. 1,255,714, Feb. 5, 1918. Date of appl., May 25, 1917.

A BATTERY of stills with a common feed pipe discharge through individual valved pipes into a

common drum in which light and heavy distillates are separated. Connected with the drum is a condenser for light hydrocarbon vapours. The heavy distillate passes by gravity from the drum to a separate still which is connected by a valved pipe to the same condenser. Steam is passed into all the stills.—L. A. C.

[Gaseous] fuel; Apparatus for producing combustible —. J. R. Rose, Edgeworth, Pa., U.S.A. Eng. Pat. 113,585, Feb. 28, 1917. (Appl. No. 16,126 of 1917.)

SEE U.S. Pat. 1,252,032 of 1918; this J., 1918, 141 A.

Gaseous fuel; Process of manufacturing —. J. R. Rose, Edgeworth, Pa., U.S.A. Eng. Pat. 113,657, Feb. 28, 1917. (Appl. No. 2983 of 1917.)

SEE U.S. Pat. 1,230,226 of 1917; this J., 1917, 862.

Process of making superphosphate. U.S. Pat. 1,255,829. See XVI.

## II.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

The sugar factory as a source of supply for acetic acid and its compounds. Coxon. See XVII.

### PATENTS.

Distilling [carbonaceous matter]; Apparatus for —. W. P. Perry, South Woodford, Essex. Eng. Pat. 113,373, May 1, 1917. (Appl. No. 6140 of 1917.)

CARBONACEOUS material is distilled in a suitable vessel, the non-volatile portion being burnt in the lower section, giving off gas, which rises through the upper layers and drives off the volatile matter. This is collected by a series of pipes arranged at varying heights across and within the vessel, and having slots with upturned lips or conical openings on the underside. The slots or openings are at the centre of the vessel.—J. E. C.

Distilling combustible materials. C. Foxwell, Baltimore, Md. U.S. Pat. 1,255,593, Feb. 5, 1918. Date of appl., Feb. 23, 1917.

IN order to recover benzol and ammonia, combustible materials are treated with a solution of calcium chloride and borax and then distilled in a by-product oven.—J. E. C.

Vapour electric device [lamp]. D. McF. Moore, East Orange, N.J., Assignor to General Electric Co. U.S. Pat. 1,255,549, Feb. 5, 1918. Date of appl., June 16, 1915.

A GASEOUS conduction device, such as a gas electric lamp, comprises a tubular receptacle containing a column of rarefied gas, electrodes within the receptacle, and a source of constant current connected to the electrodes to produce an electrical luminous discharge through the rarefied gas. A material, which evolves gas when heated, is contained in a second receptacle, in communication with the first one but removed from the path of the discharge, and a heating coil, in a closed circuit arranged as a shunt to the main circuit, is embedded in the material evolving gas. The resistance of this shunt circuit is so adjusted that the current flowing therein will vary in such a way as to keep the density of the gas in the receptacle substantially constant.—B. N.

## III.—TAR AND TAR PRODUCTS.

Crude benzols; Comparative values of coke-oven —. B. W. Haigh and H. Lamb. Coke-Oven Managers' Assoc. Gas J., 1918, 141, 201—203.

THE authors have investigated the retort test for

crude benzol and have endeavoured to devise a satisfactory method whereby in working up low-grade crude benzols, the results may be expressed on the standard 65% basis. A series of samples were submitted to two methods of distillation. In the first, the apparatus consisted of a 250 c.c. retort with a shortened neck fitted into a Liebig condenser. The thermometer was graduated in half degrees from 90° to 200° C., the bulb being  $\frac{3}{8}$  in. from the bottom of the retort. Corrections for barometric pressure and exposed stem were applied to all readings, the latter being obtained from the formula  $C = 0.000154 (T - t)N$ , where  $T$  and  $t$  are respectively the observed temperature and the mean temperature of the exposed stem and  $N$  is the number of degrees in the exposed column. The rate of distillation was two drops per second and the interruption points 120° C. and 160° C. In the second method the retort was replaced by a 150 c.c. distillation flask, the bulb of the thermometer being placed just below the side tube. The thermometer was graduated to  $\frac{1}{2}^\circ$  from 70° to 130° C. The interruption points were 84°, 90°, 100°, 120°, and 130° C. The results showed that the retort test is trustworthy if carried out under uniform conditions and corrections applied to the thermometer readings; however, it has limitations in that it fails to show in what respect a particular benzol is abnormal and gives little insight into the relative proportions of benzene, toluene, and xylene in the sample. The flask test should therefore be adopted generally as supplementary to the retort test. For comparing crude benzols, samples were fractionally distilled to 200° C., using a Young's rod and disc column with twenty discs, the residue being a creosote oil of b.pt. about 205° C., and having a high naphthalene content. The residue was found to be larger the poorer the quality of the benzol, and from the results obtained, a graph was drawn whereby the values of low-grade benzols in terms of normal 65% benzol could be calculated for the particular plant from which the samples were obtained. Sufficient samples were not available from other works to draw any conclusions as to variations in quality of the products of other plants.—L. A. C.

Trichlorobenzenes; The three — and their reaction with sodium methoxide. A. F. Holleman. Rec. Trav. Chim. Pays-Bas, 1918, 37, 195—204.

THE three trichlorobenzenes when heated with sodium methoxide in a sealed tube at 180° C. reacted as follows:—1,2,3-trichlorobenzene gave 2,3- and 2,6-dichlorophenols with the corresponding anisols; 1,2,5-trichlorobenzene gave 2,5-dichlorophenol and a little 2,5-dichloroanisole; 1,3,5-trichlorobenzene yielded 3,5-dichloroanisole and a little 3,5-dichlorophenol. (See also J. Chem. Soc., May, 1918.)—W. G.

Nitro compounds; Reaction of aromatic —. S. C. J. Olivier. Rec. Trav. Chim. Pays-Bas, 1918, 37, 241—244.

THE twelve aromatic nitro compounds examined all gave an orange-red coloration with aluminium bromide in benzene solution, the colour disappearing on the addition of water. In the absence of a solvent, or on replacing the benzene by carbon bisulphide, the colour reaction was not obtained in most cases. (See also J. Chem. Soc., May, 1918.)—W. G.

Bromination of *p*-nitrotoluene. J. F. Brewster. J. Amer. Chem. Soc., 1918, 40, 406—407.

*p*-NITROBENZYL bromide, a useful agent for characterising phenols and carboxylic acids, is prepared by adding bromine gradually to a boiling solution of *p*-nitrotoluene in carbon tetrachloride, containing a crystal of iodine and exposed to sunlight.—J. C. W.



*Tolone chlorides from calcium carbide, chlorine, and benzene.* C. Davidson. J. Amer. Chem. Soc., 1918, 40, 397—400.

By adding calcium carbide, free from iron, to a saturated solution of chlorine in benzene, tolane di- and tetra-chlorides have been obtained. It is supposed that the primary dichloroacetylene condenses with benzene to form tolane, thus:  $\text{CCl} : \text{CCl} + 2\text{C}_6\text{H}_6 \rightarrow \text{C}_6\text{H}_5\text{C} : \text{C}\cdot\text{C}_6\text{H}_5 + 2\text{HCl}$ . (See also J. Chem. Soc., May, 1918.)—J. C. W.

*Water in gas tar.* Coudehou. See IIA.

*Limitations of the Kjeldahl method of determining nitrogen.* Brill and Agcaoili. See XXIII.

#### PATENTS.

*Benzol; Process of purifying crude* —. H. B. Kipper, Muskegon, Mich. U.S. Pat. 1,253,048, Jan. 8, 1918. Date of appl., Apr. 27, 1917.

CRUDE benzol is purified from carbon bisulphide by treatment in a finely divided form with a counter-current of a solution containing caustic soda and cellulose.—F. W. A.

[*Dimethylphenylbenzyl ammonium zinc chloride; Dimethylaniline, benzyl chloride, and zinc chloride; Manufacture of a difficultly soluble complex compound from* —. F. B. Dehn, London. From Chemical Works Rohner & Co., Pratteln, Switzerland. Eng. Pat. 104,676, Feb. 7, 1917. (Appl. No. 1905 of 1917.)

BENZYL chloride and dimethylaniline in molecular proportions are mixed with an aqueous solution of zinc chloride containing a slight excess above half a molecular proportion of zinc chloride; after prolonged stirring the separation of the difficultly soluble crystals of the double compound of zinc chloride and dimethylphenylbenzyl ammonium chloride is complete. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 24,695 of 1914; this J., 1916, 248.)—F. W. A.

*$\beta$ -Naphthol benzoate; Process of making* —. G. Bieberger, Assignor to Anthony-Hammond Chemical Works, Inc., New York. U.S. Pat. 1,254,970, Jan. 29, 1918. Date of appl., Dec. 5, 1916.

To produce  $\beta$ -naphthyl benzoate, benzoyl chloride is allowed to drop on to 110 grms. of heated  $\beta$ -naphthol with stirring until about 170 grms. has been added, any evaporated benzoyl chloride being condensed and returned, and the hydrogen chloride allowed to escape.—F. W. A.

*Beta aminoanthraquinone; Process for the manufacture of* —. P. Dutoit, Monthey, Assignor to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,255,719, Feb. 5, 1918. Date of appl., Apr. 26, 1917.

$\beta$ -ANTHRAQUINONEMONOSULPHONIC acid is heated with ammonia and an aromatic nitro-compound, yielding  $\beta$ -aminoanthraquinone and an aromatic amino-compound.—L. A. C.

#### IV.—COLOURING MATTERS AND DYES.

*1,4,6-Trihydroxyanthraquinone.* M. L. Crossley. J. Amer. Chem. Soc., 1918, 40, 404—406.

WHEN 4-aminophthalic anhydride is heated with quinol and concentrated sulphuric acid at 170°—190° C., the chief product is 1,4,6-trihydroxyanthraquinone. This has no particular value as a mordant dye. (See also J. Chem. Soc., May, 1918.)—J. C. W.

*Phthalic acid derivatives. Constitution and colour.* XII. Tetrachlorophthaloxime and some of its derivatives. D. S. Pratt and C. H. Miller. XIII. Tetra-iodophthaloxime and some of its derivatives. D. S. Pratt and T. B. Downey. J. Amer. Chem. Soc., 1918, 40, 407—412, 412—415.

THE oximes mentioned are prepared by the action of hydroxylamine salts on the substituted phthalic anhydrides in the presence of alkali carbonates, and their ethers and esters through their silver salts. They differ from unsubstituted phthaloxime in occurring in only one colour, this forming two chromo-isomerides. Some of the tetrachlorophthaloxime ethers,  $\text{C}_6\text{Cl}_4\langle\text{CO}\text{---}\text{C}(\text{N}\cdot\text{OR})\rangle\text{O}$

are unique in existing in two interchangeable crystalline forms, cotton-like masses, and stable, stout prisms. (See also J. Chem. Soc., May, 1918.)—J. C. W.

#### PATENTS.

*Azo dyestuffs; Manufacture of new copper compounds of substantive* —. O. Inray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 113,141, Feb. 8, 1917. (Appl. No. 1950 of 1917.)

A SOLUTION of a direct azo dyestuff obtained by combining 1 mol. of a tetrazo compound of a *para*-diamine derived from the diphenyl nucleus and having in *ortho*-position to an amino group at least one alkyloxy group, with 1 mol. of 2-amino-5-naphthol-7-sulphonic acid or an alkyl, aryl, or acetyl derivative and 1 mol. of a 1,8-amino-naphtholsulphonic acid or a derivative thereof, or with 2 mols. of 2-amino-5-naphthol-7-sulphonic acid or a derivative thereof, is treated with copper or a copper compound, and the soluble copper compound of the dyestuff is isolated. In addition to dyeing cotton direct shades which, without any after-treatment, are of excellent fastness to light, alkali, and washing, the new copper compounds dye like substantive dyestuffs, wool, silk, mixed fabrics, straw, wood, paper, leather, and the like, and may, moreover, be used in printing and for making lakes fast to light.—F. W. A.

[*Azo*] *colouring matter; Manufacture of a* —. E. F. and H. W. Ehrhardt, Birmingham. Eng. Pat. 113,181, Apr. 5, 1917. (Appl. No. 4939 of 1917.)

DIAZOTISED picramic acid is combined with pure *p*-cresol or with the solution resulting from the fractional combination of diazotised picramic acid with a mixture of cresols, when the *o*-cresol couples first to give Metachrome Brown Y. The new *ortho*-azo dyestuff gives a chocolate-brown shade on unmordanted wool, and on chrome mordant gives an olive-brown shade, fast to milling and to light.—F. W. A.

*Disazo colouring matter; Production of a mixed* —. E. F. and H. W. Ehrhardt, Birmingham. Eng. Pat. 113,195, Apr. 30, 1917. (Appl. No. 6117 of 1917.)

DIAZOTISED *p*-phenylenediamine-azo-salicylic acid is combined with  $\beta$ -naphthylamine free from impurities soluble in or destroyed by caustic soda. The product is insoluble in water, but if ground to a uniform gelatinous mass forms a colloidal solution which may be used for dyeing unmordanted cotton pink to red shades or for dyeing wool by the metachrome process bright red shades.—F. W. A.

*N-Dihydro-1,2,2',1'-anthraquinoneazine* [Indanthrene]; *Manufacture of* —. British Dyes, Ltd., J. Turner, and D. Segaller, Huddersfield. Eng. Pat. 113,489, Feb. 26, 1917. (Appl. No. 2776 of 1917.)

CRUDE N-dihydro-1,2,2',1'-anthraquinoneazine

(Indanthrene) is treated with oxidising agents (of which the following salts are suitable: sodium, potassium, calcium, strontium, and barium salts of hypochlorous, chloric, permanganic, and persulphuric acids) in aqueous solution and in such proportion and concentration that the yellow colouring matter present as an impurity is oxidised, the Indanthrene itself not being attacked. For example, one part by weight of crude 10% N-dihydro-1,2,2',1'-anthraquinoneazine paste is boiled with four parts of sodium hypochlorite solution (sp. gr. 1.15) for six hours with vigorous stirring. After cooling, the blue dyestuff is filtered off and washed free from sodium hypochlorite.—L. A. C.

*Yellow colouring matter [tetrachlorotartrazine]; Process for the production of*—L. B. Holliday and A. Clayton, Huddersfield. Eng. Pat. 113,510, Mar. 7, 1917. (Appl. No. 3372 of 1917.)

Two mols. of 2,5-dichlorophenylhydrazine-4-sulphonic acid or its salts react with one mol. of dihydroxytartaric acid or its salts, in either acid or neutral medium, to give a colouring matter which dyes wool and silk an intense yellow, and is assumed to be identical with tetrachlorotartrazine of Eng. Pat. 3373 of 1908 (see Fr. Pat. 387,245 of 1908; this J., 1908, 801). Example: 518 lb. of 2,5-dichlorophenylhydrazine-4-sulphonic acid is intimately mixed with 1500 lb. of water containing 80 lb. of sodium hydroxide. 190 lb. of sodium dihydroxytartrate is dissolved in a mixture of 300 lb. of water and 380 lb. of hydrochloric acid (sp. gr. 1.16). The first solution is added slowly to the second and the two thoroughly mixed. The temperature is then raised to about 80° C., and maintained at that point for half an hour or more. The dyestuff settles out and is filtered off.—L. A. C.

*Vegetable dye, and method of preparing same*. I. Kitsec, Philadelphia, Pa. U.S. Pat. 1,254,914, Jan. 29, 1918. Date of appl., Nov. 26, 1915.

Fast dyes are produced from a "vegetable base" by addition of zinc chloride and ammonium copper chloride.—F. W. A.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### PATENTS.

*Fireproof composition [for fabrics]*. J. Truro, Redditch. Eng. Pat. 113,162, Mar. 5, 1917. (Appl. No. 3195 of 1917.)

A FIREPROOF composition consists of substantially 25% of ammonium phosphate, 50% of ammonium chloride, and 25% of alum made up in liquid, powder, or tablet form, vinegar being added in case the solution is to be applied to fabric, to prevent it from affecting the dye.—F. W. A.

*Cellulose; Digesters for use in the manufacture of—and like operations*. E. Schaufelberger, London. Eng. Pat. 113,491, Feb. 28, 1917. (Appl. No. 2982 of 1917.)

A CELLULOSE digester is constructed with an external system comprising a heater, a circulating device, and one or more separators to purify the liquid during circulation. In the case of a vertical digester the liquid is preferably caused to enter at the bottom and is forced upwards through the material. An arrangement may be provided in the form of a float attached to a flexible connection, whereby the outlet pipe at the top of the digester descends as the level of the liquid falls during the course of the digestion. The separator may consist of a vessel into which the liquid enters through the bottom by way of a projecting bell-mouthed orifice, which reduces the velocity

of the flow, whereby the floating impurities are collected in the upper part of the vessel while the heavy impurities settle at the bottom and the liquid passes on into the circulating system through a strainer situated in the vessel.—J. F. B.

*Paper-makers' [mechanical] pulp; Manufacture of*—W. Fennell, Wotton, S. D. Lane and W. O. Garbutt, Gloucester. Eng. Pat. 113,624, July 15, 1916. (Appl. No. 9963 of 1916.)

*Dryer [for fibrous materials]*. U.S. Pat. 1,254,073. See 1.

*Machine for impregnating fabrics with rubber*. U.S. Pat. 1,255,192. See XIV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Cotton yarn; Experiments on the mercerisation of—by caustic soda lye*. T. Nakata, Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1224—1231.

TWO-PLY 60's cotton yarn was boiled out in dilute caustic soda and soap solution and portions were mercerised without previous drying. Samples from which the mercerising soda was merely removed by squeezing had only 66% of the tensile strength of samples similarly treated and thoroughly purified by washing and drying. When moist yarn was mercerised in caustic soda of 53° Tw. (sp. gr. 1.265) without tension at 25° C., maximum shrinkage (18.61%) was recorded in the case of yarn containing 75% of additional moisture calculated on the weight of the dry, boiled yarn. Under these conditions of moisture the mercerisation process exerts its maximum effect. The strength and elongation of mercerised yarns are important factors in their textile value. These depend largely on the amount of stretching to which the yarn is subjected during mercerisation. The elongation of the finished yarn in tensile tests increases largely with the shrinkage allowed and decreases if the mercerised yarn is stretched beyond its original dimensions. No definite rule can be stated as the results are greatly influenced by the count and ply of the yarn. A 2-ply 60's yarn with an original hank-circumference of 54 ins., and 52.44 ins. after boiling, can be mercerised under tension at 30° C. in a lye of 50° Tw. (sp. gr. 1.25) so as to give a length of 54—54.5 ins. without loss of elasticity and with an increase of about 20% in tensile strength as compared with the original.—J. F. B.

### PATENTS.

*Bleaching and like treatment of cotton, cotton waste, wool, and similar materials; Means for circulating bleaching and other liquors in the—*. T. Taylor, Stockport. Eng. Pat. 112,969, June 26, 1917. (Appl. No. 1323 of 1917.)

IN the steam ejector employed, additional steam is admitted into the delivery nozzle to heat the induced fluid, giving simultaneous circulation and heating of the bleaching or other liquor. (Reference is directed, in pursuance of Sect. 7 Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 2869 of 1883, 15,174 of 1893, and 5744 of 1915.) —F. W. A.

*Textile fibres; Machines for dyeing and finishing—*. F. F. Larivei, Bradford. Eng. Pats. (A) 113,453 and (B) 113,451, June 7, 1916. (Appl. Nos. 8050 and 8051 of 1916.)

(A) A MACHINE adapted for experimental work is



constructed with rollers for supporting hanks or bunches of textile fibre over a dye vessel or vat which is divided into a number of separate and independent casings each corresponding to a particular roller. The whole vat is mounted on gear to be lifted or lowered relatively to the rollers, or the rollers may be moved relatively to the vat. Each separate chamber of the vat has its own independent inlet and outlet and separately adjustable heating arrangement, and means may be provided whereby the speed of the rollers may be adjusted independently of each other. (B) A machine of the same general type as the foregoing is constructed with two or more vats which can be controlled independently, each provided with several rollers which may be driven either independently or together.—J. F. B.

[Aniline] *Black upon vegetable fibres; Production of* —. The Calico Printers' Association, Ltd., and E. A. Fourneaux, Manchester. Eng. Pat. 113,484, Feb. 28, 1917. (Appl. No. 2610 of 1917.)

THE process described in Eng. Pat. 7345 of 1908 (this J., 1909, 701) is modified in that the use of aluminium chloride is discontinued and the proportion of *p*-phenylenediamine is reduced to  $\frac{2}{10}$ — $\frac{1}{10}$  of the weight of aniline employed. The presence of a certain amount of formic acid is necessary. When organic acids only are used, the proportion of formic acid should not be less than 2 mols. and may be as high as 4 mols. per mol. of aniline. The organic acids may be partly replaced by mineral acids to an extent not exceeding 0.3 mol. per mol. of aniline. When mineral acids are employed as well as organic acids, the proportion of formic acid should not fall below 1 mol. and may be as high as 2.5 mols. per mol. of aniline. The formic acid may be replaced by acetic, lactic, or glycollic acid to an extent not exceeding one-fifth. When organic acids only are used, formic acid should be employed in conjunction with tartaric acid. The proportion of copper salt employed should be between  $\frac{1}{15}$  and  $\frac{1}{8}$  mol. per mol. of aniline. Example: Aniline oil, 63.5; 90% formic acid, 125; tartaric acid, 18.25; *p*-phenylenediamine dihydrochloride, 5.5; sodium chlorate, 33.75; copper chloride, 13, made up to 1000 parts by weight. The copper chloride is added immediately before use.—J. F. B.

*Yarns in the form of hanks, skeins, or bundles; Method of and machines for dyeing or similarly treating* —. J. Annicq, Manchester. Eng. Pat. 113,575, Aug. 31, 1917. (Appl. No. 12,511 of 1917.)

THE dry yarn is packed into a rectangular tank with perforated side walls and perforated central chamber, the threads being laid horizontally in approximately the same direction as the flow of the liquor; pressure is then applied, *e.g.*, not less than 20 lb. per sq. in., sufficient to expel as far as possible the air from the packed bundles before the liquid is introduced, such pressure being controllable by the amount of dry yarn packed into the tank, which is fitted with extension plates, and by means of suitable pressure devices applied to the lid before it is screwed down. For instance, the yarn is piled horizontally in the vat to a height which is above the normal position of the lid. The lid is placed on the top of the pile and is guided by extension plates held in position between the packed yarn and the ends of the tank and passing through slots in the lid. The lid is forced down by screw clamps, compressing the yarn, until it can be bolted down; the extension plates are then removed and the slots covered, and liquor is circulated through the perforated sides of the chambers by means of a pump.—J. F. B.

*Fibres; Process and apparatus for treating* —. H. R. Anders, Perth Amboy, N.Y., Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,252,986, Jan. 8, 1918. Date of appl., Dec. 21, 1914.

A ROTARY container with encased heating means near the centre is divided by partitions into a number of chambers preferably of substantially equal size, connected by a channel encasing the heating means, in order to heat the liquid. The chambers are completely packed with the fibres wetted-out with the liquid with which they are to be treated. Only one of the chambers is filled with liquid, which passes through the chambers in diagonally opposite directions and is filtered consecutively through the goods in each chamber when the container is rotated.—F. W. A.

*Dyeing apparatus*. D. M. Hey, Assignor to Vacuum Dyeing Machine Co., Chattanooga, Tenn. U.S. Pat. 1,254,006, Jan. 15, 1918. Date of appl., Dec. 13, 1915.

A DYEING apparatus consists of a vat connected at its lower portion and at the top (by a spray head above the dye liquor) with the discharge side of a pump which is connected at the suction side by means of an elbow-jointed pipe (with air-inlet in elbow) to a heating chamber into which passes the overflow from the vat. Valves are arranged so that when in one position the dye liquor circulates through the heating chamber, entering the lower part of the vat and leaving at the upper part, and when in another position the circulating path does not include the heating chamber, the liquor leaving the lower portion of the vat and entering through the spray head in the upper portion.—F. W. A.

*Printing fibrous substances; Machine for* —. A. Amrein, Amsterdam, N.Y. U.S. Pat. 1,252,985, Jan. 8, 1918. Date of appl., May 9, 1917.

THE face of a rotary printing drum is made up of a series of platen and pattern index faces, index tongues of determined colour values being arranged on the index faces in accordance with a predetermined pattern. The printing rolls are engaged by the index tongues of corresponding colour values, and have their axes in constant spaced relation with the axis of the printing drum. Means are provided for transferring colour to the printing rolls, and for rendering certain of the indexes inoperative to actuate predetermined printing rolls.—F. W. A.

*Printing fabrics; Process of* —. G. Prifold, Linwood, Pa., Assignor to The Congoleum Co., Philadelphia, Pa. U.S. Pat. 1,255,049, Jan. 29, 1918. Date of appl., Apr. 24, 1917.

WOVEN fabrics are printed with a mixture of an aniline colour capable of dyeing the fabric and a starchy vehicle capable of being dried and dusted out of the fabric, and are then steamed to cause the colouring matter to enter the body of the fabric, dried, brushed to remove the vehicle from the coloured fabric, and finally sized on the back.

—F. W. A.

*Manufacture of a difficultly soluble complex compound* [dimethylphenylbenzylammonium zinc chloride] from dimethylaniline, benzyl chloride, and zinc chloride. Eng. Pat. 104,676. See III.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphur in pyrites; Determination of* —. Z. Karaoglanow, and P. and M. Dimitrow. Z. anal. Chem., 1917, 56, 561—568.

ABOUT 0.5 gm. of the finely powdered sample is oxidised with nitric and hydrochloric acids, or is

fused with a mixture of sodium carbonate and potassium nitrate, and after separation of silica in the usual way, the sulphuric acid is precipitated directly from the hydrochloric acid solution without previously removing the iron. The solution, which should contain from 30 to 50 c.c. of 6*N* hydrochloric acid, is diluted to 700 c.c., heated to boiling, and 40 c.c. of 10% barium chloride solution diluted with 100 c.c. of boiling water is added slowly while the mixture is stirred. After 15 hours, the barium sulphate is collected, washed with water containing hydrochloric acid and barium chloride, then with hot water, dried, ignited, and weighed. Under these conditions, the presence of ferric chloride does not affect the accuracy of the results obtained.—W. P. S.

*Chlorates and hypochlorites; Determination of* —. E. Rupp. *Z. anal. Chem.*, 1917, 56, 580—586.

A SUITABLE quantity, e.g., 10 c.c. of a solution containing about 0.5% of potassium chlorate and calcium hypochlorite, is diluted to 100 c.c. in a large stoppered flask, 2 grms. of potassium iodide is added, the solution is acidified with dilute acetic acid and, after 5 mins., titrated with thiosulphate solution; this titration gives the amount of hypochlorite. Another portion of 10 c.c. of the solution is treated in a stoppered flask with 1 gm. of potassium bromide and 30 c.c. of concentrated hydrochloric acid; after 15 mins., 15.0 c.c. of 1% potassium iodide solution is added, the mixture is shaken, and titrated with thiosulphate solution. The difference between the two titrations is a measure of the amount of chlorate present.—W. P. S.

*Hypobromite and bromate, or hypoiodite and iodate; Determination of* — in mixtures. E. Rupp. *Z. anal. Chem.*, 1918, 57, 16—19.

THE solution containing a hypobromite and bromate, or a hypoiodite and iodate, is treated with hydrogen peroxide and sodium hydroxide solution, the reaction proceeding according to the equation:  $\text{NaBrO} + \text{H}_2\text{O}_2 = \text{NaBr} + \text{H}_2\text{O} + \text{O}_2$ . The excess of hydrogen peroxide is then destroyed by boiling the mixture, potassium iodide and sulphuric acid are added, and the liberated iodine is titrated with thiosulphate solution. Another portion of the original solution is treated directly with potassium iodide and sulphuric acid and then titrated with thiosulphate solution. The difference between the two titrations gives the quantity of hypobromite present, whilst the second titration gives the hypobromite and bromate together.

—W. P. S.

*Iodates; Determination of* — in the presence of bromates. E. Rupp. *Z. anal. Chem.*, 1918, 57, 19—21.

BROMATES are converted gradually by dilute hydrochloric acid into hydrobromic acid and hypochlorous acid is formed at the same time, but iodates are not affected. An aliquot portion of a solution containing an iodate and bromate is treated with potassium iodide and sulphuric acid, and, after a few minutes, the mixture is titrated with thiosulphate solution; this gives a measure of the iodate and bromate together. Another portion of the solution is diluted to 50 c.c., treated with 20 c.c. of 12.5% hydrochloric acid, placed aside for 1 hour, 25 c.c. of 3% hydrogen peroxide and 15 c.c. of 15% sodium hydroxide solution are then added, the mixture is boiled for 10 mins., cooled, potassium iodide and sulphuric acid are added, and the liberated iodine is titrated with thiosulphate solution. The difference between the two titrations corresponds with the amount of bromate present.—W. P. S.

*Sodium manganate; Manufacture of crude* —. F. Wartenweiler. *J. Chem., Met., Min. Soc. S. Africa*, 1918, 18, 161—162.

ON account of the scarcity of permanganate, the manufacture of crude sodium manganate was attempted in a four-hearth muffled calcining-furnace used for roasting zinc-gold precipitate. One part of finely powdered ferruginous pyrolusite (Mn 40%) was heated for 2 hours to 650°—750° C. with 1.44 part of caustic soda; the product assayed 18.2%  $\text{Na}_2\text{MnO}_4$ . By introducing the charge into the furnace heated to 450° C., slowly raising the heat to 560° C., and maintaining it for 8 hours, then cooling for 4 hours, and withdrawing the charge, the grade of the product rose to 28%  $\text{Na}_2\text{MnO}_4$ . The latter method has been adopted for supplying the mines.—W. R. S.

*Prussian blue; Retardation of the formation of* — and other reactions in aluminium hydroxide sols. J. Reitsstötter. *Kolloid Zeits.*, 1917, 21, 197—200.

THE reaction between potassium ferrocyanide and ferric chloride is greatly retarded if the ferrocyanide solution is added to a colloidal solution of aluminium hydroxide with the subsequent addition of the ferric chloride to the coagulated system. Similar retardation effects are obtained with other pairs of substances and experiments are described which seem to offer an explanation of the phenomenon. (See also *J. Chem. Soc.*, Apr., 1918.)—H. M. D.

*Dolomite; Decalcification of* —. J. W. Mellor. *Trans. Ceram. Soc.*, 1917—1918, 17, 119.

THERE is no difficulty in separating the two main components of dolomite by chemical processes, but these are too costly to enable the product to compete with native magnesite. Separation may possibly be effected by a process based on the difference in the densities of the two constituents after calcination at high temperatures, or on the greater inertness of the magnesite when the calcined mass is exposed to water. The separation of the lime by washing the calcined product with water was patented in 1907 by Auzies and Segoffin (*Fr. Pat.* 374,216; this *J.*, 1907, 761).—A. B. S.

*Antimony dioxide*. J. von Szilagy. *Z. anal. Chem.*, 1918, 57, 23—28.

ANTIMONY dioxide may be obtained by heating the pentoxide at bright redness, or by oxidising antimony with concentrated nitric acid and heating the resulting oxide at dull redness. Antimony dioxide dissolves in a hydrochloric acid solution of potassium iodide, an equivalent quantity of iodine being liberated:  $2\text{SbO}_2 + 6\text{HCl} + 2\text{HI} = 2\text{SbCl}_3 + 4\text{H}_2\text{O} + \text{I}_2$ . The iodine may be separated by distillation, collected in potassium iodide solution, and titrated. Although metastannic acid does not liberate iodine under these conditions, the reaction is of little use for the determination of antimony in the presence of tin, owing to the interfering action of even small quantities of other metals usually present in antimony-tin alloys.—W. P. S.

*Radioactive substances; Colloidal state of* —. H. Zachs. *Kolloid Zeits.*, 1917, 21, 165—176.

SOLUTIONS of certain radioactive substances show the characteristic properties of colloids, and these properties have been examined in the case of radium A, B, and C in different solvents. (See also *J. Chem. Soc.*, Apr., 1918.)—H. M. D.

*Rate of hydration of calcined dolomite*. Edwards and Rigby. *See IX.*

*Formation of zinc ferrite*. Hamilton and others. *See X.*



*Depolarisation by sulphur dioxide.* Edwards. See XI.

*The sugar factory as a source of supply for acetic acid and its compounds.* Coxon. See XVII.

*Preparation, properties, and analysis of "white precipitate."* Kolthoff. See XX.

*Determination of phosphoric acid. Modification of the citrate method.* Grossfeld. See XXIII.

*Detection and determination of small quantities of hydrocyanic acid.* Kolthoff. See XXIII.

*Titration of chlorides by Volhard's method.* Kolthoff. See XXIII.

#### PATENTS.

*Bleaching powder; Manufacture of* ——. Castner-Kellner Alkali Co., Ltd., London, and J. W. Moore, Runcorn. Eng. Pat. 113,457, Nov. 23, 1916. (Appl. No. 16,822 of 1916.)

A CONTINUOUS stream of lime is carried, by gravity, through an inclined, rotary cylinder about 80 ft. long and 20 ins. wide, against a counter-current of chlorine. The flow of gas is maintained, and leakage prevented, by means of suction applied at the upper end of the cylinder. —W. E. F. P.

*Tungstates; Process of obtaining* ——. J. B. Ekeley and W. B. Stoddard, Boulder, Colo. U.S. Pat. 1,255,144, Feb. 5, 1918. Date of appl., June 28, 1916.

TUNGSTEN ores are ground and fused in a bath containing sodium chloride or other halide of an alkali metal, sodium carbonate or other alkali, and silica or sodium nitrate. —C. A. M.

*Lime; Method of slaking* ——. Apparatus for hydrating lime. A. Zimmerman, Assignor to L. M. Palmer, Brooklyn, N.Y. U.S. Pats. 1,255,268 and 1,255,682, Feb. 5, 1918. Dates of appl., Aug. 18, 1916, and Feb. 12, 1915.

In a continuous process, quicklime and a large excess of water are fed into the uppermost of a series of horizontal, cylindrical, rotary containers, maintained at the proper slaking temperature by external heating. The mixture is caused to traverse each cylinder in turn against a counter-current of air, so that the product is completely dried before being discharged from the apparatus. —W. E. F. P.

*Nitre-cake; Method of recovering from* ——. Products useful in the arts. L. T. Sherwood, Connellsville, Pa. U.S. Pat. 1,255,474, Feb. 5, 1918. Date of appl., Feb. 8, 1917.

THE heat of combustion of sulphur is utilised to convert nitre-cake into normal sodium sulphate and sulphur trioxide. The former product is recovered as such, and the latter, with the sulphur gases, as sulphuric acid. —W. E. F. P.

*Alumina; Purification of* ——. J. W. Hornsey, Summit, N.J., Assignor to Mineral Products Corporation, New York. U.S. Pat. 1,255,749, Feb. 5, 1918. Date of appl., Aug. 11, 1917.

IMPURE alumina, obtained by calcining alundum and leaching the product, is mixed with water to form a pulp which, after the addition of oil, is subjected to froth flotation; pure alumina is recovered from the froth. —W. E. F. P.

*Phosphorus [from basic slag]; Manufacture of* ——. H. O. H. Wennam, Bishop's Castle, Salop. Eng. Pat. 113,584, Nov. 2, 1917. (Appl. No. 15,982 of 1917.)

MOLTEN basic slag is charged direct from a steel furnace into an electric furnace; carbon and silica are then added and the mixture heated. —W. E. F. P.

*Electrochemical process for the treatment of metallic ores.* Eng. Pat. 113,508. See X.

*Method and apparatus for treating liquid [to produce colloidal tungstic oxide] for curative and therapeutic purposes.* Eng. Pat. 113,505. See XX.

#### VIII.—GLASS; CERAMICS.

*Silica bricks; Inversions in* ——. A. Scott. Trans. Ceram. Soc., 1917—1918, 17, 137—152.

SECTIONS of unused silica bricks and those made at the same time, but subsequently subjected in the crown and other parts of furnaces, to temperatures between 1000° C. and 1600° C., were examined by customary micro-petrological methods. In the kiln-fired condition and ready for use, silica bricks are composed of fragments of crystalline quartz cemented by a glass rich in calcium silicates. The greater part of the quartz is unaltered, but there has usually been some reaction between the bond and the smaller quartz grains, resulting in the formation of a glass and partial reprecipitation of silica as cristobalite. In no British bricks was there so much conversion into cristobalite and tridymite as found by Seaver (this J., 1915, 1209) in American bricks. Bricks which have been used, even in the cooler parts of a furnace, show much more conversion. The silica of the matrix is usually converted completely to a little cristobalite and much tridymite; some of the quartz is converted, but the centres of the fragments remained unaltered. The alteration is greatest in those fragments which are in contact with the bond. Bricks which have undergone very intense heating when in use generally show a very high proportion of tridymite, the original quartz and cristobalite having disappeared. Some sections show nothing but aggregates of minute overlapping hexagonal plates of tridymite enclosed in a network of interstitial glassy material, each aggregate representing an original crystal of quartz. When cristobalite occurs in these tridymite-bricks it forms well-developed skeletons in the glassy bond between the original rock-fragments. In other bricks composed largely of tridymite, the latter is of the simple twinned form and fairly large crystals occasionally develop. Pseudo-hexagonal tridymite never occurs in bricks which have proved bad in practice, but the simple twinned form is common in such bricks. Repeated heating at temperatures varying from cones 13 to 16 (1380° to 1460° C.) and prolonged heating at temperatures varying between 1500° and 1600° C. both yield tridymite as the end-product. The author supports Le Chatelier (this J., 1917, 961) and disagrees with Fenner (this J., 1913, 22); he is of the opinion that tridymite is stable at temperatures above 1400° C. When cristobalite occurs in bricks which have been heated for long periods to 1600° C., it always occurs in the matrix or along the margins of tridymite grains and has probably been formed by partial solution of the tridymite in the matrix (which is molten at 1500°—1600° C.) and reprecipitation as the brick cools. Quartz is probably stable up to 870° C., tridymite up to 1570° C. (its melting point), but any dissolved silica which is reprecipitated as cristobalite may invert to either quartz or tridymite according to the temperature. Cristobalite appears to have no stable range below 1600° C. The nature of the bond used in the manufacture of silica bricks may affect the rate of inversion. It is desirable to convert as much of the quartz as possible into tridymite, because this is the most stable form at high temperatures and because the volume-change from  $\alpha$ - to  $\beta$ -tridymite is less than that from  $\alpha$ - to  $\beta$ -cristobalite. The changes in the volume

of bricks are often much greater than would be expected from a consideration of the changes from one form of silica to another. Thus, instead of the theoretical increase of 13 to 16%, the author has observed an increase of over 30%. The excess is probably due to the newly-formed crystals not being oriented so as to occupy the minimum space. W. D. Ross (Trans. Amer. Ceram. Soc., 1917, 19, 83—93) has found that on heating a silica brick to 1500° C. the average volume expansion was 9.7%, but the reduction in sp.gr. only 2.8%. The pseudohexagonal tridymite was found in bricks with a low expansion in use. The occurrence of this type of volume increase is an additional reason for carrying the conversion as near completion as possible in the manufacture of the bricks.—A. B. S.

*Silica bricks; Estimation of proportions of quartz, tridymite, etc., in —.* A. Scott. Trans. Ceram. Soc., 1917—1918, 17, 188—191.

Owing to the impurities present, the determination of the sp.gr. of silica bricks only gives a very rough idea of the proportion of the various forms of silica present. The same is true of the separation of the constituents by means of dense liquids. Sorby's method of measuring the area of the various crystals when seen under the microscope is accurate, but tedious. Rosiwal has improved it by measuring the intercepts which the particles of each constituent make with a number of arbitrary lines, but the method is difficult to apply to the determination of more than one constituent of silica bricks owing to the fineness of the grains of cristobalite and tridymite; it, however, gives excellent results in about an hour, in estimating the percentage of unaltered quartz, with an error of less than 1%. Details of the method of measurement are given. It is desirable to measure intercepts totalling about 1500 units.—A. B. S.

*Chemical stoneware; Standardisation of —.* H. Nielsen and J. R. Garrow. Chem. Trade J., 1918, 62, 157—158, 179—180, 225—227. (See this J., 1918, 57 A.)

STONEWARE pressure vessels (montejus, acid-eggs, and vacuum receivers) should as far as possible be spherical, flattened curves being avoided. A maximum wall-thickness of 2½ ins. is suggested, several small vessels being preferred to one larger one. Such vessels should be sunk in wooden boxes filled with sand. An armour of cast iron, steel, or lead should be avoided, its use giving rise to a false sense of security. For a spherical vessel,

the theoretical wall-thickness required is  $W = \frac{dp}{2U}$ ,

where  $W$  = the wall-thickness in ins.,  $d$  = the internal diameter in ins.,  $p$  = the pressure in lb. per sq. in., and  $U$  = the tensile strength of the material. A margin of safety of 2 is regarded as sufficient, i.e., the actual wall-thickness should be twice the theoretical value. Illustrations showing the relative dimensions of the various parts are given. For large montejus, egg-ended cylinders of the same internal diameter and over-all height as the spherical ones, but with double the capacity, are suggested. The use of thin necks for liquid inlets and outlets on heavy vessels is condemned, a square boss into which is ground a standard pipe with a standard taper being preferable. In the boss are two longitudinal holes; a bolt is passed through each of these holes and these bolts serve to keep in position four eye-bolts passing through the bolt holes in the iron armature of the vessel. Maximum sizes for bosses used for montejus are suggested. Storage and transport jars should not exceed 2 tons capacity. Suggestions are made as to the thickness of the walls and other dimensions. The bottoms of the jars should be slightly arched inwards. Outlets should be the same as for

montejus. To prevent the vessels from sweating, they may be filled with a strong solution of sodium silicate, and after a week or more the solution discharged and the vessel treated with strong acid. The diameter of cylindrical jars should be chosen so that the dies used for making large stoneware pipes or towers may be used. Socketed pipes are suitable for fumes or gases under low pressures, but not for liquids. The pipes used for corrosive fumes should be of the best stoneware, vitrified throughout, with thin walls. The jointing material should be soft and should always remain semi-plastic to facilitate alterations and repairs. The socket should be provided with an asbestos ring slightly less in diameter than the inside diameter of the socket and of slightly larger bore than the pipe; this ring acts as a cushion and forms a good base for the cement. When very hot gases are to be conveyed, care must be taken to prevent the pipes being subjected to sudden changes in temperature. Water-cooling should not be applied to pipes at a temperature above 110° C. Suggested dimensions for standard sizes of pipes and bends are given. Condensing and reaction towers should be of the best material, vitrified as fully as possible and salt-glazed. All fume inlet and outlet sockets should be of ample size and should not impede draught. The bottom of the lowest piece in a tower should be arched inwards. Filling-material should not rest on the arched bottom but should be carried by the sides and by a perforated plate (with square holes) in each section. Each section should be supported by a wooden framework. The liquor outlet should be of the boss type as described for "montejus." For towers of large diameter the perforated plates should be in three parts. The plates should always rest on a thick asbestos cord and not directly of the supporting ledges. The perforations should be so arranged that any cross section along a diameter of the plate will show a sufficient proportion of solid material to provide the requisite strength for carrying the filling material. An average of 50% of perforations in plates up to 3 ft. diameter is suggested, with a smaller percentage for larger sizes. The covers of towers should be domed and should contain the liquor inlet and fume outlet. The expression "stoneware" should be restricted to dense, acid-resisting material used in chemical plant, the term "earthenware" being used for more porous bodies. Stoneware may be regarded as a form of porcelain and should resemble the latter in texture, vitrification, non-porosity, independence of glaze (except for appearance), resistance to chemical corrosion and changes of temperature. No single natural clay will produce an ideal chemical stoneware, but many British clays can be satisfactorily blended with other materials. Glazed fireclay should not be used for chemical stoneware except under scientific control, as its body is essentially porous and hence too much reliance must be placed on the glaze. Economy in manufacture should be sought in a proper arrangement of the kilns and in protecting them from the weather.—A. B. S.

*Weathering-products of bricks.* S. Nagaya. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1183—1204.

THE needle-like crystals which form an efflorescence on bricks are due to sodium sulphate, sodium carbonate, and calcium sulphate. The alkali is derived from the clay of which the bricks are made, the sulphur trioxide is chiefly absorbed from the gases used to heat and afterwards to cool the bricks during the process of burning, and the carbon dioxide is absorbed from the atmosphere. As the growth of the crystals accelerates the d composition of the bricks, they should be washed off with water or dilute hydrochloric acid.



The author advises an examination of the clay with regard to its fineness and its content of soluble salts, sulphur trioxide, and sulphur; observation of the growth of the weathering products during the drying of moulded brick; determination of the porosity of the bricks; determination of the sulphur-content of the fuel; the use of a reducing as well as an oxidising atmosphere in burning the bricks; and the avoidance of sulphur dioxide in gases in contact with cooling bricks.—A. B. S.

*Bone china body; Study of —. Part I. Colour.* W. H. Yates and H. Ellam. Trans. Ceram. Soc., 1917—1918, 17, 120—122.

BODIES composed of china clay, bone ash, and Cornish stone in various proportions were cast into cups or pressed into slabs, and fired at Seger cone 9 in a china biscuit oven. The colours of the products varied from very pale cream to yellow and reddish-brown and a deep greenish-blue. Mixtures of china clay and bone ash (without stone) yielded brown colours, but a mixture containing 40% of clay and 60% of bone gave a bluish-green colour. Mixtures of china clay and Cornish stone gave yellowish-brown colours. Mixtures of bone ash and Cornish stone were pale in colour, one containing 40% of the former and 60% of the latter giving the darkest tint. In mixtures with 60% china clay, the brown colour diminished as the proportion of stone increased. In mixtures with 40% china clay, the colour changed from green to blue-green, yellow, and brown as the proportion of bone ash diminished. The greenish-blue became reddish-brown on keeping and other mixtures low in stone also tended to turn brown in time, apparently because of oxidation. A subsequent glost fire tended to clear the colours, but firing in an enamel kiln intensified them. Glazes on bodies deficient in stone and giving greenish-blue and reddish-brown colours tended to craze. The contraction of bodies with excess of stone was high and they tended to blister.—A. B. S.

*Bone-china body; Microscopical study of —.* H. B. Cronshaw. Trans. Ceram. Soc., 1917—1918, 17, 153—158.

THE series of samples prepared by Yates and Ellam (see preceding abstract) showed, under the microscope, a preponderance of an isotropic and glassy base containing swarms of crystallites, small angular fragments of quartz, relatively large pores, but no minerals having fully developed crystalline forms. The brown colour of some of the pieces appears to be due to a clear brownish glass scattered irregularly through the mass.—A. B. S.

*Tiles; Effect of pressure on the subsequent contraction, tensile strength, and crazing of —.* E. W. Wain. Trans. Ceram. Soc., 1917—1918, 17, 111—115.

TEST-PIECES made of an ivory body composed of ball clay, china clay, flint, and Cornish stone were subjected to various pressures up to 1220 lb. per sq. in. and were then fired in a biscuit oven at Seger cone 5a. The results obtained show that the greater the pressure, the less the contraction. The tensile strength increases with increasing pressure. Using a glaze with a strong tendency to craze, it was found that the tendency to crazing is lessened when the articles are made under great pressure.—A. B. S.

*Chrome-tin pink.* E. W. T. Mayer. Trans. Ceram. Soc., 1917—1918, 17, 104—105.

To mixtures of tin oxide, lime, and silica in various proportions was added a constant proportion (3%) of potassium bichromate. The mixtures were heated in unfired cups in a china biscuit oven, crushed, re-heated, wet-ground,

washed, and dried. The products were banded and dipped in lead and leadless glaze respectively. The colours produced were as follows: Mixtures free from lime were lilac, the tint diminishing as the proportion of tin oxide decreased. Mixtures containing no tin oxide were light green. The remaining mixtures were chrome-tin pink, those corresponding to the innermost positions in a triangular diagram of which the corners represented  $\text{SnO}_2$ ,  $\text{CaO}$ , and  $\text{SiO}_2$  respectively being the most promising. This confirms Seger's previous observations. Good pinks were produced by a mixture containing 20 lime, 50 tin oxide, and 30 silica, with 3% potassium bichromate, to which were added various proportions of calcium fluoride, borax, boric acid, Cornish stone, felspar, and alumina respectively. Calcium fluoride darkens the tint; borax and boric acid respectively increase the richness of the tints. Felspar gives lighter tints than Cornish stone. Alumina inclines the colour to rose pink.—A. B. S.

*Magnesian glazes; Effect of — on underglaze colours.* C. P. Shah. Trans. Ceram. Soc., 1917—1918, 17, 106—110.

THE effect of a magnesian glaze containing 0.1 equiv.  $\text{MgO}$  in its formula on various underglaze colours was studied and it was found that the colour of light browns due to iron oxide and potassium bichromate is destroyed. Victoria greens are completely altered in tone and other greens are modified. Crimson colours are entirely destroyed and most of the colour in pinks is destroyed. Cobalt blues are not affected. Bluish-greens containing cobalt and chromic oxides are slightly changed. A black composed of cobalt, nickel, iron, manganese, and chromic oxides is greatly improved. Yellow and orange are not affected; fawn is altered when printed, but not when aerographed.—A. B. S.

#### PATENTS.

*Glass and glass tubing; Apparatus for drawing cane —.* J. Gray, London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 113,892, June 12, 1917. (Appl. No. 8400 of 1917.)

THE patent relates to improved mechanism for operating the gripping, cooling, chilling, and cutting devices of the apparatus described in Eng. Pat. 105,285 (this J., 1917, 551), whereby the speed of drawing and the output of the machine are considerably increased without materially increasing the percentage of breakage.

*[Sheet] glass manufacture.* T. J. McCoy, Kane, Pa. U.S. Pat. 1,255,450, Feb. 5, 1918. Date of appl., Apr. 4, 1912.

AN apparatus for making sheet glass comprises a tank to contain the molten glass, this tank having an extended end portion with its bottom inclined upwards to form an outer wall and adapted to form the glass flowing over it into a sheet of progressively reduced thickness. A movable carrier diverts the glass from a vertical to a horizontal plane and conveys it away. The thickness of the sheet may be regulated by means of a refractory body placed above the extended portion of the tank and movable vertically. The extended portion is suitably heated.—A. B. S.

*Grindstones [; Composition for —].* C. Assirate, London. Eng. Pat. 113,557, June 11, 1917. (Appl. No. 8317 of 1917.)

GRINDSTONES are made of a mixture of 1 lb. of emery powder of any grade, 4 oz. of water-glass, and  $\frac{1}{2}$  oz. of cobalt oxide powder, baked at a temperature of  $450^{\circ}$ — $600^{\circ}$  F. (about  $230^{\circ}$ — $320^{\circ}$  C.) for from 3 to 6 hrs. The cobalt oxide is added for hardening purposes.—A. B. S.

*Dryer [for bricks].* E. B. Ayres, Assignor to The Philadelphia Textile Machinery Co., Philadelphia, Pa. U.S. Pats. (A) 1,255,685 and (B) 1,255,686, Feb. 5, 1918. Dates of appl., May 3, 1916, and Mar. 16, 1917.

(A) THE dryer is divided into sections in each of which air is circulated independently by a fan. The final section has a separate supply of heated air which is directly discharged from this section in order that the bricks may be thoroughly dried. (B) The chambers of the dryer are separated by movable curtains, so that the bricks can be transferred from one chamber to another. Hot air is supplied to the last chamber and passes from chamber to chamber through connecting flues, the moist air being discharged from the first chamber to an exhaust flue. The bricks are supplied to the first chamber and moved forwards in the opposite direction to the air.—W. H. C.

*Enamel; Composition and process for making* —. P. Eyer, Halberstadt, Assignor to R. Koepf und Co., Oestrich, Germany. U.S. Pat. 1,256,455, Feb. 12, 1918. Date of appl., July 21, 1915.

SEE Eng. Pat. 100,777 of 1916; this J., 1918, 124A.

*Enamel composition and process of manufacturing it.* P. Eyer, Halberstadt, Assignor to R. Koepf und Co., Oestrich, Germany. U.S. Pat. 1,256,456, Feb. 12, 1918. Date of appl., Feb. 14, 1917.

SEE Eng. Pat. 101,802 of 1916; this J., 1917, 388.

## IX.—BUILDING MATERIALS.

*Portland cement; Le Chatelier's boiling test for* —. T. Hattori and K. Fujisawa. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1205—1212.

It is difficult to obtain concordant results in Le Chatelier's boiling test for cement on account of the error due to the moulds. The expansion due to dipping in water immediately after moulding should be noted, as it may exceed the expansion due to boiling. The former may be reduced by aeration; the latter increases with the time of boiling up to a certain limit. The authors consider that it is not necessary to boil for 6 hrs., the time usually specified; in one case the same expansion was observed after boiling for 3 hours as after 10 hours.—A. B. S.

*Portland cement; Grit in* —. T. Hattori. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1212—1224.

THE grit which passes through a sieve with 900 meshes per sq. cm., but is retained on a 2500-mesh, consists chiefly of irregular particles which may cause the cement to expand in the boiling test. On replacing the grit by sand particles of the same size, no expansion occurs. The flour-cement is richer in sulphur trioxide than the grit, owing to the extreme minuteness of the calcium sulphate present. By removing the grit the soundness of the cement is improved. Insoluble residues of sand and raw clay and free lime would be removed simultaneously.—A. B. S.

*Brick and mortar in the Great Wall of China; Composition of* —. J. C. Witt. Philippine J. Sci., 1918, 12 A, 257.

SAMPLES of brick and mortar, apparently part of the original structure of the Great Wall of China (3rd century B.C.), had the following composition:—

	Loss on ignition.	SiO <sub>2</sub> .	Al <sub>2</sub> O <sub>3</sub> + Fe <sub>2</sub> O <sub>3</sub> .	CaO.	MgO.	Na <sub>2</sub> O + K <sub>2</sub> O.
Brick	0.10	73.02	18.96	1.29	1.05	5.73
Mortar	43.88	2.12	0.44	48.83	4.03	0.85

The bricks had apparently been sun-dried only, since the material turned red on ignition. The results indicated that no sand had been mixed with the mortar.—C. A. M.

*Dolomite; Rate of hydration of calcined* —. C. Edwards and A. Rigby. Trans. Ceram. Soc., 1917—1918, 17, 116—119.

SAMPLES of dolomite were calcined at various temperatures between 850° and 1750° C. and then kept in desiccators over water, over 20% sulphuric acid, and over 50% sulphuric acid respectively and weighed every day for ten days. The samples calcined at 1150° C. and above hydrated rapidly until about 20% of water had been absorbed, but afterwards more slowly. The authors suggest that this is due to the lime hydrating rapidly and the magnesia more slowly.—A. B. S.

*Inversions in silica bricks.* Scott. See VIII.

*Estimation of proportions of quartz, tridymite, etc., in silica bricks.* Scott. See VIII.

*Weathering-products of bricks.* Nagaya. See VIII.

## PATENTS.

*Heat insulation and other purposes; Porous composition of matter for* —. H. S. Ashenurst. Chicago, U.S.A. Eng. Pat. 113,810, Mar. 2, 1917. (Appl. No. 3080 of 1917.)

SEE U.S. Pat. 1,230,085 of 1917; this J., 1917, 880.

*Method of slaking lime. Apparatus for hydrating lime.* U.S. Pats. 1,255,268 and 1,255,682. See VII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steel; Hardening and tempering of* —. C. A. Edwards. Manchester Assoc. of Engineers, Feb. 9, 1918. Engineering, 1918, 105, 267—270.

THE principles of hardening and tempering are briefly outlined, and special reference is made to nickel, chromium, tungsten, and chromium-tungsten steels. Stress is laid on the importance of the initial temperature and rate of cooling in dealing with nickel and other alloy steels; and it is pointed out that by carburising the surface of a nickel steel containing Ni 7 and C 0.1%, and allowing it to cool slowly, the same effect is produced as by carburising and quenching an ordinary mild carbon steel. The influence of the time factor is particularly important in the case of chromium steels; the great diversity of opinion as to the function of chromium in steel, which existed until recently, was due to the non-appreciation of this fact. Investigation with a steel containing Cr 6 and C 0.63% showed that, by increasing the initial temperature, the rate of cooling could be decreased considerably without detriment to the hardening of the metal; this suggested the possibility, subsequently realised, of producing case-hardening effects in steel containing Cr 6 and C 1% by decarburising the surface to below 0.6% C and carefully adjusting the rate of cooling. The influence of chromium and tungsten, separately and together, on the tempering properties of steel are also discussed in connection with the manufacture and treatment of high-speed cutting tools.

W. E. F. P.



[Silver ore] milling practice at the Buffalo Mines, Cobalt, Ont. R. E. Dye. Trans. Canadian Min. Inst., 1917, 20, 11—27.

MILLING practice in the Cobalt district varies: at some mills gravity concentration is still used, the slime tailing being discarded and the sand tailing stacked for future treatment; at others the whole of the ore is converted into slime, which is cyanided. At the Buffalo Mine, up to the time of introduction of flotation, the ore was treated on jigs and tables and the slime cyanided, the sand tailing (with 5—6 oz. Ag) being reserved. An experimental 50-ton Callow unit for the treatment of the accumulated tailings was installed in 1915; this was followed by a 600-ton plant, which is now successfully treating the old tailings as well as those produced at present; no final tailing is discharged except from the flotation plant. The jig and table concentrates are sold to smelters, the tailings being re-ground and subjected to flotation. The cyanide plant treats all the mine and mill slimes and the high-grade plant and flotation cleaner tailings. In the high-grade plant the flotation concentrate and the cyanide precipitate are treated by chloridising roasting; the roasted product is leached with acid which removes copper, and the residue is treated with cyanide. The silver solution is precipitated with sodium sulphide, and the silver sulphide melted down in an oil-fired reverberatory furnace.—W. R. S.

[Silver] ores; Present flotation practice on Cobalt —. J. M. Callow and E. B. Thornhill. Trans. Canadian Min. Inst., 1917, 20, 28—37.

THE first flotation plant at Cobalt was put in operation at the Buffalo Mine in September, 1916, (see preceding abstract). At the present time, seven plants have adopted flotation and use machines of the Callow type. The process as applied to Cobalt ores has not replaced gravity concentration, but is a useful adjunct for saving the friable silver minerals and fine leaf silver. Fine grinding (to -100 mesh) is required to separate the values from the gangue. The oil mixture used contains pine oil, coal tar creosote, and coal tar (this J., 1918, 93 A). The recovery varies with the plants: at the McKinley-Darragh-Savage Mines, Ltd., the average for two months was 82.13%. With the exception of the Buffalo Mine, the flotation concentrates produced are shipped to U.S. smelters at a heavy marketing charge, but efforts are now being made towards local treatment.—W. R. S.

Cyaniding flotation concentrate. J. G. Parmelee. Min. and Sci. Press, Sept. 15, 1917, 387. J. Chem., Met., Min. Soc. S. Africa, 1918, 18, 165—167.

CONFLICTING opinions have been expressed on the effect of oil in cyaniding. Laboratory tests proved that it had no effect on the cyanide solution or on the cyaniding of ores in which the metals are not present as sulphides. Sulphides, however, become coated and are thus protected from the action of the cyanide. The oil can be removed by treating the concentrate with alcohol or gasoline, or by roasting at a low temperature. With gold ores, simple roasting is effective, but silver ores require a chloridising roast.—W. R. S.

Copper castings for electrical purposes. G. F. Comstock. Amer. Electrochem. Soc., Oct., 1917. [Advance proof.] 6 pages.

CASTINGS of pure copper of high electrical conductivity are very difficult to obtain as the metal absorbs oxygen with formation of blow-holes and films of copper-cuprous oxide eutectic between the metal grains. Zinc is most usually employed as a deoxidiser; it produces sound castings, but the conductivity is often reduced to 40—50%. The

Titanium Alloy Manufacturing Co. (Niagara Falls) have succeeded in applying silicon as a deoxidiser; large and complicated sand castings have thus been made, the conductivity of which is 75—85% that of pure copper.—W. R. S.

Copper; Analysis of —. W. H. Woodcock. Analyst, 1918, 43, 88.

A METHOD for the analysis of commercial copper containing 99% or more Cu consists in removing about 99% of the copper by electrolysis a solution of 10 grms. of the metal, and then determining the residual copper and impurities in the electrolyte. Nickel is determined in the filtrate from the hydrogen sulphide precipitate, after removal of the arsenic by boiling the solution, by treating the filtrate with bromine, nearly neutralising the solution with ammonia, boiling it for a few minutes, then cooling to 50° C., and adding sodium acetate and dimethylglyoxime dissolved in alcohol. The nickel precipitate is collected after standing for 30 minutes at 50° C., and the iron is determined in the filtrate after the excess of dimethylglyoxime has been decomposed by boiling with an excess of hydrochloric acid and then oxidising with bromine.—W. P. S.

Zinc ferrite; Formation of — [in roasting zinc ores]. E. H. Hamilton, G. Murray, and D. McIntosh. Trans. Canadian Min. Inst., 1917, 20, 168—179.

MOLECULAR proportions of pure zinc oxide and ferric oxide were ground to pass a 120-mesh sieve, intimately mixed, and heated to temperatures ranging between 590° and 870° C. The ground products were leached with hot ammonia and ammonium chloride solution. It was thus found that no combination took place within 8 hours at 590° C., while at 870° C. the combination was complete in one hour. At intermediate temperatures the time factor had a distinct effect on the speed of the reaction. Zinc ferrite ( $\text{Fe}_2\text{O}_3 \cdot \text{ZnO}$ ) is yellowish-brown, magnetic, soluble in hydrochloric acid, insoluble in dilute sulphuric acid. The action of alumina on zinc oxide was similar, but aluminium silicate (e.g., fireclay) was found not to react. Tests in which zinc blendes were roasted at measured temperatures showed that for ores poor in iron, the amount of ferrite formed can be calculated with a fair degree of accuracy, since each unit of iron combines with 0.58 unit of zinc, and the reaction is 90% complete after 4 hours at 760° C. With ores showing a zinc-iron ratio of about 1:1, the amount of ferrite formed was much smaller than that calculated.—W. R. S.

Zinc; The hydrometallurgy and electrolytic precipitation of —. F. D. James. Univ. of Missouri Bull., Aug., 1917. 28 pages.

DATA on roasting and sulphuric acid leaching of blende and electrolysis of the zinc sulphate solution on a laboratory scale are given. The zinc sulphate liquor must undergo careful purification prior to electrolysis. The iron, present mostly in the ferrous state, is removed by adding manganese peroxide followed by lime and roasted ore. The ferric hydroxide carries down arsenic and antimony. The other impurities (copper, cadmium, etc.) are then precipitated by metallic zinc, generally in the form of zinc dust. The purified solution is electrolysed with lead anodes and aluminium cathodes. The zinc deposit is stripped from the latter with a cold chisel, and the clean smooth cathode can be used repeatedly. The effect of impurities is as follows: Very small quantities of arsenic and antimony suffice to prevent the formation of a coherent deposit. Iron contaminates the deposit. Copper deposits on the cathode, then drops off in spongy form; if its quantity is small, it causes black spots and

prevents deposition. Cadmium deposits along with the zinc. Only manganese is beneficial, and if not present it must be added to eliminate iron; it is re-oxidised during electrolysis.—W. R. S.

*Zinc extraction: Electrolytic* — at Trail, B.C. E. H. Hamilton. American Electrochem. Soc., Oct., 1917. (Advance proof.) 3 pages.

THE electrolytic plant of the Canadian Mining and Smelting Co. was started in April, 1916; it treats ore containing Zn 19–24, Pb 14, Fe 23–31, and S 21–29%. The ore is finely crushed (72% to –200 mesh), roasted in Wedge furnaces until the sulphur is reduced to 5%, and then treated first with partly neutralised spent electrolyte until the latter is neutral. The sulphuric acid used is derived from the gases produced on roasting the ore. Leaching is conducted in a series of Dorr classifiers and thickeners; copper and cadmium are precipitated from the solution by "atomised" zinc, with subsequent filtration. Each electrolytic tank (82 in. long, 27 in. wide, 42 in. deep) has 17 lead anodes, and 16 aluminium cathodes with a total area of 10 sq. ft. The current density is 24 amp. per sq. ft., with a potential difference of  $3\frac{1}{4}$  volts across the tanks. The electrolyte contains Zn 7.5, Fe 0.014, Cd 0.003–0.008, Cu 0.0003%, and sometimes Mn, Ca, and Mg; when spent, it assays Zn 3–5, Fe 0.013, and  $H_2SO_4$  5–7%. The cathode zinc is stripped off the aluminium and melted in a reverberatory, giving spelter 99.92% pure. The daily production is about 50 tons.—W. R. S.

*Aluminium: Metallography of* —. *Preparation and etching of microsections.* R. J. Anderson. Met. and Chem. Eng., 1918, 18, 172–178.

FOR preparing aluminium and light aluminium alloys for etching, the metal surface is treated with dry, coarse emery paper and then with dry No. 0 French Hubert paper. This is followed by successive treatments with Nos. 00, 000, and 0000 French Hubert papers—all thinly coated with paraffin wax (to prevent the entrance of fine particles of emery into the metal)—and the surface is then polished, first with tripoli on a duck-covered board, and finally with a good, non-alkaline metal polish, or with No. 3 levigated alumina, on fine broadcloth or old, well-washed calico. Only light rubbing or pressure is required, owing to the softness of the metal. For etching, satisfactory results may be obtained by the use of sodium or potassium hydroxide, provided the black deposit produced is removed by means of chromic acid; but, in the author's opinion, hydrofluoric acid is the best reagent. With a 15% aqueous solution of the latter, the structure of cast aluminium is developed in about one minute; the metal surface becomes darkened as the etching proceeds, but the darkening is readily removed by immersion in strong nitric acid for a few seconds. Typical microstructures of aluminium as cast, rolled, annealed, etc., and of cast aluminium-copper and manganese-aluminium alloys are shown in a series of twenty photomicrographs.

W. E. F. P.

*Lead: Determination of* — as phosphate, and its separation from antimony. G. Vortmann and A. Bader. Z. anal. Chem., 1917, 56, 577–580.

A METHOD, which is useful for the analysis of hardened lead, depends on the quantitative precipitation of lead as phosphate from an ammoniacal solution containing a tartrate. The solution, containing about 0.5 gm. of lead nitrate, is treated with 5 grms. of tartaric acid, then made slightly ammoniacal, heated to 80° C., and treated with 100 c.c. of 10% ammonium phosphate solution (sodium or potassium phosphate must not be used). The mixture is maintained at

about 75° C. for 16 hours, cooled, the precipitate collected, washed with dilute ammonium nitrate solution, dried, ignited at a low temperature, and weighed. If a sufficient quantity of tartaric acid is present, antimony is not precipitated under these conditions and may be separated as sulphide from the filtrate from the lead phosphate.

—W. P. S.

*Metal colouring. III. "Hiirugane."* S. Miyazawa. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1102–1114.

"HIIRUGANE" is the name applied to a blood-red coloured metal prepared either by treatment of copper with an aqueous solution of copper sulphate and verdigris or by heating a copper alloy with a paste containing a salt of copper, borax, and some water. The coloration of various kinds of alloys known as "Shakudo" and "Shibuichi" (this J., 1916, 884) has been shown to be due to the formation of a thin layer of cupric oxide on the surface of the alloy in consequence of a local electric circuit which causes the cupric ion to be produced and the solubility limit of cupric hydroxide to be exceeded. In this case the layer nearest the surface of the alloy may consist mainly of cuprous oxide produced by the reduction of precipitated cupric hydroxide by metallic copper in the boiling solution of the salt. If, instead of an alloy, metallic copper is treated, the local electric circuit will be absent and the formation of the cupric ion very restricted, with the production of an extremely thin layer corresponding to the layer of cuprous oxide nearest the surface of the metallic copper, as in the case of "Shakudo." Any further formation of the layer of cupric oxide takes place with difficulty. Thus the blood-red colour of "Hiirugane" may be due to the colour of mixed cuprous and cupric oxides. In the dry method, the salt of copper changes to cupric oxide and this coating of oxide is removed by polishing until the layer of cuprous oxide nearest the metal is reached, giving the metal a dark red colour. The author, without polishing the coating of oxide, obtained a brighter red colour by heating the metal with the paste on its surface and placing it in water or boiling it in the same solution of salt as is used for colouring "Shakudo." This result is possibly due to the partial reduction of cupric to cuprous oxide.—J. F. B.

*Manufacture of crude sodium manganate.* Wartenweiler. See VII.

#### PATENTS.

*Ingot-iron and steel: Manufacture of* —. Deutsch-Luxemburgische Bergwerks- und Hütten-A.-G., Bochum, and A. Klinkenberg, Dortmund, Germany. Eng. Pat. 113,512, Mar. 7, 1917. (Appl. No. 3398 of 1917.) Addition to Eng. Pat. 105,548 (this J., 1917, 1277).

PEAT and brown coal or other combinations of carbon, hydrogen, and oxygen, which on heating generate gas and do not part with more than 50% of their total carbon, are used instead of the carbohydrates prescribed in the chief patent, to deoxidise iron and steel baths rich in oxygen. The materials mentioned may also be used for the treatment of iron and steel baths free from oxygen and of iron alloy baths for the purpose of carbonising them.—T. H. B.

*Iron and steel: Process of producing* —. S. McDonald, Alhambra, Cal. U.S. Pat. 1,255,191, Feb. 5, 1918. Date of appl., Dec. 6, 1916.

A QUANTITY of iron ore is melted on the top of a molten charge of iron or steel in a crucible, and a combustible gas or a non-oxidising gas mixed with powdered carbonaceous material is blown



downwards against the iron ore. Some of the ore is carried below the surface of the metal and is reduced to iron or steel.—W. F. F.

*Wrought-iron; Manufacture of* — J. Ashton, Assignor to A. M. Byers Co., Pittsburgh, Pa. U.S. Pat. 1,255,499, Feb. 5, 1918. Date of appl., Aug. 18, 1917.

DISINTEGRATED refined iron is heated to welding heat in a bath of molten slag having a lower melting point than the iron, and the mingled slag and iron then subjected to squeezing and rolling.—W. F. F.

*Ores; Method and apparatus for concentrating by flotation* — G. Gröndal, Djursholm, Sweden. Eng. Pat. 108,308, July 7, 1917. (Appl. No. 9852 of 1917.) Under Int. Conv., July 26, 1916.

A GASEOUS fluid carrying oil is introduced in the form of a large quantity of very small bubbles at the bottom of a vessel into which the ore pulp is also delivered. The mixture flows through an opening into an adjoining narrow chamber in which the froth forms a high column which overflows into a discharge conduit for concentrate whilst the gangue sinks to an outlet at the bottom. Sets of chambers and receptacles are combined for passage of the liquid and mineral particles through them in succession.—T. H. B.

*Ilmenites or titanite iron ores; Process for the treatment of* — P. Raffin et Fils, Nice, France. Eng. Pat. 111,668, Nov. 19, 1917. (Appl. No. 17,036 of 1917.) Under Int. Conv., Nov. 21, 1916.

CONCENTRATION to a high content of titanite oxide is effected by adding an agglutinant such as tar or carbonaceous oil to a mixture of ilmenite and pulverised carbon, heating to between 900° and 1100° C., and subjecting the product to the action of dilute sulphuric or hydrochloric acid to dissolve iron. The ilmenite is previously deprived of silica and other impurities by magnetic separation.—T. H. B.

*Ores; Electro-chemical process for the treatment of metallic* — C. O. Griffith, Wormit, Fife. Eng. Pat. 113,508, Mar. 6, 1917. (Appl. No. 3285 of 1917.)

IN the production of metallic sulphides, e.g., of antimony, along with by-products such as sodium hydroxide or other compounds, an electric current is passed through a cell containing an electrolyte of sodium or other chloride, the anode being formed of a metallic ore or concentrate, whilst the cathode is of any suitable material. The necessary sulphur is supplied by the decomposition of a sulphide ore used as the anode, or by the cathode having a layer, block, or cake of sulphur near or in contact with it, or by adding a thiosulphate or other sulphur compound to the electrolyte, the cathode in the latter case being enclosed in a separate compartment by means of a porous partition.—B. N.

*Furnaces; Metal heating, metallurgical and analogous heating* — The Richmond Gas Stove and Meter Co., Ltd., Warrington, and J. Thompson, Sheffield. Eng. Pat. 113,594, May 14, 1917. (Appl. No. 276 of 1918.)

A METAL-HEATING furnace has a stationary heat-guard of asbestos held between metal plates, at its front and working face. There is an opening in the guard, closed by a movable transparent screen (e.g., of mica), through which articles or instruments can be passed into the furnace, and the interior of the heating chamber can be seen. By means of a pipe with a row of holes in its upper surface an upward stream of air under pressure is delivered between the furnace front and the guard.—T. H. B.

*Zinc; Recovering — from its solutions*. F. Laist and J. O. Elton, Assignors to Anaconda Copper Mining Co., Anaconda, Mont. U.S. Pat. 1,255,433, Feb. 5, 1918. Date of appl., June 16, 1916.

ZINC is deposited electrolytically on a cathode of metal, e.g., pure aluminium, which is electro-positive to zinc, and is substantially free from impurities which are electronegative to zinc, until a deposit suitable for stripping is formed.—W. F. F.

*Zinc-bearing solutions; Method of purifying* — F. Laist and J. O. Elton, Assignors to Anaconda Copper Mining Co., Anaconda, Mont. U.S. Pats. (A) 1,255,434 and (B) 1,255,435, Feb. 5, 1918. Date of appl., June 16, 1916.

(A) A SOLUTION containing zinc is agitated with finely divided zinc to precipitate a portion of the copper, and then further purified by contact with moving zinc surfaces in a tube mill. The zinc-bearing material carried forward by the solution is collected and returned to the purifying system at the first stage. (B) An electric current is passed through the solution from an anode surface of electrolytically deposited zinc sponge to produce a deposit of zinc sponge, and the solution is then agitated in contact with finely-divided zinc. Zinc may then be deposited electrolytically from the purified solution.—W. F. F.

*Zinc; Process of recovering—from ores*. F. Laist and J. O. Elton, Assignors to Anaconda Copper Mining Co., Anaconda, Mont. U.S. Pats. (A) 1,255,436 and (B) 1,255,439, Feb. 5, 1918. Dates of appl., June 16 and July 10, 1916.

(A) IN a cyclical process of recovering zinc from ores also containing arsenic or antimony, the ore is alternately leached with an acid solvent for zinc, and the zinc deposited electrolytically from the resulting solution. An acid solution containing a ferrous salt is oxidised by means of a manganese compound which is more highly oxidised than the corresponding manganous compound. The resulting ferric and manganous solution is added to the leach solution subsequently to the electrolysis, in proportion sufficient to eliminate arsenic and antimony. The ferric compound is then removed by precipitation together with the arsenic and antimony prior to the succeeding electrolysis. (B) Zinc ore is leached with an acid solvent for zinc, iron is precipitated from the solution as a ferric compound, and the solution then treated with finely-divided zinc to precipitate metals electronegative to zinc. Zinc is deposited electrolytically in spongy form from portions of the solution with zinc anodes for use in the earlier stage of the process and further quantities of zinc are deposited as reguline metal with insoluble anodes. The resulting acid solution is used for the preliminary leaching operation.—W. F. F.

*Metals [e.g., zinc]; Electrodeposition of* — F. Laist and J. O. Elton, Assignors to Anaconda Copper Mining Co., Anaconda, Mont. U.S. Pat. 1,255,437, Feb. 5, 1918. Date of appl., June 16, 1916.

IN the deposition of metals, e.g., zinc, by electrolysis, in which a purifying system, storage system, and main electrolytic system are combined, the solution flows through an "indicator cell" between the purifying and storage systems.—W. F. F.

*Zinc ores; Process of treating — Process of treating complex ores or concentrates therefrom*. F. Laist, Assignor to Anaconda Copper Mining Co., Anaconda, Mont. U.S. Pats. (A) 1,255,438 and (B) 1,255,440, Feb. 5, 1918. Dates of appl., July 10 and Nov. 3, 1916.

(A) ZINC ore containing iron is calcined and leached with dilute sulphuric acid. The iron is precipitated from the solution by a material con-

taining zinc oxide and the precipitate separated from the solution. The precipitate is calcined at a temperature below that of the first calcination, but at which the ferric compounds are rendered insoluble whilst the zinc sulphate is not decomposed. The latter is then extracted from the mixture. (B) Ores or concentrates containing zinc, lead, copper, and precious metals, are roasted and then leached with dilute sulphuric acid. The solution containing zinc is separated and purified, and the zinc deposited electrolytically. The residue containing the remaining metals and some zinc is smelted so that oxides of lead and zinc are volatilised, and a matte containing copper and precious metals is produced. The volatilised product is treated with dilute sulphuric acid to extract zinc, leaving a residue rich in lead, and the copper and precious metals are recovered from the matte.—W. F. F.

*Blast-furnace stoves; Gas burners for*—S. Sugimoto, Honkeiko, S. Manchuria. Eng. Pat. 113,740, July 31, 1917. (Appl. No. 11,032 of 1917.)

*Plating ferrous metals [with tin]; Method of*—W. E. Watkins, New York. Assignor to The Metals Plating Co. U.S. Pat. 1,256,084, Feb. 12, 1918. Date of appl., Nov. 11, 1914.

SEE Eng. Pat. 18,932 of 1914; this J., 1916, 53.

*Titanium ores; Concentration of*—M. R. Raffin, Assignor to P. Raffin et Fils, Dunkirk, France. U.S. Pat. 1,256,368, Feb. 12, 1918. Date of appl., July 6, 1917.

SEE Eng. Pat. 111,668 of 1917; preceding.

*Manufacture of phosphorus [from basic slag]*. Eng. Pat. 113,584. See VII.

[*Electrolytic*] cell. U.S. Pat. 1,255,197. See XI.

*Machine for testing the hardness of metals*. Eng. Pat. 114,126. See XXIII.

*Hardness-testing machine*. U.S. Pat. 1,255,913. See XXIII.

## XI.—ELECTRO-CHEMISTRY.

*Sulphur dioxide; Depolarisation by*—M. de K. Thompson and A. P. Sullivan. Met. and Chem. Eng., 1918, 18, 178—179.

IN continuation of previous work on the electrolytic oxidation of sulphurous acid (this J., 1917, 81), experiments have been made on the use of sulphur dioxide as a depolarising agent. Constant currents were passed through an electrolytic cell of special design (containing an anode compartment through which sulphur dioxide could be passed when required), and the anode potentials under the various conditions were measured against a standard electrode. In 10% and 16.5% solutions of sulphuric acid and copper sulphate, sulphur dioxide was found not to depolarise a smooth platinum anode at current densities above 10 and 6.2 amp. per sq. dm., respectively. In both solutions, the voltage of the anode was decreased by about 1.5 volts unless certain limits of current density were exceeded; this limit was between 7.5 and 10 amp. per sq. dm. for the sulphuric acid, and less than 7.5 amp. for the copper sulphate solution. These results are not in agreement with those obtained by M. R. Thompson (Met. and Chem. Eng., 1917, 16, 178) who found that with an anode of platinum black in a solution of copper sulphate, sulphuric acid, and sulphurous acid, sulphur dioxide ceased to depolarise at a current density of 3.23 amp. per sq. dm. Further results indicated that the current density at which depolarisation ceases is largely dependent on the metal of the electrode.—W. E. F. P.

## PATENTS.

*Insulation; Electrical*—W. T. Cushing, Assignor to The Cutler-Hammer Manufacturing Co., Milwaukee, Wis. U.S. Pat. 1,255,139, Feb. 5, 1918. Date of appl., Sept. 9, 1915.

AN electric insulating composition comprises an asbestos base, and a binder including a varnish of East India gum, China wood oil, and rosin, with a mixture of two parts of stearin pitch and one part of coal tar pitch for retarding oxidation of the varnish.—B. N.

[*Electrolytic*] cell. J. L. Malm, Assignor to The Midwest Metals Co., Denver, Colo. U.S. Pat. 1,255,197, Feb. 5, 1918. Date of appl., Apr. 26, 1915.

THE cell chamber is provided with a central trough-like well opening out through the cell wall: a metal cathode fills the well, so that the upper surface only of the cathode is in contact with the electrolyte, and the end portion extends through the side wall of the chamber. The anode substantially fills the horizontal section of the chamber and also extends through the wall. To ascertain the height of the electrolyte and the deposit of metal, an opening is provided in the anode, fitted with an insulating sleeve, and an opening is provided in the cover in alignment with the insulating sleeve so that a bar may be inserted to touch the bottom of the cell.—W. F. F.

*Battery; Electric*—R. C. Benner and H. F. French, Fremont, Ohio, Assignors to National Carbon Co. U.S. Pat. 1,255,283, Feb. 5, 1918. Date of appl., July 13, 1916.

THE negative element of the battery consists of copper oxide containing an oil holding sulphur in solution, within a perforated container, in an alkaline electrolyte. The sulphur is incorporated with the depolarising mass by soaking the latter in a hot saturated solution of sulphur in oil, whereby sulphur crystallises out on cooling.—B. N.

*Electrolyte*. J. Coulson, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,255,391, Feb. 5, 1918. Date of appl., Apr. 5, 1917.

AN electrolyte for electrolytic condensers, lightning arresters, rectifiers, and the like, comprises an aqueous solution containing not more than 2% of potassium arsenate.—B. N.

*Electric furnace-control apparatus*. A. D. Keene, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,255,613, Feb. 5, 1918. Date of appl., Sept. 5, 1917.

THE furnace comprises two movable electrodes and a stationary conductor, with an electrical circuit connected to the electrodes for operating several arcs in series between the movable electrodes and the stationary conductor. Means, operated by a motor, are arranged for adjusting one of the movable electrodes in response to variations of the current in the circuit. A resistance, as a shunt to the arc from the current-controlled electrode, and a motor-operated means are arranged for adjusting the second electrode in response to variations of voltage in the circuit.—B. N.

*Uninflamable liquids adapted for use in insulating and other similar purposes*. G. Lépine, Neuchâtel, Switzerland, U.S. Pat. 1,255,618, Feb. 5, 1918. Date of appl., June 29, 1917.

A MIXTURE of 80 parts of pentachloroethane, 18 parts of pentachlorobenzene, and 2 parts of hexachlorobenzene, with the addition of 1 part of toluidine.—B. N.



*Electric transformers, especially suitable for use in connection with electric furnaces.* J. Bibby, London. Eng. Pat. 113,899, June 22, 1917. (Appl. No. 9014 of 1917.)

*Apparatus for electrical separation of suspended particles from gases.* U.S. Pat. 1,252,183. See I.

*Manufacture of phosphorus.* Eng. Pat. 113,584. See VII.

*Electrochemical process for the treatment of metallic ores.* Eng. Pat. 113,508. See X.

*Method and apparatus for treating liquid [to produce colloidal tungstic oxide] for curative and therapeutic purposes.* Eng. Pat. 113,505. See XX.

## XII.—FATS; OILS; WAXES.

*Valenta tests [of fatty oils]; Modified acetic acid reagent for—.* A. E. Parkes. Analyst, 1918, 43, 82—85.

THE inconvenience caused by the crystallisation of acetic acid in cold weather may be overcome, as regards the use of the acid in the Valenta test, by employing a mixture of acetic acid and other lower fatty acids together with a small proportion of water. Such a mixture is conveniently prepared by adding 10% of propionic acid or butyric acid and from 1 to 2% of water to glacial acetic acid. The solidifying point of acetic acid is lowered by the presence of water and certain fatty acids, whilst the turbidity temperature in the Valenta test is raised by water and lowered by fatty acids, and by adding 10% of propionic or butyric acid to 99% acetic acid and then water in small quantities (1—2% is generally necessary) a reagent may be obtained which gives the turbidity temperature required when standardised against pure butter or other fat. This mixture of acids remains liquid when cooled to 0° C.—W. P. S.

*Cherry-kernel oil.* H. L. Maxwell. Chem. News, 1918, 117, 122—124.

THE kernels from dried cherry stones, *Prunus cerasus*, yielded on extraction with ether 37.6% of an amber-coloured oil with an odour of almonds. It gave a deposit at -5° C., became solid at -20° C., and had sp. gr. 0.922 to 0.925, and saponification equivalent 276.8. Amygdalin was present in the residue, which contained 4.2% of nitrogen. —C. A. M.

*Comparison of linseed oil and lumbang oils as paint vehicles.* Aguilar. See XIII.

## PATENTS.

*Oil from seeds and like oleaginous materials and substances; Apparatus for the extraction of—.* C. Downs and R. A. Bellwood, Kingston-upon-Hull. Eng. Pat. 113,530, Apr. 2, 1917. (Appl. No. 4711 of 1917.)

THE solvent for the extraction of oil from seeds, etc., is passed through a preliminary heater where it is heated by means of vapours coming from the evaporator, and then passes through a second heater in which it is heated by steam to the required temperature before introduction into the extraction vessel. The preliminary heater is preferably arranged between the evaporator and the condenser, and may be provided with means whereby the solvent may be conducted to the evaporator without passing through the second heater. It may also be connected directly with the condenser by means of a pipe provided with a trap.—C. A. M.

*Catalyser [for hydrogenation processes] and process of making same.* C. Ellis, Montclair, N.J. U.S. Pat. 1,255,590, Feb. 5, 1918. Date of appl., Sep. 21, 1916.

A CATALYST for use in the hydrogenation of organic substances consists of a compound of a metalloid element and a catalytic metal (e.g., nickel boride), which is protected from atmospheric oxidation (e.g., by means of a seal of oily material) prior to the hydrogenation. An active base may be used as the vehicle for the non-oxidised catalytic compound. —C. A. M.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Acroplane dopes.* G. J. Esselen, jun. J. Ind. Eng. Chem., 1918, 10, 135—137.

THE specification of the Bureau of Standards, U.S.A., requires the tensile strength of linen fabrics to be increased by at least 25% and that of cotton fabrics by at least 15% after treatment with a dope. The average weight of a suitable fabric for aeroplane wings is about 3½ to 4 oz. per sq. yd., and the tensile strength about 90 to 100 lb. per inch width. As a rule the dope when dry adds about 2 to 2½ oz. per sq. yd. to the weight, but in certain types of war machines the combined weight of fabric and dope does not exceed 6 oz. The dopes used consist essentially of a solution of cellulose nitrate or of cellulose acetate (about 8 oz. per gall.) in mixtures of amyl acetate and acetone, or (for cellulose acetate) in acetone or mixtures of tetrachloroethane and alcohol. In Europe only acetate dopes are used, but in America pyroxylin dopes are also used, whilst some manufacturers attempt to reduce the inflammability by applying coats of acetate dope above the pyroxylin coats. A dope has better penetration, adheres better, and offers greater resistance to the atmosphere when applied to unsized fabrics than to sized fabrics. Dopes are usually tested by applying them to linen stretched on frames about 12 to 15 in. square, which are then exposed on a roof. This test varies with the season of the year, and although the length of time before the flexibility of the fabric disappears affords a measure of the relative value of different dopes, the conditions are not quite parallel with those which obtain in service since the effect of vibration is wanting. If, however, a dope remains flexible, vibration alone would not cause it to crack. The Bureau of Standards requires a dope to retain its flexibility for at least 60 days when exposed to the weather. The exposed fabric shows a certain amount of weakening, which is more pronounced with nitrate than with acetate dopes, and with cotton than with linen fabrics. In one case a cotton fabric treated with a nitrate dope had its tensile strength reduced to 48.5% of the original after 3 weeks' exposure, whilst linen coated with the same dope showed a reduction to 68% of its original strength. With an acetate dope the corresponding reductions in the strength of cotton and linen were 87.2 and 84.8% respectively of the original values. The presence of tetrachloroethane in a dope gives a resilience to the aeroplane wing which no other constituent does, but owing to the poisonous character of the vapour its use is prohibited in England. It is contended, however, that efficient ventilation would eliminate this danger. Cellulose acetate in sheets 10/1000 in. thick, and weighing about 9 oz. per sq. yd., is strong enough for aeroplane wings, and owing to its transparency is almost invisible at a height. It has the drawback, however, that a tear when once started spreads very rapidly.—C. A. M.

*Linseed oil and lumbang oils as paint vehicles: Comparison of —.* R. H. Aguilar. Philippine J. Sci., 1917, 12A, 235—243.

FRESHLY expressed samples of lumbang bato (batu) oil from *Aleurites moluccana* (candle-nut oil) and of lumbang banucalag oil from *A. trisperma*, had the following characters:—

Oil.	Sp. gr. at 15° C.	Saponif. value.	Acid value.	Iodine value.
Lumbang bato .....	0.9252	192.95	1.05	150.36
Lumbang banucalag ....	0.9368	197.74	8.70	145.25

Fresh lumbang bato oil increased 11.03% in weight in 4 days in a drying test, and formed a film which was dry and firm in 4 to 6 days, whilst fresh banucalag oil increased 8.92% in weight, and formed a film which was dry but slightly opaque in 4 days. Boiled and old oils dried much more rapidly than the fresh samples. Paint films were prepared by Gardner's method of applying the paint to paper sized with glue, and when dry removing the film by immersion in water at 40° C., washing it free from glue, and suspending it on a glass rod to dry. The "paint" consisted of 15% of precipitated silica incorporated with the oil, and the films were made by applying 3 coats of 2.5 grms. each to 400 sq. cm. of sized paper. The strength and elasticity of the films were tested by means of a modification of the machine of Gardner and de Horvath (Gardner, "Paint Technology and Tests," New York, 1911, p. 79), in which a mercury manometer was used for measuring the pressure. The oil-silica films became soft and tacky, and were greatly reduced in strength after 60 days. In the case of films prepared from a mixture of 40 grms. of red lead with 25 grms. of oil, the maximum strength was reached between the third and fourth month, and subsequently there was a gradual decrease in the strength. The mixture of lumbang banucalag oil and red lead became thick and pasty in 15 mins., whereas lumbang bato oil did not behave in this way. Hence, banucalag oil by itself is not a satisfactory paint medium, although when mixed with lumbang bato oil in the proportion of 1:1 to 3 parts, it yields a rapid-drying and satisfactory paint. Mixtures of the two oils containing 50 to 75% of lumbang bato oil do not "set" as in the case of linseed or lumbang bato oil when incorporated with red lead, and do not dry into a paste in the containers. The moisture-excluding capacity of the films was determined by fastening pieces with Canada balsam over the mouths of 200 c.c. bottles containing calcium chloride or strong sulphuric acid, and determining the percentage increase in weight of the drying agent after an exposure of from 5 to 60 days. It was found that the moisture-excluding property of lumbang bato oil-red lead paint films was not as high as that of the corresponding linseed oil-red lead films. For example, in the case of sulphuric acid the increases in weight with the linseed oil and lumbang bato oil films after 5 days were 0.24 and 0.42%, and after 40 days 1.67 and 3.03% respectively. Mixtures of lumbang bato and lumbang banucalag oils in the proportions of 75 and 50% of the former yielded red-lead paint films which excluded moisture almost as well as linseed oil films, and such mixtures therefore appear to be suitable paint media.—C. A. M.

*γ-Alkali resin soaps; Colloidal nature of the —.* L. Paul. Kolloid Zeits., 1917, 21, 176—191.

SOLUTIONS of the alkali resin soaps behave like highly disperse colloidal systems. The colloidal

soaps combine with basic dyes to form coloured resin lakes and are also characterised by the readiness with which they combine with petroleum hydrocarbons. (See also J. Chem. Soc., Apr., 1918.) —H. M. D.

*Retardation of the formation of Prussian blue and other reactions in aluminium hydroxide sols.* Reitstötter. See VII.

*Brewers' pitch.* Eckhardt. See XVIII.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Testing of —.* A. van Rossem. Communications of the Netherlands Govt. Inst. for Advising the Rubber Trade and the Rubber Industry. Int. Assoc. for Rubber Cultivation in the Netherland Indies, 1917, Part V., 125—175.

It has been found that different plantation rubbers, mixed with sulphur in the proportion 92½:7½, show with a certain vulcanisation coefficient approximately the same stress-strain curve. Raw rubber should therefore be judged by its rate of vulcanisation and by its mechanical properties in comparison with the average values corresponding with the vulcanisation coefficient obtained under the standard method of vulcanisation. Rubber goods on the other hand should be tested as far as possible from the point of view of their intended application. For example, to test vulcanised rubber cylinders as to their behaviour under compression, a sample cylinder is enclosed in a steel cylinder of slightly greater diameter and then compressed by a steel piston, the pressure and corresponding compression being recorded in the form of a hysteresis loop on a chart. An examination has been made of the relative quality of Tjipetir gutta-percha collected from twigs and leaves respectively, the composition, specific insulating resistance, and puncture voltage of each quality being tested as well as the viscosity in benzene and carbon tetrachloride; both qualities were found to contain a high percentage of pure gutta-percha and to compare very favourably with the wild product, the leaf variety being slightly the better of the two. In testing the insulating material of so-called "normal-rubber-insulated wire," rigid adherence to the specified composition, viz., a minimum of 33.3% rubber and maxima of 6% resins, 66.7% filler (including sulphur), and 1% alcoholic potash extract is considered essential, and the further reduction of the maxima for resins and alcoholic potash extract to 5% and 0.5% respectively is suggested, together with additional mechanical tests as to the tensile qualities in order to check the use of reclaimed rubber.

Details are given of the standard vulcanisation method employed for testing raw rubbers, the rubber-sulphur mixture being finally heated by steam for 1½ hours at 52 lb. pressure or 148.25° C. It has been found more convenient, however, to dispense with steam as a source of heat. Vulcanised specimens free from porosity can easily be obtained by other methods if the surrounding pressure during vulcanisation is maintained above the vapour tension of water at the temperature of the rubber; an apparatus is described in which discs of the rubber-sulphur mixing 7.5 cm. in diameter and 5 mm. thick, enclosed with a little water in small moulds, are vulcanised by being suspended in a bath of liquid paraffin at 147° C. for 1½ hours, this temperature giving the same chemical and physical results as the steam treatment at 148.25° C. The new method also has the advantage that further samples may be introduced at any time without appreciably affecting



the temperature of the bath, and that the transference of heat occurs more rapidly than in the older method. For the determination of the coefficient of vulcanisation, the combined sulphur in an acetone-extracted sample is estimated by Rothe's method with magnesium nitrate; the free sulphur in the acetone extract is estimated by Frank and Marckwald's method, but if the amount exceeds 1%, its calculation from the difference between the combined sulphur and total sulphur is preferred. As the amount of resinous matter that can be extracted by acetone is practically unaffected on vulcanisation, it is often convenient to determine the free sulphur merely by making comparative acetone extracts on the raw rubber and on the vulcanised mixing and then, knowing the percentage of sulphur added to the rubber, to obtain the figure for combined sulphur by difference. Numerous experiments made by the Institute lead to the important conclusion that for rubber-sulphur mixtures there is a close connection between the vulcanisation coefficient, the breaking load, the stress necessary to produce an elongation of 850%, and the percentage permanent deformation. As the result of an examination of various tensile methods for determining the mechanical properties of rubber, the conclusion is drawn that the Schopper-Dalen machine possesses marked advantages over the Schwartz machine and still more over the Breuil apparatus. Using the Schopper machine with a standard mixing of 92.5% rubber and 7.5% sulphur, the stress-strain curves only very rarely intersect, and although the breaking strength of various rings of a normally vulcanised rubber may differ considerably the curves are usually coincident; it is therefore possible and advantageous to determine the position of the stress-strain curve by the position of one definite point and this is arbitrarily fixed at the stress in kilos. per sq. cm. necessary to produce an elongation of 850%; the figures for this stress are decidedly more constant than the final tensile strength of different rings made from the same mixing and there is generally a close relationship between them and the coefficient of vulcanisation of the rubber. One ring is usually sufficient for this method of testing. Rubbers which exhibit deviations from the probable curve expected from their coefficient of vulcanisation are classed as abnormal. The method of judging the mechanical properties of a rubber by the product of the breaking load and the elongation at break (Eaton and Grantlham, this J., 1915, 989) is regarded as of no advantage whatever, because the position of the maximum value in this case coincides with that of the maximum tensile strength. A comparison of first latex Hevea rubber with Brazilian rubber supports the common view that the latter is more uniform in its rate of vulcanisation, and with the same coefficient of vulcanisation the latter is found to offer a greater resistance than the average plantation rubber to stretching. Permanent set is measured by the Institute by placing Schopper rings over a glass plate with rounded edges for 24 hours, the dimensions being such that the ring is stretched to 400% of its original length; the permanent set generally decreases with an increasing coefficient of vulcanisation. For testing the resiliency of rubber an apparatus is used in which a pendulum with a fall of 205 mm. strikes at its lowest position against a rubber sample placed vertically, the rebound being recorded on a vertical piece of paper which rotates about a vertical axis; the resiliency decreases with an increased vulcanisation coefficient, the decrease becoming much more rapid when the coefficient exceeds 4.5. Such "elastometer" tests are probably of considerable value for examining the effect of "fillers" on the resiliency of rubber but are not considered essential in connection with raw rubber.—D. F. T.

## PATENTS.

*Rubber ; New agents for accelerating the vulcanisation of — and method of producing the same.* S. J. Peachey, Stockport. Eng. Pat. 113,570, Aug. 23, 1917. (Appl. No. 12,085 of 1917.)

PRODUCTS of the interaction of *p*-nitrosodimethylaniline (or its homologues) and sulphur are used to accelerate the vulcanisation of rubber. By heating the substances to about 130° to 135° C. a red vapour is evolved, which condenses, on cooling, to a yellowish-red product, whilst a dark reddish-brown mass is left, which when extracted with acetone or chloroform yields, on evaporating the extract, a dark brown viscous resin. Both the condensed yellowish-red product and the resinous mass may be used as accelerators in the vulcanising process. (See also Eng. Pat. 4263 of 1914; this J., 1915, 368.)—C. A. M.

*Impregnating fabrics with rubber ; Machine for —.* I. S. McGiehan, New York. U.S. Pat. 1,255,192, Feb. 5, 1918. Date of appl., May 20, 1916.

THE impregnating vat containing rubber solution is provided with drums or rollers for alternately and transversely bending the fabric while it is passing through the solution. The impregnated fabric passes through a compressing machine having spring-pressed rollers with rubber surfaces, geared so as to avoid slipping, which press the rubber into the fabric without distortion of the threads. The static parts of the machine are arranged so that the fabric does not come into contact with them. The fabric finally passes through a drying or semi-vulcanising device.—W. F. F.

*Serap rubber or other like material ; Machines for the treatment of —, known as serap washers.* F. Niblock, Singapore. Eng. Pat. 113,876, May 10, 1917. (Appl. No. 6636 of 1917.)

*Vulcanisation of india-rubber and other similar substances.* H. Olivier, Paris. U.S. Pat. 1,256,496, Feb. 12, 1918. Date of appl., May 21, 1915.

SEE Fr. Pat. 477,204 of 1914; this J., 1916, 644.

## XV.—LEATHER ; BONE ; HORN ; GLUE.

*Gelatin ; Effects of electrolytes on — and their biological significance. I. Effects of acids and salts on the precipitation of gelatin by alcohol.* W. O. Fenn. J. Biol. Chem., 1918, 33, 279—294.

THE volume of alcohol required to precipitate gelatin increases with increasing concentration of salts in the solution. In the case of acids and alkali, the presence of very small quantities entirely prevents the precipitation of the gelatin, whilst with larger quantities the amount of alcohol required slowly diminishes as the concentration of electrolyte increases. The effects of the ions on the precipitation of gelatin by alcohol bear a general resemblance to the effects produced on the viscosity, imbibition, and other properties of gelatin. (See also J. Chem. Soc., Apr., 1918.)—H. W. B.

## PATENTS.

*Tanning processes.* O. Röhm, Darmstadt, Germany. Eng. Pat. 104,338, Feb. 14, 1917. (Appl. No. 2197 of 1917.) Under Int. Conv., Feb. 21, 1916. Addition to Eng. Pat. 103,827.

MODIFICATIONS of the processes of the principal patent (this J., 1918, 14 A) are described. Iron-aldehyde tannage may be advantageously combined with treatment of the unhaired hides with neutralising materials or precipitants of iron, such as alkalis, phenols, vegetable tanning materials, soap, or sulphides. The advantage of a combined

iron-aldehyde tannage may also be obtained by carrying out the aldehyde tannage simultaneously with or after the neutralisation of the leather which follows the metal tannage. This also applies to chrome tanning. The use of mixtures of salts, *c.g.*, a mixture of iron and chromium chlorides, for tanning is also advantageous.—F. C. T.

*Tanning or tawing by means of aluminium salt.* O. Röhm, Darmstadt, Germany. Eng. Pat. 110,750, Oct. 13, 1917. (Appl. No. 14,862 of 1917.) Under Int. Conv., Oct. 27, 1916.

THE partial removal of tanning substance with the excess of alum from the hide may be obviated by using an aluminium salt of a lower fatty acid, such as formic or acetic acid, or a mixture of one of these acids with an inorganic aluminium salt, in place of alum as the tanning agent; or the moist leather coming from a tanning liquor containing an aluminium salt may be treated with a solution of a salt of a lower fatty acid, such as formic or acetic acid; or a salt of this kind may be added to the water used for washing leather which has been tanned with an aluminium salt and then dried.

—C. A. M.

*Tanning of hides; Apparatus for the —.* G. D'Angelo, Turin, Italy. Eng. Pat. 113,408, Sept. 11, 1917. (Appl. No. 13,036 of 1917.)

A MASONRY vat is provided with heated pipe coils in compartments formed by partitions which act as baffle-plates. The liquor is circulated over the coils, and also through the main part of the tank, by means of a paddle-wheel.—F. C. T.

*Hides; Apparatus for treating —.* J. E. Vorel, Milwaukee, Wis. U.S. Pat. 1,251,179, Jan. 22, 1918. Date of appl., Mar. 6, 1917.

A TANK for treating hides with liquids is provided with devices for keeping the liquid in motion.—F. C. T.

*Production of a nitrogenous fertiliser from leather waste.* U.S. Pat. 1,255,643. See XVI.

## XVI.—SOILS; FERTILISERS.

*Determination of phosphoric acid. Modification of the citrate method.* Grossfeld. See XXIII.

### PATENTS.

*Fertilisers [; Manufacture of —].* G. A. R. Borghesani and G. Stampa, Rome. Eng. Pat. 113,493, Feb. 27, 1917. (Appl. No. 2924 of 1917.)

CRUSHED leucite and phosphorite are mixed with small quantities of nitre-cake and pyrites ash, placed in an electric furnace and fused at 1200°—1400° C. The product is removed, cooled, and ground. The powder is soluble in a 2% solution of citric acid.—J. H. J.

*Straw and other vegetable substances; Treatment of — for manurial purposes.* E. H. Richards, Harpenden, Herts. Eng. Pat. 113,571, Aug. 24, 1917. (Appl. No. 12,186 of 1917.)

STRAW is mixed with chalk and placed in a horizontal cylinder of wire netting which can be rotated. The lower part of the cylinder dips into a trough containing water to which chalk and a culture of *B. lactis aerogenes* and *B. azotobacter* have been added. The apparatus is maintained at about 25° C. and the cylinder rotated from time to time. Fermentation is allowed to proceed for some weeks until the pentosans have been destroyed. The mass is then transferred to a tank with a conical bottom, and excess of water and a small quantity of liquid manure are added. The temperature is

maintained at 25°—30° C. by means of a heating coil in the bottom of the tank. When the mass has become liquefied, dry material from the first part of the process is added to absorb the liquid, and the product is raised by an endless lift from the tank. It may be used as a manure directly or after filter-pressing.—J. H. J.

*Leather waste; Production of a nitrogenous fertiliser from —.* R. P. Rose, Pittsburgh, Pa. U.S. Pat. 1,255,643, Feb. 5, 1918. Date of appl., July 31, 1914.

LEATHER waste is hydrolysed with dilute sulphuric acid and 1% of an oxidising agent under a steam pressure of 50 lb. per sq. in.—J. H. J.

*Superphosphate; Process of making —.* H. Blumenberg, jun., Los Angeles, Cal., Assignor to H. F. Stewart, South Pasadena, Cal., F. Blumenberg, New York, and G. D. Robertson, Los Angeles, Cal. U.S. Pat. 1,255,829, Feb. 5, 1918. Date of appl., Oct. 16, 1917.

GROUND calcareous phosphate material is mixed with acid hydrocarbon sludge, and heated until all the volatile matter is driven off and the residual carbon is left in a finely divided state. The mass is then heated with ground calcareous phosphate rock. The product contains acid calcium phosphate, gypsum, and finely divided carbon, and has fertilising properties.—J. H. J.

## XVII.—SUGARS; STARCHES; GUMS.

*Plant juices; Preservation of — for determination of sugar content.* D. O. Priestersbach. J. Amer. Chem. Soc., 1918, 40, 431—436.

THE preservation of sweet juices for subsequent analysis requires an agent which will not only prevent fermentation but also inversion, and at the same time will not interfere with the usual analytical processes. Good results are obtained with toluene and potassium mercuri-iodide combined, and it is possible that mercuric nitrate may even be better. A definite volume of the juice is neutralised with calcium carbonate, boiled, mixed with 2 grms. of anhydrous sodium carbonate per litre and also with the preservative, and then made up to the original volume again.—J. C. W.

*Sugar solutions; A special "nomon" for calculating the purity of —.* A. F. Blake. Intern. Sugar J., 1918, 20, 73—78.

IN order to obviate the calculation involved in determining the purity of mill juices and other liquors from the density and the polarisation, the so-called "nomographic reckoner" devised by H. G. Denning (J. Amer. Chem. Soc., 1917, 39, 213), which enables one to obtain almost instantly results to four figures with an error of one unit in the last place, may be used with advantage. Instead of the general series of scales as originally published, the author has compiled a special series adapted to the calculation of the purity value, in which unnecessary data are eliminated, the exponents expressed clearly with decimal points properly placed, and the ranges set forth for the greatest accuracy and ease of operation.—J. P. O.

*White sugar and molasses; Method for the manufacture of —.* E. Haddon. Bull. Soc. Chim. de Maurice, 1917, 8, 45—47. Intern. Sugar J., 1918, 20, 88—89.

SYRUP is boiled to grain, and the resulting masse-cuite centrifuged with the addition of a little poor after-product, whilst the sugar obtained is mixed either with rich after-product or with syrup and affined in small centrifugals. The after-product from this affining treatment is diluted to 30° B.



(sp.gr. 1.262) and sulphited, being subsequently used as syrup; and the poor after-product from the first centrifuging is similarly diluted and sulphited, being afterwards boiled to grain and treated in crystallisers, yielding sugar and molasses. Before centrifuging it, some molasses at 30°–35° Brix is added to facilitate the operation, and the second sugar obtained is mixed with that from the first centrifuging, the whole being affined at the same time in small centrifugals. This process is claimed to be superior to so-called "double turbinage" in that the yield is higher and there is no repeated returning of after-products to increase the viscosity of the products.

—J. P. O.

*Water; Determination of — in molasses and other sugar factory products by the distillation method.* T. van der Linden, M. Kauffman, and F. Leistra. *Archief Suikerind. Ned.-Indië*, 1917, 25, 951–962. *Intern. Sugar J.*, 1918, 20, 89–90.

The direct determination of the water in cane-molasses by distillation with xylol gives very satisfactory results, provided a certain rate of distillation is observed: if heating be prolonged some decomposition of the product with the formation of water occurs. It is necessary to make a "meniscus correction" in measuring the volume of water passing over (this amounted to 0.16 c.c. under the conditions obtaining, xylol and not air being above), and also a correction for loss of water, either by volatilisation or absorption by the liquid distilled (for the apparatus used this was found to be 0.3 c.c.). Fifty grms. of the sample of molasses or other product with 350 c.c. of xylol are placed in a copper distillation flask connected with an upright condenser, the lower end of which discharges into a 250 c.c. measuring cylinder, graduated to twentieths of a c.c. Heating is so regulated that about 100 c.c. of distillate passes over in  $\frac{3}{4}$  hour; after this the distillation is accelerated, so that in the fourth  $\frac{1}{4}$  hour another 100 c.c. distils over: the receiver is removed immediately when 200 c.c. has been collected. Other volatile immiscible liquids, as benzene, toluene, kerosene, and mixtures of these, gave less satisfactory results.—J. P. O.

*Acetic acid and its compounds: The sugar factory as a source of supply for —.* F. Coxon. *Intern. Sugar J.*, 1918, 20, 70–71.

It is suggested that the bagasse of cane sugar factories might be used as a gas producing fuel in a standard producer, the gas being burnt under the boilers after the separation of its by-products by condensation and washing. In the case of factories operating with a surplus of bagasse the latter might be submitted to destructive distillation for the preparation of calcium acetate and acetone.

—J. P. O.

#### PATENT.

*Cane-juice filter: Automatic —.* B. Salinas y Souza, Assignor to A. C. Smith, Habana, Cuba. U.S. Pat. 1,254,160, Jan. 22, 1918. Date of appl., Mar. 23, 1916.

A WIDE, slightly tapering, rotary, tubular screen is mounted with its axis horizontal. Juice is introduced at the narrower end, and the solid matters retained by the screen travel towards the wider end and are there discharged. The rotation of the screen is effected by the weight of the inflowing juice, the latter being delivered into buckets, a ring of which is mounted on the screen inside its narrower end. These buckets discharge their contents into the body of the screen when in their lowest position.—J. H. L.

#### XVIII.—FERMENTATION INDUSTRIES.

*Hops; Behaviour of the bitter substances of — during wort boiling.* W. Wöllmer. *Z. ges. Brauw.*, 1917, 40, 372.

THE bitter flavour of hopped wort and beer is attributed chiefly to the  $\alpha$ -resin, humulone, or its transformation products. The  $\beta$ -resin, lupulone, has a less intense flavour, dissolves less readily in wort, and has a much greater tendency to separate from solution with the proteins coagulated during wort boiling. Humulone also separates to some extent with the coagulated protein, so that the more of the latter a wort contains the greater will be the loss of bitter flavour. During wort-boiling both resins undergo, more or less, transformation into more soluble products of intensely bitter flavour. In the case of humulone, the transformation is favoured by low wort acidity, hence relatively alkaline brewing waters tend to give beers of very bitter flavour. The corresponding transformation of lupulone depends more on the presence of air than on the reaction of the wort. (See also this *J.*, 1916, 648.)—J. H. L.

*Yeast: Action of arsenic compounds on —.* F. Boas. *Z. Gärungsphysiol.*, 1917, 6, 1–12. *Z. ges. Brauw.*, 1917, 40, 199.

FROM experiments with sodium and potassium arsenates and sodium meta-arsenite it is concluded that the sensitiveness of living yeast towards these compounds varies considerably according to the type of yeast and its physiological condition and also to the temperature, low temperatures accentuating the toxic action. Yeast appears to be capable of recovering from the toxic influence of quantities of arsenic compounds which at first inhibit both reproduction and fermentation. Sodium meta-arsenite retards fermentation. In media containing nitrogenous nutriment the initial toxic influence of arsenates on the zymase of living yeast may become transformed, after some hours, into a pronounced stimulating action. (For the influence of arsenates on fermentation by yeast juice see Harden and Young, this *J.*, 1911, 705.)—J. H. L.

*Yeast from thin beers: Microscopic appearance of —.* H. Will. *Z. ges. Brauw.*, 1917, 40, 209–211, 217–220.

YEAST crops obtained in the production of bottom-fermentation "war-beers," from worts of 3–6 gravity, were found to show signs of imperfect nutrition. The yeast deposited in the young beers during storage was in better condition, and might be used in pitching. Yeast employed for such thin worts should be periodically refreshed by cultivation in a small quantity of wort of high gravity.—J. H. L.

*Beer; De-ionisation of iron in wort and —.* A. Reichard. *Kolloid-Zeits.*, 1917, 20, 39–46. *Z. ges. Brauw.*, 1917, 40, 231.

BEER left in contact with iron for 24 hours in presence of air, reacted positively to direct tests for iron in the ferrous and ferric states, but no longer did so after being shaken to remove carbon dioxide. Subsequent addition of hydrochloric acid restored the iron present to its normal reactive state. A similar masking of small quantities of iron in wort or beer may take place whenever the liquid contains suspended matters, e.g., substances coagulated by heating, gluten particles formed on chilling, or even dead yeast cells. Particularly effective in this respect are the particles which separate from beer as the result of intimate contact with air, and which are often so minute as to be detected only by their "Tyndall effect." In all these cases dissolved iron tends to be adsorbed by the suspensoid

matters, and thereupon ceases to respond to the usual tests except after treatment with acid.

—J. H. L.

*Beer; Influence of shaking on the biological stability of* — H. Will. Z. ges. Brauw., 1917, 40, 249—252.

THE results of some experiments on the effect of shaking on beer in bottles, indicate that when there is some air in the bottles agitation may hasten the appearance of cloudiness or turbidity due to the development of wild yeasts. In the complete absence of air, shaking appears to have little or no influence on the period during which the beer remains bright.—J. H. L.

*Saccharin; Non-decomposition of Peligot's during alcoholic fermentation. Some optical properties of this saccharin.* H. Pellet. Bull. Assoc. Chim. Sucr., 1917, 34, 327—330.

CANE molasses diluted ten-fold was treated with a small quantity of Peligot's saccharin (see Berichte, 1880, 13, 196) and fermented by a quantity of yeast equal to that of the sugar present; the rotatory power of the fermented liquid indicated that no destruction of the saccharin had occurred. The rotatory power of Peligot's saccharin  $[\alpha] = +93.5$ , according to Peligot) is greatly reduced in presence of basic lead acetate and even entirely annulled by a sufficient excess of the latter, but it is restored to its normal value after acidification with hydrochloric acid. Contrary to the statement of Cuisinier, the rotatory power of the saccharin is not modified in solutions containing 10% of acetic acid. The treatment described by Muller (this J., 1916, 558), which destroys dextrose, levulose, glucose, arabinose, xylose, and mannose (but not raffinose), reduces the rotatory power of Peligot's saccharin by about one-third, or more if the heating is prolonged beyond 15 mins.—J. H. L.

*Wines; New methods of determining the concentration of hydrogen ions in acid liquids; application to* — M. Duboux. J. Chim. Phys., 1917, 15, 473—501.

THE methods of determining hydron concentration based on catalysis of sucrose hydrolysis (cp. Paul, this J., 1915, 1150), or of the decomposition of ethyl diazoacetate (Fränckel, Z. physik. Chem., 60, 202) are tedious and require considerable skill. The second method is much more sensitive than the first, and has the further advantages that it can be applied to darkly coloured liquids and at ordinary temperatures. The hydron concentration in liquids like wine and beer cannot be calculated from a single determination of electrical conductivity owing to the presence of conducting salts. If the progressive change in the conductivity of a wine in the course of neutralisation is investigated, by means of a series of conductivity determinations after addition of successive increments of alkali, and represented as a curve, the original hydron concentration may be found approximately by a graphical method. It is shown, however, that more accurate results may be obtained by diluting the wine with four volumes of water, and determining the conductivity before and after addition of half the volume of standard alkali required for neutralisation (to litmus paper), the results being calculated with the help of certain factors which are given in tables. The results for 30 wines show an average error of 5% when compared with those by the diazoacetate method, which is regarded as the best standard. It is claimed that determinations by the new method can be completed in ten minutes, and that the principles on which the method is based could be applied to other liquids of fairly uniform composition.—J. H. L.

*Soy ("shoyu"); Yield of products in the preparation of Japanese* — A. Kinoshita. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1121—1142.

THE proportions of ingredients employed are as follows:—soya beans, 0.5 koku; wheat, 0.5; common salt, 0.5, and water 1.0 koku (1 koku = 180.39 litres). The beans, when washed, steeped, and steamed, gain about an equal amount in weight and volume as compared with the original. The wheat, when perched and crushed, increases to about 1.8 times its original volume and loses about 0.16 of its original weight. The mixture of beans and wheat is transformed into "koji," the yield of koji being about 1.6 by volume and 0.9 by weight. The koji is mixed with a salt solution (sp. gr. 1.143—1.152) and stirred occasionally. When the summer comes, active fermentation sets in and after one year, finished "moromi" is obtained, its weight being 85 kwan (1 kwan = 3.75 kilos.). This moromi is pressed, and the liquor is pasteurised, yielding about 1 koku (180.39 litres) of Japanese soy.—J. F. B.

*"Shoyu-moromi" [Japanese soy]; Stimulating actions of neutral salts on the enzymic actions of* — T. Tadokoro. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1114—1124.

THE enzymic activities of Japanese soy have a very important effect on the formation of its proper odour and taste during fermentation. These activities are very much checked by the presence of a large quantity of common salt, such as is usually added in the preparation of the "shoyu." They may, however, be stimulated either by decreasing the quantity of salt or by increasing the temperature during fermentation. The author has found yet another method whereby the inhibitive effect of the salt may be controlled, by the antagonistic action of other neutral salts. Potassium chloride, aluminium chloride, calcium chloride and magnesium chloride are good antagonistic agents; aluminium chloride and calcium chloride, show a powerful stimulating effect on amylase and pepsin in "shoyu-moromi." Better results are obtained by using a mixture of two or more salts together than with a single salt. When the concentration of common salt is 3N, a beneficial result is obtained by adding 0.0001—0.002% of a mixture of antagonistic salts to the common salt, whereby the enzymic activities of the shoyu-moromi are increased by 20%.—J. F. B.

*Mucor Boulard; Influence of different agents on the saccharifying and fermenting powers of* — [in distilleries]. Bettinger. Bull. Assoc. Chim. Sucr., 1917, 34, 254—258. (Cp. Grove, this J., 1914, 562.)

EXPERIMENTS made with *Mucor Boulard* No. 5 (see Fr. Pat. 464,601, this J., 1914, 497) have shown that the development of acidity is greater the larger amount of sugar present and the lower the temperature. The acidity increases also with access of air, for a time, and then diminishes owing to utilisation of the acids by the mould itself. Succinic acid is the only non-volatile acid produced. The volatile acidity is due chiefly to acetic acid, which appears to be formed when the mould is submerged but disappears in surface cultures. Under industrial conditions, i.e., in distillery mashes, added nitrogenous matter at first retards saccharification but later this effect is more than compensated by acceleration of the process. Peptone and asparagine give the best results in respect of saccharification and fermentation by the *Mucor*. Vegetative growth is also greatly promoted by peptone and to an almost equal extent by asparagine and ammonium phosphate, but ammonium sulphate has a relatively slight



effect on growth or saccharification. The influence of added phosphates on saccharification appears to depend chiefly on the nature of the base present; the calcium and potassium salts, and to a smaller extent the ammonium salt, accelerate saccharification, whilst sodium phosphate has scarcely any effect. Potassium phosphate increases vegetative growth much more than the sodium and calcium salts.—J. H. L.

*Pitch: Brewers' —*. F. Eckhardt. Z. ges. Brauw., 1917, 40, 273—276, 281—284, 289—291, 297—300, 306—309, 313—317.

The author deals at length with the characters required in a brewers' pitch, the methods of examination, the changes which resin and pitch undergo on heating, and many practical points in the pitching of casks. The addition of paraffin wax to pitch made from resin and resin-oil improves the quality in several respects; it raises the flash-point, renders the pitch more fluid at temperatures below that of the pitch-kettle, and thus prevents too rapid thickening in the casks and consequent waste of pitch. Paraffin also reduces the loss which pitch undergoes on continued heating. This loss may be considerable when a large surface is exposed; it is due to evaporation of the more volatile constituents, including some of the resin oil, and has the effect of raising the softening point of the pitch and increasing its viscosity. It cannot be entirely prevented, since the resin itself undergoes slow decomposition at the temperatures employed in pitching, and yields, amongst other products, resin-oil and more volatile substances. For this reason a pitch cannot be completely freed, once for all, from volatile matters likely to affect the flavour of beer. If resin is heated under conditions which permit the more volatile decomposition products, but not the resin-oil, to escape, the softening-point of the residual product becomes lowered, and by heating in this way, say at about 300° C., old pitch or resin itself may be converted into a product possessing the physical characters of brewers' pitch made from resin and resin-oil.—J. H. L.

*Notes on alcohols [in microscopy]*. Garnett. See XXIII.

#### PATENTS.

*Yeast: Process for the production of a blended —*. J. A. Stevenson, Burton-on-Trent. Eng. Pat. 113,486, Feb. 23, 1917. (Appl. No. 2633 of 1917.)

BREWERS' yeast is washed and freed from bitterness and pressed. This yeast is then re-fermented in a grain wort, 1 lb. of the yeast being used to 2—2½ lb. of wort prepared from grain of 336 lb. standard, of which at least 50% should be malt. Distillers' yeast is also re-fermented in a separate vessel in a similar manner. After re-fermentation, the surplus wort is removed from each yeast and the two yeasts are then blended in the proportion of about 40% of brewers' yeast to 60% of distillers' yeast and finally pressed.—J. F. B.

*"Beer": Non-intoxicating — and process of brewing the same*. A. Hadley, Bristol. Eng. Pat. 113,560, June 16, 1917. (Appl. No. 8677 of 1917.)

A BEVERAGE closely resembling beer, but containing less than 2% of alcohol, is prepared by boiling a solution of glucose or invert sugar, or both, with hops and yeast, cooling the solution by means of refrigerators, fermenting with brewers' yeast at an initial gravity of about 1016 (6 brewers' lb.), cooling by means of attenuators when the gravity has fallen to about 5 lb., and clarifying the liquid with "finings."—J. F. B.

*Cooling plates for continuous-rectification apparatus*. Eng. Pat. 105,549. See I.

#### XIXA.—FOODS.

*Milk: New constituents of —. III. A new protein, soluble in alcohol*. T. B. Osborne, A. J. Wakeman, C. S. Leavenworth, and O. L. Nolan. J. Biol. Chem., 1918, 33, 243—251.

WHEN caseinogen is separated from milk, it is usually accompanied by another protein. The latter can be extracted from the caseinogen precipitate by treatment with alcohol, in which it is readily soluble. The composition and properties of the new protein are such as to differentiate it from all known types of proteins of vegetable or animal origin. The amount present in milk appears to be too small to be of much technical significance. (See also J. Chem. Soc., Apr., 1918.)—H. W. B.

*Butter and margarine: Detection of artificial colouring matters in —*. A. E. Parkes. Analyst, 1918, 43, 87.

AFTER the Valenta number has been ascertained, the mixture of fat and acetic acid is treated with a few drops of dilute sulphuric acid and allowed to stand until the fat has separated. The lower acid layer is coloured in the presence of artificial colouring matters; red, orange, and yellow are the most common colours used. Natural butter fat under the same conditions gives a colourless or at times a faint blue or green acid layer, the colour of which fades quickly.—W. P. S.

*Inulin: Degradation of — and of the "inulides" in chicory root*. B. Geslin and J. Wolff. Comptes rend., 1918, 166, 428—430.

DURING storage the inulin and the "inulides" undergo degradation, the inulin being gradually converted into the more complex inulides and these being converted into the less complex inulides. (See also J. Chem. Soc., May, 1918.)—W. G.

*Cherry-kernel oil*. Maxwell. See XII.

*Effects of electrolytes on gelatin and their biological significance. I. Effects of acids and salts on the precipitation of gelatin by alcohol*. Fenn. See XV.

*Preservation of plant juices for determination of sugar content*. Spriestersbach. See XVII.

#### PATENTS.

*Bread: Process of making —*. D. Chidlow, Ridgefield, Conn., Assignor to J. F. and W. S. Strachan, Montreal, Canada. U.S. Pat. 1,255,292, Feb. 5, 1918. Date of appl., Jan. 4, 1917.

THE bread is made from whole grain by first removing the outer skin of the grain without injuring its germinating property. The grain is then germinated, and whilst still moist from the germinating process is crushed to form a dough, which is afterwards baked.—J. H. J.

*Bread: Process and machine for making —*. D. Chidlow, Ridgefield, Conn., Assignor to J. F. and W. S. Strachan, Montreal, Canada. U.S. Pat. 1,255,293, Feb. 5, 1918. Date of appl., June 12, 1917.

THE bread is made from whole grain which has its outer skin first removed and is then germinated in tanks in presence of liquid, from which it is afterwards separated and conveyed to crushing machines, where it is crushed to a dough in presence of 15%—25% of its weight of the liquid used in the germinating tanks.—J. H. J.

*Aerated waters; Manufacture of* — J. Wilson, Accrington, Lancs. Eng. Pat. 113,542, Apr. 17, 1917. (Appl. No. 5381 of 1917.)

THE water to be aerated is placed in a closed tank, the charging tube of which is perforated at its lower end with a large number of holes,  $\frac{1}{4}$  in. in diameter. The bottom of the tube is closed by a valve. The carbon dioxide used for aerating escapes slowly through the small perforations, the valve being kept closed by the gas pressure during the aeration. When aeration is completed, the pressure in the tank opens the valve, and the water is discharged through the full bore of the tube which is adapted, externally to the tank, to act as a siphon.—J. H. J.

### XIXB.—WATER PURIFICATION ; SANITATION.

*Water; Colloid-chemical methods for estimating the hardness of* — L. Berczeller. Biochem. Zeits., 1917, 84, 149—155.

POSSIBLY a method of estimating the hardness of water might be based on the facts that the surface tension of soap solutions is diminished by addition of alkalis, but increases again on subsequently adding small amounts of calcium or magnesium salts. (See also J. Chem. Soc., April, 1918.)  
—S. B. S.

*Vanadium; Detection of small quantities of* — in water. V. L. Meaurio. Anal. Soc. Quim. Argentina, 1917, 5, 185—189.

A SOLUTION of 0.2 gram. of diphenylamine in 100 c.c. of water in presence of hydrochloric acid gives a violet coloration with aqueous solutions of vanadium compounds. The coloration is unaffected by the presence of nitrates, iron, or titanates, and the test will detect the presence of vanadates in solutions of 0.0002% strength. (See also J. Chem. Soc., Apr., 1918.)—A. J. W.

*Disinfectant action of quinine alkaloids on pathogenic bacteria.* R. Bieling. Biochem. Zeits., 1918, 85, 188—211.

THE alkaloids investigated were quinine, optochine, eucupine, and iso- $\alpha$ -etyl-, decyl-, and dodecylhydrocupreines. These have a specific disinfectant action on bacilli of diphtheria, splenic fever, and tetanus. Eucupine and iso cetylhydrocupreine are generally the most effective. (See also J. Chem. Soc., May, 1918.)—S. B. S.

### PATENTS.

*Water; Process of softening* — *Water-softening apparatus.* G. H. Uecke, Minneapolis, Minn. U.S. Pats. (A) 1,255,358 and (B) 1,255,359, Feb. 5, 1918. Dates of appl., Feb. 19 and Mar. 21, 1917.

(A) A SUBSTANCE which has been used for water softening is regenerated by displacing the water in which it is immersed by a rising column of a regenerating solution which is denser than pure water. The regenerating solution is used at first weak and subsequently strong. (B) The apparatus consists of a tank holding the softening material and of a tank holding a regenerating solution such as brine. There is a pipe connection from the brine tank to the bottom of the softening tank, so that the brine may be applied as a rising column through the softening material. An independent draw-off pipe for the brine is situated at the upper level of the softening material.—J. H. J.

### XX.—ORGANIC PRODUCTS ; MEDICINAL SUBSTANCES ; ESSENTIAL OILS.

*Opium alkaloids; Microchemistry of some* — L. van Itallie and J. van Toorenburg. Pharm. Weekblad, 1918, 55, 169—178.

AN account of tests applicable to the alkaloids pseudo-morphine, protopine, tritopine, cryptopine, laudanine, laudanidine, and laudanosine. (See also J. Chem. Soc., Apr., 1918.)—A. J. W.

*Ilex paraguayensis [yerba maté] and its mixtures with other varieties of ilex and herbs not containing caffeine; Volumetric method of identifying* — A. Sabatini. Anal. Soc. Quim. Argentina, 1917, 5, 192—198.

THE power of caffeine to reduce ferric salts is the basis of a volumetric method for detecting adulteration of *Ilex paraguayensis*, ammonium thiocyanate being employed as indicator.—A. J. W.

*Adonis vernalis; Examination of the leaves of* — F. W. Heyl, M. C. Hart, and J. M. Schmidt. J. Amer. Chem. Soc., 1918, 40, 436—453.

TINCTURE of *Adonis vernalis*, or false hellebore, has been used in medicine in recent years. The activity is usually assigned to a digitalis-like glucoside, "adonidin," which Merck's factory has placed on the market, obtaining this principle from the root, according to their latest report. A large consignment of leaves, from Leipzig, has now been examined. The tincture is slightly more toxic than digitalis tincture, but no appreciable quantity of adonidin or any glucoside is present. Most of the toxicity is due to an unknown base, precipitable by phosphotungstic acid, whilst considerable quantities of adonitol, which is the pentitol corresponding with ribose, are present as well. (See also J. Chem. Soc., May, 1918.)  
—J. C. W.

*Oxalyl chloride as a reagent in organic chemistry. II. Preparation of aromatic acid anhydrides. Reaction with alcohols.* R. Adams, W. V. Wirth, and H. E. French. J. Amer. Chem. Soc., 1918, 40, 424—431.

A VERY good method for the preparation of aromatic acid anhydrides consists in adding oxalyl chloride (1.2 mol.) to a boiling benzene solution of the acid (2 mols.). Examples are given. Just as phenols react with oxalyl chloride in the presence of pyridine at 0° C. to give oxalates, so primary and secondary alcohols give these esters in 50% yields on an average. Tertiary alcohols scarcely react at all. The preparation of oxalates of such complicated alcohols as benzoin and piperonoin may therefore be achieved. (See also J. Chem. Soc., May, 1918.)—J. C. W.

*Acetic, propionic, and butyric acids; Determination of* — R. D. Crowell. J. Amer. Chem. Soc., 1918, 40, 453—460.

THE method depends on the fact that most of the butyric acid, and some propionic acid, can be extracted by means of kerosene after saturating the solution with a mixture of calcium chloride and a little potassium chloride. (See also J. Chem. Soc., May, 1918.)—J. C. W.

*"White precipitate"; Preparation, properties, and analysis of* — I. M. Kolthoff. Pharm. Weekblad, 1918, 55, 208—218.

A COMPARISON of the methods for preparing "white precipitate" given in the Dutch, German, English, Belgian, Swiss, and U.S. Pharmacopœias. The author is of opinion that the Dutch method is the most satisfactory.—A. J. W.



*Bromination of p-nitrotoluene.* Brewster. See III.

*Non-decomposition of Peligot's saccharin during alcoholic fermentation. Some optical properties of this saccharin.* Pellet. See XVIII.

*Disinfectant action of quinine alkaloids on pathogenic bacteria.* Bielng. See XIXB.

*Notes on alcohols [in microscopy].* Garnett. See XXIII.

*Limitations of the Kjeldahl method of determining nitrogen.* Brill and Agcaoili. See XXIII.

#### PATENTS.

*Acetic aldehyde [from acetylene]; Manufacture of —.* H. Dreyfus, Basle, Switzerland. Eng. Pat. 105,064, Mar. 6, 1917. (Appl. No. 3272 of 1917.) Under Int. Conv., Mar. 6, 1916.

ACETYLENE, freed from impurities such as hydrogen sulphide, hydrogen phosphide, and ammonia, is passed under an increased pressure of about 0.5 atmos. into dilute sulphuric acid containing a compound of mercury, *e.g.*, the oxide, under the following conditions: The solution contains between 10 and 15% total acid and between 1 and 10% (preferably between 3 and 6%) of mercury, the compound having been brought completely into solution by heating the liquid; the acetylene is introduced with strong agitation between 25° and 40° C., slowly at first until the mercury compound precipitated has become grey or greyish-black, and then as rapidly as possible, but avoiding non-absorption; the acetaldehyde formed is distilled off at intervals by raising the temperature to not above 50°–60° C.; the temperature is then reduced again for further absorption, and the process repeated until the absorbing power of the solution has diminished considerably, when the solution is heated above 60° C. to distil off the last traces of acetaldehyde. The mercury residue may be regenerated by treatment with oxidising agents. A suitable absorption apparatus consists of a cylindrical iron vessel, *e.g.*, of 1000–5000 litres capacity, having its inside covered with lead which is treated by heating with sulphuric acid, and, preferably, an oxidising agent, *e.g.*, nitric acid, thus forming a layer of compounds of lead which protects the lead against amalgamation with the mercury and does not dissolve in the absorbing liquid. The stirrer is covered with lead treated in a similar manner. For heating the solution, the apparatus may be provided with a double bottom, with a tank or bath, or pipes of lead treated as above may be introduced into the interior. Yields of 90–95% or more of acetaldehyde can be obtained from the acetylene introduced, amounting to 5–10 times or more the weight of mercury compound employed.—L. A. C.

*Physiologically active substance and new compounds containing nitrogen from ovaries, corpus luteum, or placenta; Manufacture of new —.* O. Imray, London. From Soc. Chem. Ind. in Basle, Switzerland. Eng. Pat. 113,311, Feb. 13, 1917. (Appl. No. 2151 of 1917.)

PHYSIOLOGICALLY active hormones can be extracted from the tissue of the ovaries, *corpus luteum*, or placenta by several methods, as illustrated by the following example: Pieces of human placenta are dried in a vacuum at a low temperature and powdered, and 5000 grms. of the product extracted with ether or a similar solvent in a continuous extraction apparatus. The solvent is evaporated off and phosphatides precipitated from the residue by the addition of four times its volume of acetone. The liquid separated from the precipitate is freed from acetone in a vacuum. The residue is mixed with cold methyl or ethyl alcohol (*e.g.*, about half a litre) and the insoluble portion (the greater part of

the cholesterol and its esters) separated. The alcohol is evaporated off from the filtrate and the residue purified by fractional distillation under high vacuum. Under a pressure of 0.06 mm. of mercury, a mixture of hormones and cholesterol and its esters distils over between 190° and 210° C. This is purified by further treatment with cold alcohol (*e.g.*, 100 c.c.), and redistillation. The product is a light viscid oil containing only carbon, hydrogen, and oxygen, and showing the typical reactions of cholesterol. Analysis gave C=81.33–81.62%, H=11.32–11.49%. It is not soluble in water, but is absorbed by the colloidal aqueous solution of the phosphatides. The substance promotes the growth of the uterus, of the ovaries, of the oviducts, and of the vagina, and increases the secretion of the mammary glands. The phosphatides isolated appear to be pentamino-diphosphatides; they react with permanganate as unsaturated compounds and contain nitrogen and phosphorus in the proportion of 5:2. They are distinct from the substances extracted from the *corpus luteum* as described in Eng. Pat. 10,877 of 1915 (this J., 1916, 943).—L. A. C.

*Quinine group; Manufacture of therapeutically valuable compounds of the —.* O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 112,974, Jan. 29, 1917. (Appl. No. 1441 of 1917.)

AN alkaloid of the quinine group or a derivative thereof is allowed to interact in molecular proportions with CC-diallylbarbituric acid or a CC-allylalkylbarbituric acid in the form of the free acid or a salt, in presence or absence of a suitable solvent or diluent, when therapeutically valuable compounds are produced.—F. W. A.

*Arsenobenzene derivatives; Preparation of aqueous solutions of —.* A. Mouneyrat, Paris. Eng. Pat. 112,984, Feb. 1, 1917. (Appl. No. 1650 of 1917.)

STABLE aqueous solutions of arsenolene derivatives are obtained by dissolving their soluble salts in aqueous solutions of reducing sugars, such as glucose (dextrose), levulose, mannose, galactose, etc., contained in bottles filled with nitrogen; the solutions are preserved in sealed capsules in absence of light, if necessary with addition of local anaesthetics. The presence of these reducing substances avoids the oxidation of the arseno compound, with the resulting increased toxicity. The use of dextrose solutions permits of the administration by the intra-muscular method.—F. W. A.

*Rendering liquids radio-active; Means for —.* O. A. Elias, London. Eng. Pat. 113,483, Feb. 22, 1917. (Appl. No. 2600 of 1917.)

A CARTRIDGE or other receptacle made of cotton or other absorbent material which has been treated with a radio-active salt, is rendered impervious to water by means of a coating of varnish consisting of a condensation product of formaldehyde and phenol in equimolecular proportions dissolved in alcohol. After stoving for 2 hours at a suitable temperature, the cartridge is ready for introduction into the bottle of aerated water, etc., and is preferably attached to the glass tube of a siphon bottle. A coating of fluorescent zinc sulphide may be applied to the cartridge to indicate the presence of the radioactive emanation by being made luminous.—C. A. M.

*Treating liquid [to produce colloidal lungslie oxide]; Method of and apparatus for — for curative and therapeutical purposes.* W. S. Simpson, Westminster. Eng. Pat. 113,505, Mar. 5, 1917. (Appl. No. 3244 of 1917.)

DISTILLED water or other liquid is exposed to the action of the light and emanations from an arc

struck between a pair of tungsten electrodes. The tungsten is slowly converted into tungstic oxide ( $\text{WO}_3$ ) in such a fine state of division that it remains in permanent suspension in the liquid. The apparatus consists of a casing in the lower part of which is a circular opening having walls extending above and below the bottom of the casing, and under which is placed the arc feeding mechanism holding the tungsten electrodes. Inside the casing and above the arc is a hollow vertical spindle provided at its lower end with a hollow circular perforated cup and connected at its upper end with a reservoir. The spindle is rotated by a motor which also drives a circulating pump. The spindle on being revolved causes a film of liquid coming from the reservoir to be propelled by centrifugal force against the sides of the casing and it flows down forming a curtain of liquid directly over the arc. From the bottom, the liquid is pumped back to the reservoir and is continuously circulated until sufficiently impregnated, when it is drawn off. It may then be aerated. A current of fifteen amperes and a flow of ten galls. per hr. gives a satisfactory result.

—L. A. C.

*Isovaleric acid; Manufacture of derivatives [esters] of —.* G. B. Ellis, London. From Chem. Fabr. von Heyden A.-G., Radebeul, Saxony. Eng. Pat. 113,983, Dec. 9, 1916. (Appl. No. 17,759 of 1916.)

ESTERS of isovaleric acid with hydroxy acids of the fatty series (*e.g.*, glycollic acid, lactic acid, or those containing an aryl group, such as mandelic acid) are prepared by treating the fatty acids with isovaleric acid (preferably in the presence of dehydrating agents), or with isovaleric anhydride or chloride, or by treating salts of isovaleric acid with the halogen-fatty acids or their salts. Instead of isovaleric acid and its derivatives, halogen-isovaleric acids (especially  $\alpha$ -bromoisovaleric acid) may be used. The esters may be neutralised with inorganic or organic bases, *e.g.*, sodium or calcium carbonate, morphine, quinine, etc. Methods of preparation of isovaleryl-inactive-mandelic acid, both from isovaleric chloride and from isovaleric acid, its calcium and quinine salts; of isovalerylglycollic acid, both from glycollic acid and from sodium chloroacetate, and of  $\alpha$ -bromoisovalerylmandelic acid are given as examples.—L. A. C.

*Pharmaceutical product [p-benzylphenyl carbamate].* W. Kropp, Elberfeld, Germany., Assignor to Synthetic Patents Co., New York. U.S. Pat. 1,252,452, Jan. 8, 1918. Date of appl., July 21, 1915. Renewed Nov. 23, 1917.

CARBONYL derivatives of phenols containing in the nucleus a substituent, R, which has more than one carbon atom, and having the following general formula:  $\text{R.C}_6\text{H}_4\text{.O.CO.R'}$ , in which R' is  $\text{OC}_6\text{H}_4\text{R}$  or  $\text{NXX'}$ , X and X' representing hydrogen or an alkyl group, are white, practically tasteless and odourless powders, generally soluble with difficulty in water, and are valuable anæsthetic remedies. *p*-Benzylphenylcarbamate, which separates from alcohol in crystals of m. pt.  $144^\circ\text{C}$ ., is particularly claimed.—F. W. A.

*Iodine and iodine compounds [tetra-iodohexamethylenetetramine]; Method and means for applying —.* G. B. Pegram, New York. U.S. Pat. 1,255,335, Feb. 5, 1918. Date of appl., June 23, 1917.

AN addition compound of iodine and hexamethylenetetramine (tetra-iodohexamethylenetetramine) may be brought into solution by the addition of an alkali halide (potassium iodide) to the solvent. One part of tetra-iodohexamethylenetetramine and three to seven parts of potassium iodide may be mixed to form a dry tablet.—L. A. C.

*Acetylsalicylic acid; Process of forming methyl ester of —.* L. Thorp, Detroit, Mich. U.S. Pat. 1,255,950, Feb. 12, 1918. Date of appl., Aug. 21, 1916.

ONE equivalent of methyl salicylate and slightly more than one equivalent of acetic anhydride are heated with an alkali acetate to  $90^\circ$ – $100^\circ\text{C}$ . for 10–20 hrs.; alcohol is added, and then hot water until a slight permanent turbidity is produced. The solution is cooled and the product separated by filtration.—L. A. C.

*Ethyl-isopropyl-barbituric acid.* L. Thorp, Detroit, Mich. U.S. Pat. 1,255,951, Feb. 12, 1918. Date of appl., Jan. 20, 1917.

ETHYLISOPROPYLBARBITURIC acid (ethyliso-propylmalonylurea)  $(\text{C}_2\text{H}_5)_2(\text{C}_3\text{H}_7)\text{C}(\text{CO}\cdot\text{NH})_2\text{CO}$ , crystallises from water as snow-white crystals, m.pt.  $197^\circ$ – $198^\circ\text{C}$ . It is readily soluble in alcohol or ether; forms salts with the alkali metals which are readily soluble in water; and has a faint bitter taste. The use of the acid and of its sodium salt as hypnotics is claimed.—L. A. C.

*Anæsthetic bodies and method of preparing the same.* Parke, Davis, and Co., Assignees of L. Thorp, Detroit, Mich., and E. A. Wildman, Indianapolis, Ind., U.S.A. Eng. Pat. 105,744, Feb. 27, 1917. (Appl. No. 2908 of 1917.) Under Int. Conv., Apr. 17, 1916.

SEE U.S. Pat. 1,193,649 of 1916; this J., 1916, 979.

*Manufacture of a difficultly soluble complex compound [dimethylphenylbenzyl ammonium zinc chloride] from dimethylaniline, benzyl chloride, and zinc chloride.* Eng. Pat. 104,676. See III.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Spectral selectivity of photographic deposits.* L. A. Jones and R. B. Wilsey. Phot. J., 1918, 58, 70–81.

IN the usual method of determining plate speeds and other constants, densities are measured visually. As the sensitiveness of ordinary printing materials lies chiefly in the blue, violet, and ultra-violet, if a negative possesses any selective colour absorption its visual densities will not correctly represent its action when used for making prints. The authors consider this subject from the theoretical standpoint and suggest two other methods of measuring the various steps of a sensitometer strip. The values obtained by these when plotted against logarithms of exposures lead to curves and constants similar to the usual "visual" ones, but more nearly representing the actual qualities for printing purposes of the strip or of a negative obtained under the same conditions of development. The constants are, of course, different for different printing materials unless they have the same distribution of spectral sensitiveness. In the first method, that of "photographic density," this is defined as being the logarithm of the ratio of the intensities required to produce equal effects in the same time on the printing material in question (a) through the density being measured and (b) by direct exposure. This method is a null one, avoiding any effect due to the failure of the reciprocity law; it takes no account of the nature of the sensitiveness curve of the printing material. In the second method, that of "effective printing density," this is also considered. In one way of obtaining the effective printing density curve, three preliminary curves are required, the usual



visual sensitiveness curves of the negative and of the printing material and that representing the effect of printing through the negative sensitometer strip on to the printing material: the development of the two latter must be the same. The combination of these three curves gives the effective printing density curve of the negative sensitometer strip for the particular set of conditions employed. The colour coefficient, the ratio of the photographic gamma to the visual gamma, is considered in some detail.—B. V. S.

*Photographic intensification; Sensitometry of —.* A. H. Nietz and K. Huse. *Phot. J.*, 1918, 58, 81—93.

THE object of intensification of a negative is to increase contrast for printing purposes, and as the sensitiveness of printing materials is not uniform over the spectrum (including the ultra-violet) the amount of intensification produced by a process is affected by any colour change that may occur in the image. Taking into account the possibility of colour in the original negative as well as after intensification, the authors divide intensifying processes into four general classes according to the relationship between visual and photographic contrasts before and after the process, the essential being only that the photographic or effective printing contrast shall be greater in the intensified negative than in the original. In the experimental work the methods suggested by Jones and Wilsey (see preceding abstract) are adopted. A list of twenty intensifying methods is given and from these a number were selected as representative and examined in more detail as to both visual effect and photographic effect. They include four mercury bleaching methods with various blackening agents, Piper's chromium intensifier, two ferricyanide bleaching methods with sodium sulphide to blacken, Desalme's copper chloride-sodium stannite process, and a permanganate-hydrochloric acid-sodium stannite process. A number of data and curves are given, but it is noted that these refer only to the sensitive materials used and the particular method of development of the negative, since the effect is dependent not only on the intensifying process and the printing material, but also on the character of the original negative deposit. For a similar reason, also, certain intensifiers were not examined, as they are not practicable on the emulsion used. Colour coefficients of various intensifiers, the degree of increased contrast (*i.e.*, the ratios of photographic gammas of intensified and original negatives), and similar factors are also considered.—B. V. S.

*Scattering media: Behaviour of — in fully diffused light.* H. J. Channon, F. F. Renwick, and B. V. Storr. *Proc. Roy. Soc.*, 1918, A, 94, 222—227. *Phot. J.*, 1918, 121—134.

THE paper deals with the relationships existing between the amount of diffuse light incident on a sheet of diffusing medium, that part of it which is rejected (*i.e.*, returned in opposite directions to the incident light), that which is absorbed, and that which is transmitted, including the case when the source of light is a sheet of diffusing medium in contact with the sheet being examined. A mathematical solution of the problem is presented and experimental data are given in support of the theoretical deductions.—B. V. S.

#### PATENTS.

*Chemical substances [developers, etc.] for use in photography; Preparation of —.* A. de Brayer, Paris. *Eng. Pat.* 105,920, Apr. 24, 1917. (*Appl.* No. 5732 of 1917.) Under Int. Conv., Apr. 25, 1916.

IN order to indicate when deleterious substances

such as developers, fixers, etc., have been completely removed from photographic plates, paper etc., by washing, a suitable dye is incorporated with the substance. This dye colours the film and the last traces of colour disappear only with the last traces of the substance. The mixture is prepared as a paste by grinding together with a viscous, water-soluble substance such as glycerin or a solution of sugar, glucose, or alkali silicate. —B. V. S.

*Coloured pictures; Production of —.* W. F. Fox, Whitestone, N.Y., Assignor to Natural Color Pictures Co., New York. U.S. Pat. 1,256,675, Feb. 19, 1918. Date of appl., Jan. 13, 1917.

Two colour-selection negatives are used. A print is made from one of them and toned bluish-green with Prussian blue. A second print from the other negative is superposed on the same surface in register with the first and toned yellow with a uranium salt or a uranium salt and a basic dye. —B. V. S.

*[Photographic] developing composition.* T. J. Brewster, Chester, Pa. U.S. Pat. 1,255,508, Feb. 5, 1918. Date of appl., Feb. 25, 1915.

SEE *Eng. Pat.* 9331 of 1915; this J., 1916, 907.

## XXII.—EXPLOSIVES; MATCHES.

*Reaction of aromatic nitro compounds.* Olivier. See III.

#### PATENT.

*Initial igniter.* [Nitropentaerythritol and lead azide.] O. Matter, Troisdorf, Assignor to Vereinigte Köln-Rottweiler Pulverfabriken, Berlin, Germany. U.S. Pat. 1,251,147, Jan. 22, 1918. Date of appl., Apr. 17, 1913.

SEE *Fr. Pat.* 451,925 of 1912; this J., 1913, 674.

## XXIII.—ANALYSIS.

*Alcohols [in microscopy]; Notes on —.* H. Garnett. *Pharm. J.*, 1918, 100, 127.

ABSOLUTE alcohol exposed to the air for a few days will absorb several per cent. of moisture, which may seriously impair its usefulness for dehydrating purposes. A rapid method of detecting such deterioration is to mix the alcohol with pure oil of cedarwood, *Juniperus virginiana* (not the so-called thickened oil used for immersion lenses); absolute alcohol will mix in all proportions without cloudiness. Alcohol of 98.2%, which is sufficiently strong for most purposes, also mixes without cloudiness at 15.5°C., but if the temperature is lowered only 1° turbidity is produced. Alcohol which has fallen below this standard of strength may be made practically anhydrous, without distillation, by adding a small quantity of powdered calcium carbide and leaving it for a few days in a warm place; the disagreeable odour of alcohol treated in this way may be eliminated by shaking with a trace of potassium permanganate. Ethyl alcohol may be substituted for methyl alcohol in certain staining solutions, *e.g.*, Delafield's hamatoxylin stain, but for others, such as Leishman's blood stain, methyl alcohol appears to be indispensable. Terpeneol,  $C_{10}H_{15}OH$ , has valuable properties as a clearing agent and may with advantage replace oil of bergamot and oil of cloves. Where desirable, for rapid penetration, it may be diluted with a little xylene.—J. H. L.

*Nitrogen: Limitations of the Kjeldahl method of determining* —. H. C. Brill and F. Agcaoili. *Philippine J. Sci.*, 1917, 12 A, 261—265.

THE Kjeldahl method of determining nitrogen gives low results with pyridine, piperidine, quinoline, isoquinoline, hydroxyquinoline, pyrrole, and in some cases with nicotine, apparently owing to the formation of sulphonic derivatives which resist decomposition. In the case of most of these compounds it is not possible to obtain the theoretical quantities of ammonia even by increasing the period of heating to 10 hours, and using potassium sulphate with different quantities of mercuric oxide. In the case of pyridine, however, good results may be obtained by the Gunning-Arnold modification by continuing the heating for a considerable time after the solution becomes clear. When mercury is used it is necessary to precipitate it subsequently as sulphide, since otherwise loss of nitrogen may occur owing to the difficulty of decomposing the ammonium mercury compound. If sodium sulphate be substituted for potassium sulphate in the digestion, the results are too low.—C. A. M.

*Chlorides: Titration of* — by Volhard's method. I. M. Kolthoff. *Z. anal. Chem.*, 1917, 56, 568—576.

WHEN chlorides are precipitated in nitric acid solution by an excess of silver nitrate, the precipitate absorbs about 0.7% equivalent of silver, and a corresponding correction should be made when using Volhard's method. To obtain a sharp endpoint, the mixture of chloride and an excess of silver nitrate should be diluted to a definite volume, shaken, the precipitate allowed to settle, and the excess of silver then titrated with thiocyanate solution in an aliquot portion of the clear liquid; the above-mentioned correction must be applied. Satisfactory results may also be obtained if the titration is interrupted at the first change in colour of the indicator; the mixture is then stirred until the precipitate has collected together, and the titration completed. Addition of ether or other immiscible liquid is not recommended.—W. P. S.

*Phosphoric acid. Determination of* —. Modification of the citrate method. J. Grossfeld. *Z. anal. Chem.*, 1918, 57, 28—33.

THE hydrochloric acid solution of the sample (fertiliser, ash, etc.) is treated with an excess of ammonium oxalate solution, a few drops of methyl orange solution are added, and saturated ammonium acetate solution is run in until the colour of the indicator changes from red to yellow. The mixture is diluted to 100 c.c., mixed, filtered through a "kieselguhr" filter (this J., 1916, 1105), an aliquot portion of the filtrate is treated with 5 c.c. of 20% citric acid solution, and the phosphoric acid is precipitated in the usual way as ammonium magnesium phosphate.—W. P. S.

*Hydrocyanic acid: Detection and determination of small quantities of* —. I. M. Kolthoff. *Z. anal. Chem.*, 1918, 57, 1—15.

THE sensitiveness of the various tests for hydrocyanic acid is as follows, expressed in mgrms. CN per litre of solution: Prussian blue, 2; ferric thiocyanate, 0.1; picric acid, 1; guaiacum, 0.004; phenolphthalein, 0.05; silver, 0.03; iodine-starch, 0.1. (See also this J., 1918, 122 A.) —W. P. S.

*Tungsten: Colorimetric estimation of* —. Travers. *Comptes rend.*, 1918, 166, 416.

IN the colorimetric estimation of tungsten by titanous chloride, the solution should not contain more than 0.1 gm. of the metal in 100 c.c. and its acidity should not exceed N/10. Vanadium, phosphorus, and molybdenum must be removed before the method can be used. (See further J. Chem. Soc., May, 1918.)—W. G.

*Thorium: Oxidimetric determination of* — precipitated as oxalate. F. A. Gooch and M. Kobayashi. *Amer. J. Sci.*, 1918, 45, 227—230.

THORIUM may be determined by titrating its oxalate with permanganate solution. The thorium oxalate should be precipitated by adding the thorium salt solution to the oxalic acid solution; in the reverse procedure, there is a tendency to the formation of basic thorium compounds, especially in hot solutions, and the titration results are less reliable. (See also J. Chem. Soc., May, 1918.)—W. P. S.

*Effect of low-temperature oxidation on the hydrogen in coal and the change in weight of coal on drying.* Katz and Porter. See IIA.

*Compressibility of natural gas and its constituents, with analyses of natural gas from 31 cities in the United States.* Burrell and Robertson. See IIA.

*[Determination of] calorific value of petrol.* Blount. See IIA.

*Determination of sulphur in petrol.* Blount. See IIA.

*Comparative values of coke-oven benzols.* Haigh and Lamb. See III.

*Reaction of aromatic nitro compounds.* Olivier. See III.

*Determination of sulphur in pyrites.* Karaoglanow and others. See VII.

*Determination of chlorates and hypochlorites.* Rupp. See VII.

*Determination of hypobromite and bromate, or hypoiodite and iodate, in mixtures.* Rupp. See VII.

*Determination of iodates in presence of bromates.* Rupp. See VII.

*Antimony dioxide.* Von Szilagyi. See VII.

*Estimation of proportions of quartz, tridymite, etc., in silica bricks.* Scott. See VIII.

*Le Chatelier's boiling test for Portland cement.* Hattori and Fujisawa. See IX.

*Analysis of copper.* Woodcock. See X.

*Determination of lead as phosphate and its separation from antimony.* Vortmann and Bader. See X.

*Modified acetic acid reagent for Valenta tests [of fatty oils].* Parkes. See XII.

*Testing of rubber.* Van Rossem. See XIV.

*Preservation of plant juices for determination of sugar content.* Spriestersbach. See XVII.

*Determination of water in molasses and other sugar factory products by the distillation method.* Van der Linden and others. See XVII.

*De-ionisation of iron in wort and beer.* Reichard. See XVIII.

*New methods of determining the concentration of hydrogen ions in acid liquids, applicable to wines.* Duboux. See XVIII.

*Detection of artificial colouring matters in butter or margarine.* Parkes. See XIXA.

*Colloid-chemical methods for estimating the hardness of water.* Berczeller. See XIXB.

*Detection of small quantities of vanadium in water.* Meaurio. See XIXB.

*Microchemistry of some opium alkaloids.* Van Itallie and Van Toorenburg. See XX.

*Volumetric method of identifying *Ilex paraguayensis* and its mixtures with other varieties of *Ilex* and herbs not containing caffeine.* Sabatini. See XX.



*Determination of acetic, propionic, and butyric acids.* Crowell. See XX.

#### PATENTS.

*Metals; Machine for testing the hardness of —.*  
D. Chattaway, Farsley, Yorks. Eng. Pat. 114,126, Dec. 8, 1917. (Appl. No. 18,215 of 1917.)

THE specimen to be tested is mounted on a table forming the head of a vertical screwed spindle, so that it may be adjusted vertically. The hardened ball which is forced against the article to be tested is mounted on a spindle sliding vertically in an arm projecting horizontally from the upright standard of the machine, and the spindle is attached to one end of a lever fulcrumed on a knife edge. The other end of the lever is connected by a vertical link to the short arm of the main beam of the machine, which rests horizontally on a knife edge at the top of the upright standard. A weight is adapted to be moved along the beam by means of a suitable hand wheel and screw, so that the load on the ball may be increased uniformly from zero to a maximum. The hardness of the specimen is estimated from the diameter of the indentation produced in it.—W. F. F.

*Hardness-testing machine.* F. L. Morse. Ithaca, N.Y. U.S. Pat. 1,255,913, Feb. 12, 1918. Date of appl., June 26, 1914.

IN a machine for measuring hardness, the lower abutment for supporting the specimen is mounted in a recess in a frame and supported by a spiral spring. An indenting tool to which pressure is applied, is mounted so as to slide in the upper part of the frame. The upper and lower portions of the frame are connected by guide-bolts, one of which is hollow, and a cylinder connected rigidly to the spring-pressed abutment slides within the bolt. A piston fitting in the cylinder is carried by the indenting tool so that the relative movement of the piston in the cylinder is a measure of the indentation produced in the specimen. The cylinder contains a heavy liquid at the bottom and a lighter liquid at the top, and a measuring gauge is connected to the heavier liquid to indicate the displacement. Another indicating device is provided to measure the movement of the abutment relatively to the frame, which is proportional to the force exerted on the specimen.—W. F. F.

*Laboratory [steam-generating] apparatus.* K. P. McElroy, Washington, D.C. U.S. Pat. 1,254,048, Jan. 22, 1918. Date of appl., May 16, 1917.

WATER is constantly supplied to a receptacle closed at the bottom and having a concentric vertical overflow tube, the inlet of which is near the top of the receptacle. A tube extends horizontally from the bottom of the receptacle and has two substantially right-angled bends in a vertical plane, so that its free end is inclined slightly upwards at such a height that the water level is in this part of the tube. Heat is applied by a burner to the water in the inclined tube and the steam escapes at the open end.—W. F. F.

*Filter-funnel.* F. S. Hawken, St. Louis, Mo. U.S. Pat. 1,257,956, Feb. 26, 1918. Date of appl., Apr. 30, 1917.

IN a filtering funnel the upper part of the interior surface of the cone is smooth so that the filter paper adheres to it, and the lower part of the cone is made with thinner walls so that an internal shoulder is formed at the junction. The lower part is roughened internally and provided with grooves and projections just below the shoulder.  
—W. F. F.

*Radiation pyrometers.* A. Hilger, Ltd., and T. R. Merton, London. Eng. Pat. 113,863, Mar. 31, 1917. (Appl. No. 4686 of 1917.)

### Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

#### I.—GENERAL; PLANT; MACHINERY.

##### APPLICATIONS.

Brownell. Apparatus for drying, etc., materials. 5629. Apr. 2. (U.S., Apr. 6, 1917.)

Donald. Kilns. 6233. Apr. 12.

Gieve. Device for blending or mixing. 6172. Apr. 11.

Lonsdale. Non-conducting composition for insulating pipes, etc., in chemical works. 5919. Apr. 6.

Ransome and Marles Bearing Co., and Marles. Grinding machines. 5651. Apr. 3.

Schär. Grinding-mills. 6305. Apr. 13. (Switzerland, May 3, 1917.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

105 (1917). Van Musschenbroek. Drying wet or watery materials, especially fruits and vegetables. (114,442.) Apr. 17.

5915 (1917). Major. Distillation or evaporation of liquids. (114,353.) Apr. 10.

8697 (1917). Kirkham, Hulett, and Chandler, and Hersey. Apparatus for effecting intimate contact between liquids and gases. (114,370.) Apr. 10.

11,459 (1917). Van Iterson and Kuypers. Cooling-towers of reinforced concrete. (108,863.) Apr. 17.

17,974 (1917). Braam. Apparatus for use in preparing solutions of gases. (111,858.) Apr. 17.

#### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

Allgem. Ges. f. Chem. Ind. 6067. See III.

Beswick. Gas generators or producers. 5974. Apr. 8.

Cross, and Latham, Harris, and Co. Treating or refining petroleum. 5704. Apr. 3.

Davis. Coke-ovens, and carbonisation of coal. 5597. Apr. 2.

Freear and Schauer mann. Liquid fuel. 5805. Apr. 4.

Harger. 5594. See VII.

Helps. Manufacture of a producer-gas for lighting, heating, and power. 5701. Apr. 3.

Pearson and Son, and Smith. 6280. See III.

Rew. Retorts for gas production. 6077. Apr. 10.

Rew. Retorts. 6313. Apr. 13.

Simpson. Heating flues of chambered ovens or retorts for producing gas and coke or fuel. 6230. Apr. 12.

Ten Bosch. Removing water from peat. 5692. Apr. 3. (Holland, Feb. 21.)

Walker. Gas-generating and heating apparatus. 5736. Apr. 4.

West and Wold. Charging retorts or chambers for destructive distillation of carbonaceous materials. 5788. Apr. 4.

##### COMPLETE SPECIFICATIONS ACCEPTED.

4873 (1917). Drefflein and Flinn. Gas-producing and consuming plants. (114,474.) Apr. 17.

5037 (1917). Macintosh and Hunt. Manufacture of coal gas, etc. (105,563.) Apr. 17.

5272 (1917). Euler. Gas generators. (114,491.) Apr. 17.

8789 (1917). Anderson. See III.

16,290 (1917). Thuman (Chrisman). Gas wash-boxes. (114,397.) Apr. 10.

19,019 (1917). Boorne. Wet carbonising apparatus and process. (114,603.) Apr. 17.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Allgem. Ges. f. Chem. Industrie. Continuous and uniform treatment of hydrocarbons with sulphurous acid. 6067. Apr. 9. (Ger., Mar. 21, 1917.)

Pearson and Son, and Smith. Treatment of coal-tar pitch, etc., for production of fuel. 6280. Apr. 13.

Thorp and Thorp. Refining benzol, etc. 6306. Apr. 13.

## COMPLETE SPECIFICATIONS ACCEPTED.

3948 (1917). Thompson (Reilly). Process and apparatus for distilling coal tar or the like. (114,453.) Apr. 17.

8789 (1917). Anderson. Carbonisation of pitch. (114,371.) Apr. 10.

## IV.—COLOURING MATTERS AND DYES.

## COMPLETE SPECIFICATION ACCEPTED.

4426 (1917). Inray (Soc. Chem. Industry in Basle). Manufacture of disazo dyestuffs for wool. (114,339.) Apr. 10.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Chambers and Hammond. Machines for washing flocks, rags, etc. 5946. Apr. 8.

Chambers and Hammond. Treatment of wool-scouring, etc., liquors containing grease or oil. 6005. Apr. 9.

Cretney, and Latham, Harris, and Co. Treating shoddy refuse from cotton waste. 5659. Apr. 3.

Croll. Manufacture of waterproof paper, etc. 6151. Apr. 11.

Johnston and Sutherland. Degumming or cleansing stalk fibres. 6217. Apr. 12.

Mond. Means for doping aeroplane, etc., fabrics. 5619. Apr. 2.

Zdanowich. Manufacture of cellulose acetates. 5952. Apr. 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

4248 (1917). Berglund. Apparatus for washing and airing cellulose and like fibrous materials. (114,456.) Apr. 17.

4991 (1917). Wallace, Marshall, Brown, and Bertrams, Ltd. Removing esparto, etc., from stationary digesters and the like. (114,348.) Apr. 10.

5406 (1917). Williams. Waterproofing composition for textile materials, etc. (114,494.) Apr. 17.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Larivei. Textile-yarn lustreing machines, 6016. Apr. 9.

Larivei. Machinery for dyeing, etc., textiles. 6017. Apr. 9.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Craig. 5653. See X.

Darrasse. Catalyser for production of oxy-chloride of carbon, etc. 6062. Apr. 9. (Fr., Apr. 23, 1917.)

Harger. Manufacture of hydrogen. 5594. Apr. 2.

Heap and Newbery. Manufacture of anhydrous chlorides. 5596. Apr. 2.

Maxted. Synthetic production of ammonia. 5741. Apr. 4.

Partington and Rideal. Oxidation of ammonia. 6112. Apr. 10.

Passmore. Utilisation of nitre cake. 6063. Apr. 8.

Taliani. Catalytic oxidation of ammonia. 5979. Apr. 8.

Titan Co. Aktieselskab. Titanium products. 6111. Apr. 10. (Norway, May 24, 1917.)

Wyld. Apparatus for distilling or treating ammoniacal liquors, etc. 6122. Apr. 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

5683. (1917). Dreyfus. See XX.

6009 (1917). Potter and Co., and Potter. Manufacture of sulphate of soda from chrome residues. (114,501.) Apr. 17.

6551 (1917). Comment. See XIII.

7213 (1917). Norske Aktieselskab for Elektrokemisk Industri. Manufacture of titanate acid. (108,850.) Apr. 17.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Calcott, Pickard, Sherlock, and Smith. Cement for repairing ceramic articles, glassware, etc. 5821. Apr. 5.

Levi. Refractory substances. 5592. Apr. 2.

Mississippi Glass Co. Light-refracting glass. 5715. Apr. 3. (U.S., Aug. 17, 1917.)

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

Bravin. Concrete. 5884. Apr. 5.

Hickson. Cement and process of manufacturing same. 5725. Apr. 3.

Kelsey. Waterproof preparation for roofing, etc. 6218. Apr. 12.

## COMPLETE SPECIFICATION ACCEPTED.

8298 (1917). Scarlett. Cement. (114,527.) Apr. 17.

## X.—METALS; METALLURGY. INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Annable and Janson. Treatment of ores and mixtures containing scheelite. 5723. Apr. 3.

Annable and Janson. Extraction of tin from its ores. 5858. Apr. 5.

Coles. Rendering aluminium and its alloys more durable. 5710. Apr. 3.

Cooper Co. Alloy. 6097. Apr. 10.

Craig. Recovering potash salts from blast furnaces. 5653. Apr. 3.

Cressey and Cressey. Recovery of metals from dross, skimmings, etc. 5901. Apr. 6.

Downes. Furnaces for melting metals. 5948. Apr. 8.

Ghijssen, and Naaml. Vennootsch. Vitrite Works. Smelting furnaces. 6057. Apr. 9.

Gontermann. Thomas process for producing steel. 5811. Apr. 4. (Ger., Apr. 4, 1917.)

Harvey. Annealing and reheating furnaces, etc. 6119. Apr. 10.

Hunt. Electrolytic treatment of ores. 6101. Apr. 10.

Marks (U.S. Nickel Co.). Separating nickel from nickel-copper mattes. 6193. Apr. 11.

Nelson and White. Manufacture of metallic lead powder and lead paints. 6065. Apr. 9.

Quinan. Production of metals. 6259. Apr. 12.

Ramén. Mechanical roasting furnaces. 6271. Apr. 12.

Verein. Hüttenwerke Burbach-Eich-Düdelingen A.-G. Production of metallic alloys. 6274. Apr. 12. (Ger., June 23, 1916.)

## COMPLETE SPECIFICATIONS ACCEPTED.

1388 (1917). Fennell, and British Carbonizing Co. Carburising or case-hardening iron or steel articles. (114,446.) Apr. 17.



2814 (1917). Shiga. Gold alloys. (114,447.) Apr. 17.  
 4112 (1917). Hamilton. Manufacture of grey-cast-iron. (114,328.) Apr. 10.  
 4746 (1917). Reynolds. Casting steel ingots. (114,466.) Apr. 17.  
 6821 (1917). Richmond Gas Stove and Meter Co., and Thompson. Steel furnaces. (114,359.) Apr. 10.  
 8225 (1917). Pozzo and Colonnetti. Testing of iron materials. (107,376.) Apr. 17.  
 11,307 (1917). Rollason. Melting and deoxidising mill scale, etc. (114,561.) Apr. 17.  
 13,405 (1917). Granberg. Condensers for redistilling spelter. (112,008.) Apr. 17.  
 17,231 (1917). Reid Bros., and Brown. Apparatus for testing the hardness of metals. (114,593.) Apr. 17.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

British Thomson-Houston Co. (General Electric Co.). Insulating materials. 6180. Apr. 11.  
 Fleming. Electric furnaces. 6039. Apr. 9.  
 Hunt. 6101. *See* X.  
 Marconi. Electric accumulators. 5867. Apr. 5. (Italy, Apr. 5, 1917.)  
 Sokal (Sokal). Primary batteries. 6059. Apr. 9.

##### COMPLETE SPECIFICATIONS ACCEPTED.

2838 (1917). Bonora. Primary electric batteries. (114,323.) Apr. 10.  
 4886 (1917). Wade (Flanders). Manufacture of storage battery plates and paste therefor. (114,475.) Apr. 17.  
 14,933 (1917). Dixon. Electric furnaces. (111,104.) Apr. 17.  
 15,722 (1917). Hoyle and Fawcett. Electrodes of electric furnaces. (114,395.) Apr. 10.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Blisey. Compound for washing, cleaning, etc. 6108. Apr. 10.  
 Carper and Tulloch. 5635. *See* XIV.  
 Chambers and Hammond. 6005. *See* V.  
 Feldenheimer and Plowman. Soap or soap derivatives. 6170. Apr. 11.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

##### APPLICATIONS.

Britton, and Griffiths Bros. and Co. Resins, and treatment thereof. 5724. Apr. 3.  
 Nelson and Wlute. 6065. *See* X.  
 New Jersey Zinc Co. Ziuc-lead pigments, and method of manufacturing same. 6270. Apr. 12. (U.S., Oct. 26, 1917.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

6551 (1917). Comment. Preparation of anhydrous sulphide of zinc. (106,489.) Apr. 10.  
 15,025 (1917). Morris. Manufacture of a red pigment. (114,581.) Apr. 17.  
 18,944 (1917). Akashi. Solid ink. (114,601.) Apr. 17.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### APPLICATIONS.

Carper and Tulloch. Preparation of solvents for indiarubber and animal fats. 5635. Apr. 2.  
 Dunlop Rubber Co., and Macbeth. Rubber-mixing, etc., machines. 5634. Apr. 2.  
 Gillott and Marple. Gum driers and solidifiers for rubber, etc. 6086. Apr. 10.  
 Jackson (Morgan and Wright). Calendering machines for treating rubber. 5796. Apr. 4.  
 Porzel. Treatment of rubber. 5758. Apr. 4.  
 Porzel. Vulcanising rubber, etc. 5759. Apr. 4.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### APPLICATIONS.

Ashley and Parsons. Artificial leather. 5600. Apr. 2.  
 Bennert. Utilisation of leather waste. 6050. Apr. 9. (Ger., Apr. 30, 1917.)  
 Gilardini. Instantaneous tanning of hides and skins. 5874. Apr. 5. (Italy, Apr. 6, 1917.)

##### COMPLETE SPECIFICATION ACCEPTED.

6391 (1917). Barclay. Manufacture of leather. (114,358.) Apr. 10.

#### XVI.—SOILS; FERTILISERS.

##### COMPLETE SPECIFICATION ACCEPTED.

5119 (1917). Svendsen. Construction of cyanamide and like silos. (114,487.) Apr. 17.

#### XVIII.—FERMENTATION INDUSTRIES.

##### APPLICATIONS.

Greville. Malt products. 5674. Apr. 3.  
 Greville. Brewing, and manufacture of brewing sugar, etc. 5676. Apr. 3.  
 Wardle. Extraction of liquid from yeast cells and its separation from the cellular matter. 6037. Apr. 9.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

Andrews. Sea-water evaporators. 5914. Apr. 6.  
 Bullot. Preservation of meat, etc. 5793. Apr. 4. (Austral., July 21, 1917.)  
 Greville. Treatment of flour. 5671, 5672, and 5673. Apr. 3.  
 Greville. Food products containing potatoes. 5675. Apr. 3.  
 Ohata. Process of canning. 5894. Apr. 6.

##### COMPLETE SPECIFICATIONS ACCEPTED.

105 (1917). Van Musschenbroek. *See* I.  
 5082 (1917). Lasseu and Hjort. Apparatus for purifying and softening water. (114,485.) Apr. 17.  
 19,008 (1917). Hyatt and Fellowes. Insecticides. (114,602.) Apr. 17.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Abbott Laboratories.  $\beta$ -Bromoethyl-*p*-nitrobenzoate. 6262. Apr. 12. (U.S., Dec. 21, 1917.)  
 Allgem. Ges. f. Chem. Ind. 6067. *See* III.  
 Paschall. Substance for treating tuberculosis, leprosy, etc. 5864. Apr. 5. (U.S., May 1, 1917.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

5683 (1917). Dreyfus. Manufacture of acetic acid from acetic aldehyde. (108,459.) Apr. 10.  
 8810 (1917). Soc. Chim. des Usines du Rhône. Production of acetic anhydride and polymerised or non-polymerised acetic aldehyde. (110,906.) Apr. 17.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

##### APPLICATIONS.

Burnett. Manufacture of photographic negative film. 5810. Apr. 4.  
 Dubois. Photographic printing. 5931. Apr. 6. (Fr., Dec. 17, 1917.)  
 Shiel. Kinematograph films. 6299. Apr. 13.

#### XXIII.—ANALYSIS.

##### COMPLETE SPECIFICATIONS ACCEPTED.

8225 (1917). Pozzo and Colonnetti. *See* X.  
 17,231 (1917). Reid Bros., and Brown. *See* X.

## I.—GENERAL ; PLANT ; MACHINERY.

*Refrigerating plants ; Relation between efficiency of — and the purity of their ammonia charge.* F. W. Frerichs. Amer. Inst. Chem. Eng., Dec., 1917. J. Ind. Eng. Chem., 1918, 10, 202—212.

AMMONIA refrigerating machines are of two types: absorption machines, in which the ammonia gas is absorbed in aqueous solution and regenerated under high pressure by heating at about 150° C. in a retort or boiler, so that ammonia is liquefied on cooling in the condenser; and compression machines, in which the gas is liquefied by pumping it into the condenser at a pressure of about 250 lb. per sq. in. In both types refrigeration is effected by reversing the liquefied ammonia through an expansion valve into a coil immersed in the cooling liquid. The question of the purity of the ammonia charge is of the highest importance. If substances are present either in suspension or solution which retard the evaporation of the ammonia or the transmission of heat through the walls of the tubes, the efficiency of the machine is greatly impaired. An apparatus is described for measuring the rate of evaporation at constant temperature. It is attached to the discharge nozzle of the transport cylinder and is adapted to draw off 100 c.c. of liquid ammonia and evaporate it by heat derived from a bath of ice-water surrounding a calcium chloride bath in which the apparatus is immersed. The less volatile impurities are collected at the bottom and by making several tests in succession, the cumulative influence of these impurities on the rate of evaporation may be observed. One of the most obnoxious impurities, which may not be in the original ammonia but is always liable to be present in the working machine, is lubricating oil, which coats the coils of the cooling tank and very seriously interferes with the efficiency of the heat transmission. Non-condensable gases constitute another serious source of loss of efficiency, particularly on the compression side of the plant; the pressure required to produce liquefaction increases with the degree of contamination. These gases accumulate in the condenser and can be blown out periodically through a vent cock. Non-condensable gases may also be developed from the lubricating oil at the temperature of the compressor, and an apparatus is described for testing oils in this respect. Impurities, such as acetic acid and acetonitrile, in the ammonia may lead to rapid corrosion of the plant; a small quantity of acetic acid may corrode a large amount of iron since the acid is always regenerated. Details are given of continuous tests made with two refrigerating plants, one using pure ammonia and the other an inferior quality. The former produced 16,862 tons of ice at an ammonia cost of 1.27 cents per ton, while the latter produced only 11,308 tons in the same period at an ammonia cost of 15.16 cents per ton. In addition, the second plant was considerably corroded. The production of permanent gases during working was a special characteristic of the inferior ammonia, necessitating frequent purging through the vent cock; these gases could be ignited and gave a very large flame over a period of 25—30 minutes. Analysis of the volatile carbon compounds present in the two grades of ammonia showed 0.002% (as CO<sub>2</sub>) in the pure ammonia and 0.002% in the inferior. The volatile carbon compounds can be concentrated and the ammonia purified by fractional distillation of the liquefied gas; the degree of purity obtained corresponds to the former figure quoted above. In machines of the absorption type, careful attention must be paid to the purity of the distilled water used for absorbing the ammonia; it must be distilled from water freed from suspended organic matters. The

author proposes a method for the estimation of minute quantities of volatile impurities in liquid ammonia, based on the increase of vapour tension which is caused by the presence of volatile impurities. The apparatus consists of two similar barometer tubes enclosed in the same water jacket, which can be heated uniformly. An impure sample of ammonia neutralised with pure sulphuric acid is placed over the mercury in one barometer and a solution of pure ammonium sulphate in the other. The difference in level of the two barometers at various temperatures indicates the relative vapour tensions of the two samples and may be standardised in terms of benzene.—J. F. B.

*Thermochemical processes ; Laws of — and of photochemical processes.* M. Trautz. Z. anorg. Chem., 1918, 102, 81—129.

THE author summarises his views on the laws of gas reactions, with particular reference to the relation between the velocity constant, heat of reaction, and "heat of activation." The equations and laws deduced for thermochemical reactions are then applied to photochemical reactions. (See also J. Chem. Soc., May, 1918.)—E. H. R.

## PATENTS.

*Organic chemical reactions ; Process of effecting—.* A. M. Aylsworth, and Savings Investment and Trust Co., East Orange, N.J., Eng. Pat. 110,547, Jan. 17, 1917. (Appl. No. 14,152 of 1917.) Under Int. Conv., Jan. 24, 1916.

A BATH of fused sodium nitrite is used for supplying heat to a chamber in which organic chemical reactions are conducted. The salt fuses at about 213° C. and may be heated to 390° C. or even higher without decomposing. The fused salt is a thin fluid so that uniformity of temperature is insured. It is claimed to be superior to a bath of fusible metal. The reaction chamber may be in the form of a coil of tubing through which the reaction mixture is forced.—J. H. P.

*Storing, treating, or conveying corrosive liquids or substances ; Apparatus for —.* J. Wolf, London. Eng. Pat. 113,992. (Appl. Nos. 2017, Feb. 9, and 2295, Feb. 15, 1917.)

METALLIC vessels are provided with perforated supports spaced apart from the walls around which the resisting lining material is cast or moulded.—W. H. C.

*Compression of granular and other substances [e.g., peat].* T. W. S. Hutchins, Middlewich, Cheshire. Eng. Pat. 114,002, Mar. 8, 1917. (Appl. No. 3344 of 1917.)

IN the compression of granular and other substances by extrusion, a horizontal cylinder is provided having an inlet and a feeding hopper at one end. The inlet is controlled by a reciprocating plunger which forces the material towards the discharge nozzle at the other end, and a small secondary reciprocating plunger is provided moving concentrically within the main plunger, which imparts the final compression to the charge and produces a more uniform compression. When the press is used for expressing liquids, e.g., for removing water from peat, a perforated cylinder is used.—W. F. F.

*Furnaces ; Gaseous fuel —.* A. C. Ionides, jun., London. Eng. Pat. 114,223, Apr. 16, 1917. (Appl. No. 5352 of 1917.)

IN a furnace of the type described in Eng. Pat. 108,701 (this J., 1917, 1086), a series of upright combustion chambers or passages are arranged in or upon the encircling wall of an upright tubular heating chamber. These passages communicate



with the heating chamber by one or more narrow openings and are supplied with a combustible gaseous mixture by jets situated at or near their lower ends. The passages may be spaced equidistantly around the heating chamber and formed in the thickness of the walls.—J. H. P.

[*Muffle*] *furnace*. F. W. Steere, Detroit, Mich. U.S. Pat. 1,257,649, Feb. 26, 1918. Date of appl., July 31, 1915.

A MUFFLE furnace is provided with valve-controlled sets of regenerating chambers through which the fuel and exhaust gases are passed alternately, to and from the combustion chamber. The muffle is arranged adjacent to the combustion chamber and is adapted to receive "stock" at one end and discharge it at the other end into a receptacle arranged in a tower into which the exhaust gases from the regenerator also pass. The oxidation of the "stock" is thereby prevented.—W. F. F.

*Separators; Pneumatic* —. G. W. S. Simpson, London. Eng. Pat. 114,288, Dec. 17, 1917. (Appl. No. 18,737 of 1917.)

The separator comprises a screening surface consisting of a number of louvres arranged to form an inclined plane. The size of the openings between the louvres can be varied. The material to be separated descends this inclined plane against a current of air which carries the smaller and lighter particles between the louvres, whilst the larger particles fall into a shoot and are discharged. The current of air laden with particles is conveyed to a separator of the cyclone type, in which the inlet pipe terminates within a hood in a number of curved outlets adapted to impart a spiral motion to the air within the hood. The hood is contained in a closed hopper from which the dust is discharged, whilst the air is returned to the intake of the blower used for circulating the air. The apparatus is specially applicable to the grading of sand, earths, nitrate caliche, etc.—J. H. P.

*Condenser*. H. A. Hills, Grand Rapids, Mich. U.S. Pat. 1,256,229, Feb. 12, 1918. Date of appl., July 28, 1916.

AN outer cylindrical vessel containing cooling liquid is open at the top and is provided with condensing tubes fixed in the bottom plate and extending upwards. An inverted conical member is attached below the tube-plate to collect and discharge condensed liquid. The upper ends of the tubes are fixed in a tube-plate of smaller diameter than the cylinder and this plate is surmounted by a conical hood. Vapour to be condensed is supplied to the top of the hood and passes through and over the edges of a perforated rectangular horizontal plate mounted in the hood above the upper ends of the tubes. Cooling liquid is supplied to the bottom of the outer vessel and is discharged at the top.—W. F. F.

*Separator; Centrifugal* —. F. L. Jefferies, Riverside, Ill. Assignor to Corn Products Refining Co. U.S. Pat. 1,256,697, Feb. 19, 1918. Date of appl., July 21, 1917.

A ROTARY container is provided with a discharge opening in the cover for the lighter constituent of the material treated and also with a discharge duct for the heavier constituent. The inlet branch of this duct extends downwards inside the container near the wall, and the duct passes through the container wall and has a discharge branch also extending downwards. Both discharge openings have a larger cross-sectional area than is required for the quantity of material which normally passes through them.—W. H. C.

*Separator; Centrifugal* —. M. B. Miller, Long Island City, N.Y., Assignor to Sharples Specialty Co., West Chester, Pa. U.S. Pat. 1,257,515, Feb. 26, 1918. Date of appl., Oct. 10, 1917.

THE separator consists of a rotary vertical bowl having an axial bottom inlet and an unobstructed lower inner portion. A spreader is provided above the inlet and means for "catching and accelerating the revolution" of the contents of the bowl are arranged in its upper part.—W. H. C.

[*Centrifugal machines;*] *Froth-reducing discharge apparatus and process* [for —]. M. Leitch and B. R. Wright, Poughkeepsie, N.Y., Assignors to the De Laval Separator Co., New York. U.S. Pat. 1,256,810, Feb. 19, 1918. Date of appl., Oct. 29, 1915.

To reduce froth formation in the discharge of liquid from high-speed centrifugals with an overflow discharge, the liquid is propelled in an outward and rearward direction from the overflow, and the usual thin, flat stream is caused to form a thicker stream without reducing the cross-sectional area, whereby the velocity of the stream and its exposed surface are both reduced. The centrifugal is provided with a number of relatively long discharge weirs, over which the liquid flows in thin streams. A curved discharge passage extending outwards and rearwards, converging in one direction, extends from each weir and is proportioned and arranged to discharge the liquid in a solid stream thicker than at the weir, without increase in the nozzle velocity.—J. H. P.

*Liquid-purifier; Centrifugal*. —W. G. Howell, Warrenton, Va. U.S. Pat. 1,257,235, Feb. 19, 1918. Date of appl., Dec. 13, 1917.

A HORIZONTAL cylinder mounted on a hollow shaft is divided by a partition into an inner axial chamber and an outer narrow annular chamber. The cylinder is rotated rapidly on the hollow shaft through which the liquid is introduced into the axial chamber. From the axial chamber the liquid passes into one end of the outer annular chamber and the separated solids are collected in pockets formed on the periphery. The clarified liquid is discharged from the opposite end.—W. H. C.

*Refrigerating apparatus*. W. A. Richter and E. S. H. Baars, Milwaukee, Wis. U.S. Pat. 1,256,730, Feb. 19, 1918. Date of appl., Dec. 29, 1916.

A SUPPORTING frame carries a gas chamber above and an expansion chamber below. A number of compressing cylinders are disposed radially within the gas chamber, the pistons being operated from cams on a central vertical shaft. The gas chamber is surrounded by a condenser which has a conduit communicating with the expansion chamber. Below the expansion chamber, and communicating with it, are vertical cooling coils.—W. H. C.

*Evaporating tank*. W. E. Fletcher, Tamaqua, Pa., Assignor to Atlas Powder Co., Wilmington, Del. U.S. Pat. 1,257,209, Feb. 19, 1918.

REMOVABLE spur tubes, slightly spaced from each other, are arranged just above the bottom of the tank and extend through flanged ports in the opposite ends of the tank. The ends of the spur tubes are connected by flanged elbows with flanged outlets of a steam manifold, one at each end of the tank. Each elbow is provided with a "bleed pipe," which projects into the adjacent spur tube.—J. H. P.



*Evaporating apparatus.* C. R. Harris, Assignor to A. P. Campbell, Los Angeles, Cal. U.S. Pat. 1,257,854, Feb. 26, 1918. Date of appl., Aug. 12, 1916.

The apparatus consists of a vessel provided with a central inlet tube which passes through the bottom and projects upwards within the vessel. The inlet tube supports a number of superposed concavo-convex diaphragms, whilst in the upper portion of the vessel are retarding discs mounted on a rod projecting downwards from the top of the vessel. A cap carried by this rod is arranged above the discharge end of the inlet tube, whereby the liquid is directed upon the diaphragms. An outlet for the vapour is provided at the top of the vessel. The outlet tube for the liquid is at the bottom of the vessel and surrounds the inlet tube. —J. H. P.

*Reaction tower.* C. S. Robinson, Cambridge, Mass., Assignor to E. B. Badger and Sons Co., Boston, Mass. U.S. Pat. 1,257,400; Feb. 26, 1918. Date of appl., June 16, 1917.

A TOWER for effecting reaction between a liquid and a gas in the course of which heat is evolved and the specific gravity of the liquid is increased, comprises a series of reaction chambers, one above the other, and means for admitting liquid to the uppermost chamber and transferring it to the one below and for admitting gas to the bottom chamber and leading it up through the others. Means are provided for local cooling and circulation of the liquid. —J. H. P.

*Pulverising-machine.* C. A. Koehler, Easton, Pa., and C. C. Crosson, Alpha, N.J. U.S. Pat. 1,257,882, Feb. 26, 1918. Date of appl., Aug. 14, 1916.

A STATIONARY horizontal drum has a hollow shaft by means of which the grinding beaters are rotated. The drum is divided into two chambers by a vertical partition between the last set of beaters and the end wall, the portion of the partition above the shaft being perforated and forming a sieve. The portion of the hollow shaft between the screen and the end wall is provided with inlets opening into the drum. —W. H. C.

*Distilling methods and apparatus.* E. A. R. Chenard, Cognac, France. Eng. Pat. 5014, Mar. 31, 1915. Addition to Eng. Pat. 10,783 of 1913.

SEE Addition of Mar. 31, 1914, to Fr. Pat. 443,499 of 1912; this J., 1915, 480.

*Evaporators.* W. L. De Baufre, Annapolis, Md., U.S.A. Eng. Pat. 114,164, Oct. 24, 1916. (Appl. No. 15,136 of 1916.)

SEE U.S. Pat. 1,213,596 of 1917; this J., 1917, 376.

*Compressing elastic fluid; Method of —. Method of compressing air or gases.* H. A. Humphrey, Westminster, Assignor to Humphrey Gas Pump Co., New York. U.S. Pats. 1,257,004, Feb. 19, and 1,257,007, Feb. 26, 1918. Dates of appl., July 17, 1908, and Feb. 19, 1910. Renewed July 14 and 18, 1917.

SEE Eng. Pat. 22,646 of 1907; this J., 1908, 1101.

*Drying apparatus.* U.S. Pat. 1,255,915. See V.

*Gas-fired furnaces, oil-fired furnaces, and the like.* Eng. Pat. 114,177. See X.

*Drying apparatus.* U.S. Pat. 1,256,825. See XIXA.

*Means for maintaining suspended matter in liquids in a state of suspension without precipitation.* Eng. Pat. 114,016. See XIXE.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coals; Moisture content of some typical —.* G. A. Hulett, E. Mack, and C. P. Smyth. Amer. J. Sci., 1918, 45, 174–184.

FOUR samples of American bituminous coals contained the following amounts of moisture:—New River, 1.50; Pittsburgh, 2.45; Indiana, 6.82; Wyoming, 8.20%. The samples were passed through a 60-mesh sieve and precautions were taken to avoid loss or gain of moisture after this operation. The method employed consisted in condensing the moisture evolved on heating the coal, on a tube containing solid carbon dioxide and inserted in the top of the tube containing the coal, the air being exhausted from the apparatus. The results obtained were about 30% higher than those yielded by the standard method. —W. P. S.

*Coke; Determination of moisture in —.* A. C. Fieldner and W. A. Selvig. Tech. Paper 148, U.S. Bureau of Mines, 1917. [14 pages.]

IN the determination of moisture in coke, the temperature, time of drying, humidity of drying atmosphere, and fineness of sample may be varied considerably without appreciably affecting the result. Temperatures ranging from 105° to 200° C. produced a variation not exceeding 0.3%. The circulation of air dried by sulphuric acid through the drying oven, as specified for coal analysis, is unnecessary; also, no gain of weight takes place during prolonged drying owing to absorption of oxygen, as is usually the case with coal. A rapid and sufficiently accurate method ( $\pm 0.5\%$ ), and one which may be used advantageously at the place of sampling, is to heat to constant weight a large sample of lump coke in any convenient oven, or on a stove, hot plate, or steam coil between 100° and 200° C. —L. A. C.

*Gasoline; Testing natural gas for —.* G. G. Oberfell. J. Ind. Eng. Chem., 1918, 10, 212–214.

FOR testing casing-head gas at the source of production a simple portable apparatus has been devised for working under atmospheric pressure. The absorption vessel has a capacity of about 1 cub. ft., and consists of a galvanised iron cylinder, 10 ins. in diameter and 2 ft. long, with conical ends with  $\frac{1}{4}$  in. nipples and valves at each end. The vessel is calibrated with water. The absorber is attached to the gas supply in a vertical position by rubber tubing and gas is passed through it for 30 mins. Connection is made from the top when the gas is lighter than air and from the bottom when it is heavier. In either case the connection is made at the top for the last 15 mins., so as to expel any small amount of oil left from the previous test. All the air having been displaced, the valves are closed with the gas at atmospheric pressure; temperature and barometer reading are noted. The bottom nipple is then connected with a cylindrical glass oil-pipette, delivering about 850 c.c. of oil between two marks, the connecting tube having previously been filled with oil. The oil employed is "mineral seal oil" which has been ascertained to give no distillate at 350° F. (176.5° C.). The admission of oil to the absorber is started by applying pressure, after which the diminution in volume of the gas will draw in the remainder. If there is much diminution, air must be admitted to avoid collapse of the vessel. The oil is shaken in the absorber for 20 mins., then withdrawn and tested for gasoline content by distillation. The apparatus consists of a copper flask of 1000 c.c. capacity, having a tall metal vapour pipe with the bulb of a thermometer extending  $2\frac{1}{2}$  ins. below the branch outlet. The condenser is a glass tube with three bulbs of about



80 c.c. total capacity, surrounded by an ice-water bath in an inverted 1-gallon can from which the bottom has been removed. The receiver is a cylinder graduated to read in  $\frac{1}{10}$  c.c. 800 c.c. of oil is taken for distillation, which is carried on until the thermometer registers 350° F. (176.5° C.) The quantity of distillate may be less than 1 c.c. and its specific gravity is taken either in a graduated cylinder for samples between 4 and 10 c.c. or in a pycnometer made from small-bore tubing for samples less than 4 c.c. Results are calculated as yield of gasoline in gallons per 1000 cub. ft. of gas at standard temperature and pressure.

—J. F. B.

*Fats and fatty acids from petroleum.* R. J. Moore and G. Egloff. Met. and Chem. Eng., 1918, 18, 308—311.

FOUR types of reaction have been proposed for the synthesis of fats and fatty acids from paraffin, olefine, and naphthene hydrocarbons:—(1) Conversion of saturated paraffins into halogen substitution products, the products being converted into alcohols which can be further oxidised to fatty acids. (2) The Grignard reaction has been applied to chlorinated paraffins containing up to ten carbon atoms and the corresponding fatty acids prepared. (3) The naphthenic oils from the Russian and Galician wells are known to contain oxygen compounds; octanaphthenic and adipic acids have been identified. On neutralisation such acids yield soaps. (4) Oxidation of paraffins and olefines forms the most direct process. Vaseline has been converted into a soap by oxidation with peroxides. Molten paraffin wax has been oxidised to fatty acids by prolonged treatment with a blast of air or oxygen. Olefines which are easily obtainable by pyrogenic treatment of paraffins are still more readily oxidised. The authors consider the commercial synthesis of fats as reasonably probable in the near future. The economic importance of this in view of the present depletion of the world's herds of livestock is indicated.—H. J. H.

*Analysis of spent oxide [from gas works].* See VII.

*Iron trisulphide.* Mecklenburg and Rodt. See VII.

*Tank-furnace for white glass fired by gas from a coke-producer.* Roxburgh. See VIII.

*Thermal leakage and calorimeter design.* White. See XXIII.

#### PATENTS.

*Peat; Manufacture of fuel from*—W. Goodwin and A. Macrae, Rhyl, Wales. Eng. Pat. 114,026, Mar. 15, 1917. (Appl. No. 3752 of 1917.)

PEAT is passed through a crushing mill, similar to that used for crushing sugar cane, to break up the fibre and expel a large proportion of the water, and the resulting thin cakes are passed between rollers maintained at a high temperature by superheated steam or electrical resistances. The peat is then allowed partly to dry in a storage chamber and is subsequently distilled at a low temperature in a series of retorts heated by electrical resistances embedded in the peat, the removal of the remaining moisture being facilitated by the maintenance of a vacuum in the retorts. The products are drawn through the usual condensers and washers to suitable receivers, and any permanent gas passes through the exhaustor to a gas holder. The residue is discharged through the bottom of each retort into a gas producer, where it is converted into carbon monoxide, which may be used for generating steam and also for driving the gas engines which operate the crushing and heating rolls and the dynamos for producing the electric current for the heating resistances. The perma-

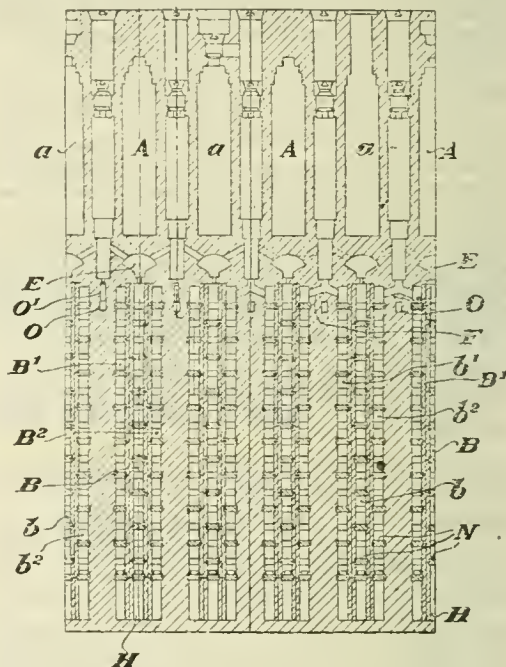
nent gas may also be used in this manner. About 8—10% of peat tar is obtained which is converted into motor spirit and heavy oils by fractional distillation, and ammonium sulphate is obtained from the washers.—W. F. F.

*Artificial fuel; Process for the manufacture of— from waste products.* R. Brown, Southall. U.S. Pat. 1,256,494, Feb 12, 1918. Date of appl., Sept. 17, 1917.

ASH-BIN refuse normally containing coal, etc., is finely divided and pressed into blocks or briquettes without a binder other than the contained moisture. The blocks are then treated with a liquid binding material consisting of equal parts of oil tar and pitch in the proportion of 10% of the weight of the combustible portion of the waste material.—W. F. F.

*Coke ovens and muffle and like furnaces; Gas fired* — Simon-Carves, Ltd., and J. H. Brown, Manchester. Eng. Pat. 113,696, Mar. 24, 1917. (Appl. No. 4255 of 1917.)

In a battery of coke oven or muffle or like furnaces, the combustible gas or mixture of gases, with or without part of the necessary air, is fed to a series of horizontal passages, H, below every alternate



oven, A. The gas passes upwards through the vertical chambers, B, provided with baffles, N, to the horizontal passages, E, and thence to the combustion chambers on each side of the ovens, A. The remainder of the air passes through similar vertical chambers, b, below alternate ovens, a, to the same combustion chambers. These chambers are each divided into two parts by a transverse vertical partition extending nearly to the top, and the burnt gases pass down the other side of the partition and through passages, F, to the two vertical chambers, B¹, B², or b¹, b², provided with baffles, and enclosing the gas chamber, B, or air chamber, b, to preheat the gas or air supply. Rich coke-oven or other gas may also be admitted to the combustion chamber through the horizontal passages, O, and conduits, O¹.—W. F. F.



*Gas plant; Method for preventing the choking up of*—A. Riedel, Kössern, Saxony. Eng. Pat. 101,219, Aug. 16, 1916. (Appl. No. 11,574 of 1916.) Under Int. Conv., Aug. 16, 1915.

IN apparatus for the dry distillation of coal, magnesium chloride or calcium chloride is added to the charge in sufficient quantity to convert the whole of the nitrogen into ammonium chloride. The pipe connections between the retort and condenser are insulated and maintained above 450° C., and the condenser is maintained just below 450° C., to condense the ammonium chloride in a comparatively pure state.—W. F. F.

*[Gas] retorts and methods of working the same.* J. West, Southport, and W. Wild, Blackpool. Eng. Pat. 113,981, Dec. 9, 1916. (Appl. No. 17,714 of 1916.)

RETORTS for the destructive distillation of coal or other carbonaceous material are arranged horizontally or slightly inclined, and are flattened in cross-section. Each retort increases in size slightly from the inlet to the outlet, and the retorts are arranged one above the other in pairs, the pairs being spaced apart horizontally in a casing. Coal is fed into one end from a hopper, and pressed forward by a plunger reciprocating continuously so that the retort is kept fully or nearly fully charged. Each pair of retorts is surrounded at the inlet end by a combustion chamber, so that the coal is immediately subjected to a very high temperature and the outer layer of the charge is immediately coked. Progression of the charge along the retort is thereby facilitated. The hot combustion gases then circulate through a chamber surrounding the middle zone of the retort, and secondary air passes to the combustion chamber through a heating chamber surrounding the discharge end of each retort. Three distinct temperature zones are thus provided for each retort. Separate discharge passages for the coke and conduits for the gas are provided at the larger end of each retort.

—W. F. F.

*Gas generators.* T. R. Wollaston, Manchester. Eng. Pat. 113,856, Mar. 19, 1917. (Appl. No. 3962 of 1917.)

IN an apparatus for generating gas from coal or other carbonaceous material, in which the gas is afterwards treated for the extraction of by-products, a producer of ordinary construction is provided with narrow vertical retorts mounted on the top, through which the producer gas passes to a common central outlet. The coal is fed downwards through the retorts, each of which is provided with means for regulating the passage of the coal and the upward flow of gas and coal distillates. The controlling device may be a perforated helical strip mounted on a vertical spindle which is slowly rotated. In another form the controlling device may consist of a series of perforated cones mounted on a vertical spindle, which pass within inverted perforated cones fixed to the retort wall. The vertical spindle is reciprocated vertically to allow the passage of coal between the pairs of conical members.—W. F. F.

*Gas washer-scrubbers.* F. Thuman, London. From J. A. P. Crisfield, Philadelphia, Pa., U.S.A. Eng. Pat. 113,937, Nov. 14, 1917. (Appl. No. 16,744 of 1917.)

A CLOSED vertical cylindrical casing is provided with an inner concentric cylindrical partition depending from the cover and dipping into the liquid which approximately half fills the casing. The gas to be washed is introduced by a central pipe dipping into the liquid and passes into the

outer vessel through an opening in the side. A spray of liquid is also directed outwards through this opening, and the gas and spray impinge on a series of angle bars projecting from the wall of the outer casing. The washed gas then passes round the annular space surrounding the cylindrical partition to an outlet at the opposite side. Projecting baffles are provided on both walls of this annular space to intercept the gas current. The liquid is maintained at the desired level in the casing by an overflow pipe arranged in a small chamber attached to the side of the main casing.

—W. F. F.

*Fullers' earth [from mineral oil refining] and similar materials; Process for treating*—C. M. Husted, Jersey City, N.J., Assignor to Standard Oil Co. U.S. Pat. 1,256,233, Feb. 12, 1918. Date of appl., June 25, 1915.

FULLERS' earth, which has been used for treating hydrocarbons, is supplied continuously together with acid to a chamber, where it is agitated by tumbling. The mixture is continuously removed from the chamber, and impurities and reaction products are separated by washing and settling. The earth is again washed by agitation, and then dried. Any earth contained in the impurities and reaction products is separated, washed, and dried.

—W. F. F.

*Wax-bearing oil; Apparatus for cooling*—for pressing. E. P. Folger, Brooklyn, N.Y., Assignor to Standard Oil Co., Bayonne, N.J. U.S. Pat. 1,256,460, Feb. 12, 1918. Date of appl., Jan. 13, 1914.

A VERTICAL cooling chamber of annular horizontal cross-section is provided both inside and outside with chambers for a cooling liquid, and suitable inlet and outlet openings are provided for the passage of the oil and cooling liquid in opposite directions. A head is attached to the top of the central cooling cylinder, the outlet of which forms a conduit extending vertically upwards above the head and is supported on it by a ball bearing. The outer surface of the conduit forms a bearing for a rotating sleeve, having a portion projecting horizontally over the annular cooling chamber and carrying a scraping device. The scraper comprises an annular frame composed of a number of rings, one of which rests on a ball bearing on the bottom of the chamber, the intermediate rings being connected by vertical strips. The scraper bars are mounted in the frame and acted upon by springs in such a way that they are pressed simultaneously against the outer and inner walls of the cooling chamber and thus remove deposited material.—W. F. F.

*Petroleum; Method and apparatus for treating*—R. C. Dundas, Los Angeles, Cal. U.S. Pat. 1,257,199, Feb. 19, 1918. Date of appl., Aug. 11, 1917.

EACH of a series of chambers contains a vaporising flue suspended free of the wall and connected at the top with the next chamber, the last chamber of the series being connected with a condenser. Heated gas is passed through a pipe connected with the condenser into the first chamber of the series and passes through the chambers in succession, first downwards round the outside of the vaporising flue and then upwards within the flue; the oil to be treated is sprayed into the bottom of each vaporising flue. Within each chamber is a means for collecting condensed products.—L. A. C.

*Aromatic bodies from petroleum oils; Process for the production of*—F. W. Mann, Berkeley, and M. L. Chappell, El Segundo, Cal., Assignors to Standard Oil Co., Richmond, Cal. U.S. Pat. 1,257,906, Feb. 26, 1918. Date of appl., Nov. 12, 1917.

PETROLEUM oils containing oils of the carbocyclic



series are dehydrogenated by treatment with a volume of air ranging from 500 to 800 cub. ft. for each 15 to 20 galls. of oil, in the presence of nickelous oxide, at a pressure of from 1 to 4 ins. of mercury below atmos. pressure and between 600° and 750° C., the air being mixed with the products of combustion required to maintain this temperature.—L. A. C.

*Sulphur; Eliminating*—from oils. E. V. Evans, London. U.S. Pat. 1,257,829, Feb. 26, 1918. Date of appl., Oct. 25, 1915.

SEE Eng. Pat. 22,147 of 1914; this J., 1915, 1240.

*Compression of granular and other substances* [e.g., peat]. Eng. Pat. 114,002. See I.

*Gaseous fuel furnaces.* Eng. Pat. 114,223. See I.

*Gas-burning apparatus.* U.S. Pat. 1,256,301. See IIB.

*Process of making drying oils from mineral oils.* U.S. Pat. 1,256,535. See XIII.

*Thinned paint and process of making paint thinner* [from mineral oils]. U.S. Pat. 1,256,537. See XIII.

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Gas-burning apparatus* [for heating and lighting]. C. Ellis, Montclair, N.J., Assignor to Surface Combustion, Inc., Wilmington, Del. U.S. Pat. 1,256,301, Feb. 12, 1918. Date of appl., May 8, 1912. Renewed June 28, 1917.

IN a heating and lighting apparatus of the surface combustion type, a thick porous diaphragm mounted in a casing is formed of porous granules of substantially uniform size, the surface granules being coated with rare earth actinic material to promote luminosity. A gas chamber extends over one side of the diaphragm and an air chamber extends similarly over one end. A number of cooling passages are provided in the diaphragm, through which air passes to the air chamber, and the air is conveyed into the gas chamber and mixed with gas by means of a gas jet passing through an injector tube which connects the two chambers. The combustible mixture then passes through the porous diaphragm where it is burnt. A regulating damper is provided to control the amount of air drawn through the cooling passages by the injecting nozzle.—W. F. F.

*Incandescence lamps; Electric*—L. Ham-burger, D. Lely, jun., and Naaml. Vennoots. Philips' Gloeilampenfabrieken, Philipsdorp, Netherlands. Eng. Pat. 113,626, Nov. 29, 1916. (Appl. No. 17,161 of 1916.)

THE inner surface of the bulb is provided with a thin layer of calcium fluoride, which during the burning of the lamp does not evolve a gas giving an active or measurable vapour pressure, but prevents the obscuring of the bulb.—B. N.

*Carbonaceous material; Separation of volatile products from solid*—W. W. White, London. U.S. Pat. 1,257,772, Feb. 26, 1918. Date of appl., Sept. 1, 1916.

SEE Eng. Pat. 14,800 of 1915; this J., 1917, 286.

*Metallic wires, filaments, and the like; Process of producing*—Filament or wire formed of a single crystal. O. Schaller, Südde, Germany. U.S. Pats. 1,256,929 and 1,256,930, Feb. 19, 1918. Date of appl., May 16, 1914.

SEE Fr. Pat. 469,212 of 1914; this J., 1915, 36.

## III.—TAR AND TAR PRODUCTS.

*Ammonia recovery at tar-distilling plants.* C. R. Woodward. Met and Chem. Eng., 1918, 18, 299—301.

IT is not customary at tar distillation plants in America to recover the ammonia from the liquor settling out of the tar or from the aqueous portion of the distillate. Analyses of the liquors settling from gas works and coke oven tars showed up to 1.5% NH<sub>3</sub> and in the distilled liquor up to 2.52% and 3.12% if caustic soda was present during distillation. The ammonia in the distilled liquor was free and thus required no lime for recovery. These liquors form a fruitful and at the present time profitable source of ammonia.—H. J. H.

*Monomethylaniline and dimethylaniline; New method of preparation of*—by catalysis. A. Mailhe and F. de Godon. Comptes rend., 1918, 166, 467—469.

IF a mixture of the vapours of aniline and a slight excess of methyl alcohol is passed over aluminium oxide at 400°—430° C., a mixture of methyl- and dimethylaniline, containing only traces of aniline, is obtained. Methylaniline may be similarly converted into dimethylaniline. During the process the alumina in time becomes yellow and then brown, and loses some of its activity. This may be restored by a simple calcination. For this process it is not necessary that the aniline should be free from water or the methyl alcohol from acetone. (See also J. Chem. Soc., May, 1918.) —W. G.

*Methyltoluidines; Preparation of the*—by catalysis. A. Mailhe and F. de Godon. Comptes rend., 1918, 166, 564—566.

THE method used for the methylation of aniline (see preceding abstract) is equally applicable to the three toluidines. The first passage of the mixed vapours of the toluidine and methyl alcohol over alumina at 350°—400° C. gives a mixture of methyl- and dimethyltoluidine, which is entirely converted into dimethyltoluidine by a second passage over the alumina with methyl alcohol. (See also J. Chem. Soc., May, 1918.)—W. G.

*Isomers; Limits of accuracy of Holleman's method of determining the composition of a mixture of two or three*—[Application to the nitranilines.] J. B. Nichols. J. Amer. Chem. Soc. 1918, 40, 400—403.

THE method (Holleman, "Die direkte Einföhr. von Substituenten in den Benzolkern," 1910) was applied to mixtures of the three nitranilines containing a relatively small proportion of the ortho-compound. The temperature at which the mixture begins to crystallise and the eutectic temperature were measured. The difference between the observed eutectic temperature and the eutectic temperature for mixture of *m*- and *p*-nitraniline is proportional to the amount of *o*-nitraniline present. Since the temperature at which *p*-nitraniline crystallises out is not appreciably altered when a small proportion of the admixed *m*-compound is replaced by the same amount of the *o*-compound, it is apparent that the two temperature readings will permit of an estimation of the proportions of the three isomers in a mixture. In the case of

the nitranilines, the method appears to give satisfactory results if the percentage of the third isomer (*o*-nitraniline) does not exceed about 10%. (See also J. Chem. Soc., May, 1918.)—H. M. D.

#### PATENTS.

*Cresols; Process for the separation of*—G. A. Darzens, Paris. Eng. Pat. 107,961, Dec. 4, 1916. (Appl. No. 17,398 of 1916.) Under Int. Conv., July 11, 1916.

THE capacity of *p*-cresol and *m*-cresol to form addition products with oxalic acid and with anhydrous sodium acetate respectively is utilised to effect a separation of the three isomers. It is necessary during the formation of these products to exclude moisture and to use solvents having a chemical nature different from that of the cresols, *e.g.* ligroin, benzene, toluene, the xylenes, chloroform, chlorobenzene, or like chlorohydrocarbons. The addition products separate as solids, and are filtered off; they are readily decomposed by water. It is advantageous to separate the *o*-cresol first by fractional distillation *in vacuo*, the compound being obtained sufficiently pure to crystallise on cooling, and by draining the crystals, very pure technical *o*-cresol may be produced. *Example:* 100 parts of the residue obtained after distilling off the *o*-cresol is mixed with 100–150 parts of ligroin and 15–17 parts of anhydrous oxalic acid in a mixer provided with a cover and an agitator. After sufficient time, the solid compound is separated, washed with ligroin, and treated with a suitable quantity of water in a lead-lined still provided with a steam heating and distilling apparatus. The *p*-cresol distils off, leaving a solution of oxalic acid, which is readily recovered. The liquid left behind in the separation of the solid compound may be treated in a similar manner with sodium acetate to recover the *m*-cresol.—L. A. C.

*Phenols: Process of producing*—Bostaph Engineering Co., Assignees of A. S. Ramage, Detroit, Mich., U.S.A. Eng. Pat. 106,274, Mar. 27, 1917. (Appl. No. 4136 of 1917.) Under Int. Conv., Apr. 20, 1916.

SEE U.S. Pat. 1,208,833 of 1916; this J., 1917, 203.

*Process of effecting organic chemical reactions.* Eng. Pat. 110,547. See I.

*Process for the production of aromatic bodies from petroleum oils.* U.S. Pat. 1,257,906. See IIa.

#### IV.—COLOURING MATTERS AND DYES.

*Camwood, barwood, and sanderswood; Colouring matters of*—[and of St. John's wort]. P. O'Neill and A. G. Perkin. Chem. Soc. Trans., 1918, 113, 125–140.

THE more insoluble colouring matter of camwood ("iso-santalin") is most simply expressed as  $C_{22}H_{16}O_6(OCH_3)_2$ ; it is isomeric with the santalin of sanderswood, for which the same formula is therefore proposed in place of  $C_{15}H_{11}O_4(OCH_3)$  suggested by Cain and Simonsen (this J., 1912, 580). Santalin and iso-santalin are respectively accompanied in the dyewood by the more readily soluble deoxysantalin and deoxyisasantalin, which probably have the formula  $C_{22}H_{16}O_5(OCH_3)_2$  or  $C_{22}H_{16}O_5(OCH_3)$ . Santalin from sanderswood appears to be identical with the colouring matter obtained in a similar way from barwood. Barwood also contains santal,  $C_{15}H_{11}O_4(OCH_3)$ , and santalone,  $C_{22}H_{15}O_4(OCH_3)_2$ , the latter being

identical with the substance,  $C_{14}H_{12}O_4$  described by Weidel (Z. für Chem., 1870, 6, 83). The colouring matter of St. John's wort is quercetin, and no gossypetin is present.—F. W. A.

#### PATENTS.

*Azo dyestuffs; Manufacture of red*—British Dyes, Ltd., J. Turner, and H. Dean, Huddersfield. Eng. Pat. 113,825, Mar. 6, 1917. (Appl. No. 3287 of 1917.)

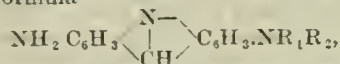
THE diazo compound from *p* chloro-*o* anisidine ( $OCH_3$ ;  $NH_2$ ;  $Cl=1:2:4$ ) is combined with 1·4-, 1·5-, or 2·6-naphtholsulphonic acid, giving a product which dyes wool a bright red, showing excellent fastness to milling or fulling.—L. A. C.

*Hydrazone dyestuffs; Manufacture of new metal compounds of*—, and process of dyeing with such compounds. O. Inray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 114,189, Mar. 20, 1917. (Appl. No. 4057 of 1917.)

HYDRAZONE dyestuffs produced by condensing a hydroxyarylhydrazine or an arylhydrazinecarboxylic acid or a hydroxyarylhydrazinecarboxylic acid, or a substitution product of one of these, with an aldehyde of the benzene, naphthalene, or furfural series, or a ketone or diketone of the aromatic series, or a substitution product of one of these, are converted into new metal compounds, soluble in water, by treating an aqueous solution or suspension of the dyestuff with a metal or metal compound, *e.g.*, copper, or a copper salt, or a salt of chromium, nickel, cobalt, iron, or the like. Similar compounds from other dyestuffs are described in Eng. Pats. 1611, 12,249, 15,456, 16,803, and 16,916, of 1915 (this J., 1916, 301, 922, 1255, and 1917, 78). The new compounds dye animal fibres in an acid bath vivid tints which are fast to light and washing without any after-treatment. Those compounds which contain both a hydroxyl and carboxyl group can be used for dyeing as described in Eng. Pat. 13,204 of 1915 (this J., 1916, 419). Where the metal or metal salt does not decompose the hydrazine compound, the metal compound may be prepared by condensation in the presence of the metal or metal salt, as described for the preparation of copper compounds of *o*-hydroxyazo dyestuffs in Eng. Pat. 15,127 of 1915 (this J., 1916, 1104). *Example:* 18 parts of the sodium salt of the dyestuff from 1-hydroxy-2-phenylhydrazine-4-sulphonic acid and resorcinyl aldehyde are dissolved in 300 parts of water at 30° C. and the solution mixed with a solution of 13 parts of crystallised copper sulphate in 20 parts of water. On adding common salt, the copper compound separates as a green-yellow precipitate, which dyes wool yellow-green in an acid bath.—L. A. C.

*Dyes of the acridine series; Halogenated*—H. Grünhagen, Berlin-Karlshorst, Assignor to Act.-Ges. für Anilin-Fabrikation, Berlin, Germany. U.S. Pat. 1,255,739, Feb. 5, 1918. Date of appl., July 29, 1916.

A NEW dyestuff of the acridine series which forms a brown-black powder dyeing leather reddish-yellow, is obtained by heating formyl-chloro-*m*-phenylenediamine ( $NH_2$ ;  $NH\cdot CHO$ ;  $Cl=1:3:6$ ) with *m*-aminodimethylaniline hydrochloride. The claims cover other dyestuffs of this series of the general formula

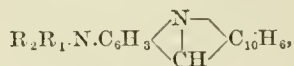


in which  $R_1$  and  $R_2$  signify univalent substituents, including hydrogen, and in which one or more hydrogen atoms in the benzene nuclei are replaced by halogens.—L. A. C.

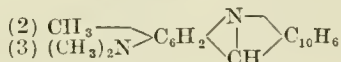


*Dyes [; Acridine—] of the benzene-naphthalene series and process for their manufacture.* H. Grünhagen, Berlin-Karlshorst, Assignor to Act.-Ges. für Anilin-Fabrikation, Berlin, Germany. U.S. Pat. 1,255,740, Feb. 5, 1918. Date of appl., July 29, 1916.

NEW dyes of the acridine series are prepared by heating a formyl derivative of a  $\beta$ -naphthylamine with a salt of a *m*-diamine of the benzene series. The general formula is:



in which  $R_1$  and  $R_2$  represent univalent substituents. In particular, the compound



forms a red-brown powder which dyes leather deep orange-yellow.—L. A. C.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Cellulose-dextrose relationship; A re-investigation of the—.* M. Cunningham. Chem. Soc. Trans., 1918, 113, 173—181.

THE theory of the ultimate and approximately complete hydrolysis of cellulose to dextrose depends in recent years on the work of Ost and Wilkening (this J., 1910, 688) who made use of the initial action of 72% sulphuric acid in the cold, the acid being subsequently diluted to 3% and heated at 110° C., and on that of Willstätter and Zechmeister (this J., 1913, 822), using cold 40% hydrochloric acid. The former supported their conclusions by the preparation of dextroazone and by fermentation results, but residual acid groups were always present. The latter authors, however, were content with optical and cupre-reducing values, which alone are not sufficient for the determination of theoretical questions of this order. The author has studied the action of cold 73% sulphuric acid upon esparto cellulose, which substantially follows a similar course to its action on cotton cellulose. Sulphuric esters of polysaccharides are formed which are isolated for analysis in the form of barium salts. The barium: sulphuric acid ratio in these products is in excess of that corresponding to an acid sulphuric ester and it would appear that an acid function is developed in the carbohydrate hydroxyl groups sufficiently powerful to decompose barium carbonate. The furfural-yielding group of the original cellulose is modified but still present in substantial amount, the furfural value falling from 12% to about 8%. The action of 40% hydrochloric acid has been studied comparatively with both cotton and esparto celluloses. The reaction, as represented by polarimetric observations after considerable dilution, follows a similar course in both cases, but in the case of esparto the initial action of the acid is less delayed and the rise in rotatory power is not so regular in the middle portion of the curve. The furfural value is practically unaffected in 24 hours and only falls to 9.9% after six days. After 48 hours the rotatory power approaches that of dextrose in both cases but the products consist of polysaccharide esters having acid properties and a lower specific rotation than dextrose and containing combined chlorine. The main results of the author's investigation of the action of both acids is that no quantitative resolution of cellulose to

dextrose has been obtained; the claim of Willstätter and Zechmeister, being based on non-specific data, cannot be admitted and the whole question of the structure of the cellulose complex remains open.—J. F. B.

*Esparto cellulose and the problems of constitution.* C. F. Cross and E. J. Bevan. Chem. Soc. Trans., 1918, 113, 182—187.

ESPARTO cellulose differs from the normal cotton cellulose mainly by the presence of furfural-yielding groups, generally assumed to represent pentosan components of the complex. Early analyses indicated a percentage of carbon considerably lower than that in cotton cellulose, but an analysis now recorded shows: carbon, 44.68 and hydrogen, 6.16%, calculated on the dry, ash-free substance, and since the yield of furfural was 12.5%, it would appear that the composition of esparto cellulose corresponds to that of a normal cellulose containing approximately 30% of the furfuroid constituent. There is no proof that this furfuroid is of pentosan configuration. On the other hand, certain treatments, of a not very energetic character, so modify the constitution of the complex that the total yield of furfural by standard methods becomes considerably reduced. Treatment at 15—20° C. with 17.5% sodium hydroxide solution produces a concentration of the furfuroid in the soluble fraction, giving 34.14% of resistant cellulose, with a furfural value of 4, and 15.86% of hydrolysed products, with a furfural value of 26, the total furfural value being reduced from 12.5 to 7.48. A still more striking fractionation was obtained with diluted sulphuric acid in the molecular ratio of  $H_2SO_4 : 3H_2O$ , acting at 18° C. The cellulose reprecipitated on dilution amounted to 78.4%, with a furfural value of only 1.5, while the hydrolysed products yielded 34.9% of furfural; in this case the total furfural value was reduced to 8.87. Stronger acid, corresponding to  $H_2SO_4 : 2H_2O$ , having a less selective hydrolysing and more pronounced esterifying action, gave 23% of precipitated colloid, with furfural value 4.6, and 77% of hydrolysed product with furfural value 8.6; the total furfural had fallen to 7.68, showing a more profound modification than in the case of the weaker acid. In the manufacture of cellulose from raw esparto, a water-soluble gum or pectic furfuroid has been isolated, showing a furfural value of 50%, and this colloid also undergoes modification on treatment with 73% sulphuric acid, with a fall in furfural value to 31.1%. The above results are interpreted as throwing doubt on the usually accepted furfuroid-pentose relationship, just as those of Cunningham (see preceding abstract) are held to re-open the question of the cellulose-dextrose relationship. The fixation of SO<sub>3</sub> groups indicates the reactivity of oxygen of basic function in excess of that which characterises the alcoholic hydroxyls of a hexose configuration.—J. F. B.

*Rope fibres; Distinguishing manila from all other "hard" —.* C. E. Swett. J. Ind. Eng. Chem., 1918, 10, 227.

WHEN the fibre is treated with a solution of bleaching powder acidified with acetic acid, and then with ammonia, the manila fibre acquires a russet-brown colour, while all the other hard fibres of the same group, *e.g.*, sisal, phormium, istle, Mauritius hemp, and maguay, will assume a cherry-red. The strand of fibre is rinsed in ether, the ether is allowed to evaporate, one end of the sample is immersed for 20 secs. in a mixture of 30 c.c. of bleaching powder solution, containing about 5% of chlorine, with 2 c.c. of glacial acetic acid. The fibre is rinsed first with water and then with alcohol and immersed in strong ammonia solution. When the test is so applied, the colour is rather

fugitive, and for quantitative purposes it is better to suspend the treated end of the strand over the ammonia for a few minutes. The colour does not develop so rapidly, but is more permanent, and the various coloured fibres can be picked out by the help of a reading glass. The correctness of the sorting can then be confirmed quickly by a qualitative test of the other ends of the fibres.

—J. F. B.

*Paper; Blue and brown print*—: characteristics, tests, and specifications. F. P. Veitch, C. F. Sammel, and E. O. Reed. J. Ind. Eng. Chem., 1918, 10, 222—226.

THE best grades of engineering blue and brown print papers are bond papers of high quality, preferably made entirely from rag fibre, so sized as to be resistant in a proper degree to the absorption of the sensitising solution and yet to take the coating uniformly. The paper is subjected to much handling both in the wet and dry condition. The durability against wear is best measured by the folding test, but the resistance to tearing in the washing treatment depends more on the tensile strength of the paper in the wet condition. This is very important when sheets of paper 10 ft. or more in length have to be handled. For papers not intended as permanent records, up to 50% of a mixture of straw or sulphite pulp may be used with the rag stock. The determination of folding endurance need only be made in the weaker direction (usually transverse) and a high resistance can only be obtained by the use of a high grade rag stock. This must be so beaten and sized as to produce a flexible sheet with a well-closed texture. Wet strength depends not only on the character of the stock and the beating, but also on the effectiveness of the sizing and the formation of the sheet. For proper coating, the surface sizing and glazing should not be too high. The mechanical properties of coated and developed blue print paper should not be inferior to those of the original paper, but brown print paper suffers great deterioration after coating, owing to the action of the sensitising chemicals, the bursting strength and, particularly, the folding resistance being very much lower. Three grades of blue print paper cover all types of requirements, but for brown printing only the highest grade is permissible. The tensile strength in the wet condition is determined on strips 15 mm. wide and 10 cm. long after immersion in water at 70° F. (21° C.) for 20 mins. Sulphite pulp mixed with the rag gives paper with low folding resistance and lacking durability. Straw mixed with rag usually gives a good wet strength and fair folding test, but the paper lacks durability. Animal sizing is favourable to the folding endurance, while excess of rosin is unfavourable. For satisfactory coating and durability the paper should not contain more than 3% of either animal or rosin size. The wet strength and the folding resistance do not necessarily run parallel. The following are the principal points in the standard specifications for uncoated and coated blue print papers and for uncoated brown print papers of medium substance (17×22 ins., 500's—24 lb.):—

Grade.	Rag stock.	Bursting test.	Wet strength.		Folding test.
			Longitudinal.	Transverse.	
	%	points.	grms.	grms.	Weak direction.
Blue 1st .....	100	50	800	500	800
" 2nd .....	100	45	800	500	500
" 3rd .....	50	38	800	500	150
Brown 1st .....	Same as Blue 1st				
" 2nd .....	100	45	800	500	400

## PATENTS.

[*Fabrics; Coating* [—] *with liquid materials.* W. B. Makins, London. Eng. Pat. 113,844, Mar. 9, 1917. (Appl. No. 3508 of 1917.)

A MECHANICAL arrangement for coating surfaces with liquid materials, particularly applicable to the "doping" of aeroplane wings, consists of a system of travelling spraying devices, arranged in a closed chamber, so as to deliver slightly overlapping bands of liquid coating material as the sprays travel over the object to be coated. The valves of the spraying devices are controlled for opening or closing, by a rod common to all, by a multiple linking, the links bearing studs for levering up the valves, and the rod being operated by tappets so as to open or close the valves at or near the ends of the stroke of the reciprocating carriage. It is important that all the sprays should act at an equal distance from the coated surface and means are provided to accommodate the adjustment for curved or irregular surfaces.—J. F. B.

*Fabric; Colloid-treated* —. S. P. Lovell, Brockton, Mass. U.S. Pat. 1,256,240, Feb. 12, 1918. Date of appl., June 27, 1917.

A SHEET of fibrous fabric is impregnated with a dissolved or diffused colloidal material which is insoluble in water, and the fabric is immediately passed to a water bath in which it is immersed until the solvent is displaced and the colloid precipitated. The interstices of the fabric are thus filled with powdered colloid material, insoluble in water.—J. F. B.

*Cloths; Method and apparatus for removing oils, fats, and greases from*—. G. W. Nagel, Assignor to W. L. Newman, jun., and C. F. Bliss, Chicago, Ill. U.S. Pat. 1,256,356, Feb. 12, 1918. Date of appl., Feb. 8, 1917.

THE cloths are subjected to centrifugal action in a perforated drum, surrounded by a casing provided with a discharge outlet. A perforated coil is fitted in the space between the casing and the drum, from which jets of steam are directed against the outer wall of the drum.—W. P. S.

*Drying-machine [for fabrics]*. J. S. Johnston, Utica, N.Y. U.S. Pat. 1,256,570, Feb. 19, 1918. Date of appl., Mar. 21, 1913.

THE fabric to be dried is drawn over the surface of a hot air pipe by a stretching and folding frame.—W. H. C.

*Straw; Process of treating*—*in the preparation of textile fibres.* V. Volpato, Milan, Italy. Eng. Pat. 113,853, Mar. 16, 1917. (Appl. No. 3873 of 1917.)

STRAW, particularly rice straw, is digested under pressure with a solution containing sodium carbonate and lime, preferably in the proportions of 2.5 kilos. of calcium oxide, 5 of sodium carbonate, and 100 of water. It is then immersed for about 50—60 minutes in an alkaline bath of suitable concentration (15°—16° B.; sp. gr. 1.109—1.118) and is subsequently washed in a bath of ammonium fluoride at 1°—2° B. (sp. gr. 1.007—1.013) and

—J. F. B.



finally in water containing a small quantity of sodium carbonate to neutralise any remaining acidity.—J. F. B.

*Cotton stalks; Process of treating*—L. D. Jones, Assignor to H. D. Deacon and Z. E. Marvin, Dallas, Tex. U.S. Pat. 1,257,080, Feb. 19, 1918. Date of appl., May 10, 1917.

COTTON stalks are disintegrated in the presence of steam, the disintegrated stalks are subjected to the action of a hot solution of caustic alkali and soap, and the treated mass is dried.—J. F. B.

*Drying apparatus [for paper]*. P. Mueller, Berlin. U.S. Pat. 1,255,915, Feb. 12, 1918. Date of appl., July 30, 1915.

A DRYING apparatus for wet sheets of paper or other material comprises two oppositely inclined flat surfaces meeting at their upper edges in a rounded joint. A heat-radiating device is provided between the surfaces.—W. F. F.

*[Paper making] machine; Fourdrinier*—C. E. Pope, Holyoke, Mass., Assignor to Great Northern Paper Co., Millinocket, Me. U.S. Pat. 1,257,737, Feb. 26, 1918. Date of appl., Apr. 27, 1915.

IN a Fourdrinier paper machine the wire after passing the couch press is carried forward round another roll situated between the couch press and the wet felt roll. The extra roll, which guides the wire back, is positively driven, e.g., from the couch roll drive, so as to maintain the wire between it and the couch press taut and at a predetermined inclination. Beneath the wire at this place a blower is arranged, so as to blow the paper off the wire after leaving the couch press and before reaching the end-roll and deliver it to the wet-press felts.—J. F. B.

*Wood-pulp; Process of producing chemical*—J. O. Lundberg, Flateby, Norway. U.S. Pat. 1,257,290, Feb. 19, 1918. Date of appl., Nov. 9, 1914.

SEE Ger. Pat. 284,628 of 1914; this J., 1915, 1048.

*Waterproof material for use in constructional purposes, as a substitute for leather, for street paving and the like*. Eng. Pat. 111,485. See IX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Wool; Absorption of sulphuric acid by*—, with some notes on the theory of dyeing. W. Harrison. J. Soc. Dyers and Col., 1918, 34, 57–58.

WOOL, after boiling with dilute sulphuric acid acquires a positive electrical charge which changes over to negative when boiling water is passed through the wool, showing that the acid has been removed. This conclusion was apparently not confirmed by Fort and Lloyd (this J., 1914, 133), who found that some of the acid was permanently retained against washing with boiling water. In the present experiments, 5 grms. of wool was boiled for one hour, with 300 c.c. of water containing 4.90% of sulphuric acid on the weight of the wool. The wool was rinsed well in cold water and then packed tightly in a glass tube. The unabsorbed acid was titrated and the wool extracted with boiling water, 300 c.c. of water being pumped through the tube for each test. Twenty-four extractions were made, the liquid from each of them being titrated separately. The wool contained 3.08% of acid when packed in the tube, about 1% after the fourth extraction, and was free from acid after the twenty-third. No measurable amount of acid was retained and the treated wool had no greater affinity for acid dyestuffs than untreated wool.

The above observations have a bearing on the dyeing of wool with acid colours. Wool previously treated with acid and washed in the ordinary way is bound to give up acid when boiled with water containing acid dyestuffs. The qualitative experiments of Fort (this J., 1913, 907) are thus accounted for. It is in accordance with the known facts that there should be conditions of concentration and temperature where the amount of sulphuric acid passing into the solution should bear a molecular relationship with the amount of dyestuff or other salt absorbed. Similar relationships have been noted between starch and barium hydroxide and between starch and iodine, but when the concentrations are altered the molecular relationship does not hold. It would therefore appear that the dyeing of wool with acid dyestuffs is a colloid-chemical problem and not an ordinary chemical reaction.—J. F. B.

*Dyeing; Theory of*—H. R. Kruyt and J. E. M. van der Made. Proc. K. Akad. Wetensch., Amsterdam, 1918, 20, 636–641.

IN the distribution of basic dyes between isobutyl alcohol and water the effect produced by the presence of the sodium salts of different acids affords a series in which the anions are arranged in the same order as in the lyotropic series. Results obtained with Crystal Violet, Magenta, and Methylene Blue show the anions in the order— $\text{SO}_4$ ,  $\text{PO}_4$ ,  $\text{Cl}$ ,  $\text{Br}$ ,  $\text{NO}_3$ ,  $\text{I}$ ,  $\text{CNS}$ . With acid dyessuch as Naphthol Yellow S and Alkali Blue, the anion influence is scarcely appreciable. Other experiments in which the influence of salts on the absorption of dyes by blood charcoal was examined show that the lyotropic influence is predominant in certain cases, whilst in others this is more or less masked by the influence of the electrical charges carried by the ions. Auramine O belongs to the former group and Methylene Blue B extra and Crystal Violet to the latter. The facts are discussed in reference to the various theories which have been put forward to explain the adsorption of dyes and the need for further experiments is emphasised. (See also J. Chem. Soc., May, 1918.)

H. M. D.

*Laundry [textile] goods; Oxygen tendering of*—P. Heermann. Chem.-Zeit., 1918, 42, 85–86.

THE use of perborate soaps and similar peroxide bleaching agents in laundries has been proved to produce, after several treatments, a very serious tendering which is more pronounced in linen fabrics than in cotton. On the other hand laundry treatments with caustic soda, soap and sodium silicate, or soap with scrubbing are comparatively mild in their effects. The destructive action of peroxide agents is greatly stimulated by the catalytic influence of certain metallic salts. Copper is extremely active and iron in a less degree (see also Briggs, this J., 1916, 80), and cloth spotted with solutions of salts of these metals is quickly tendered by perborate solutions; hypochlorite solutions act in a similar manner. The tendering of laundry goods may be attributed to the catalytic action of traces of metallic compounds in the "dirt" with which they are contaminated, and the use of all kinds of oxygenated washing agents is strongly to be condemned.

—J. F. B.

## PATENTS.

*Car-seats [dyed materials]; Process of treating [renovating]*—J. M. Wilson, Montclair, N.J., Assignor to F. Murphy, Newark, N.J. U.S. Pat. 1,256,189, Feb. 12, 1918. Date of appl., Dec. 20, 1913. Renewed June 15, 1916.

DYED materials having a nap are cleansed and renovated by first carding the material to loosen the fibres of the nap by abrasion so that micro-



scopic filaments will extend therefrom, then treating with a solution which will cleanse the nap and release a portion of the excess dye at the base of the nap, e.g., a solution containing sodium oleate and alcohol, then bending the nap by pressure applied alternately in contrary directions to bring the free ends of each portion of the nap in contact with the released dye at the base of adjacent portions, then subjecting the material to the action of trisodium phosphate to cause the re-absorbed dye to reset, lastly removing any excess of the solution from the material, and drying.—J. F. B.

*Dyeing elongated bodies of yarn or the like; Process of* —. H. Krantz, Aachen, Prussia. U.S. Pat. 1,257,886, Feb. 26, 1918. Date of appl., Apr. 8, 1914.

SEE Fr. Pat. 470,734 of 1914: this J., 1915, 174.

*Manufacture of new metal compounds of hydrazone dyesluffs and process of dyeing with such compounds.* Eng. Pat. 114,189. See IV.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Alunite deposits of Australia and their utilisation.* Advisory Council of Science and Industry. Bull. 3. pp. 38 and 9 plates.

DEPOSITS of alunite are worked at Carrickalinga, S.A. ( $K_2O$  content 10%), Warnertown, S.A. ( $K_2O$  content 7–9%), and Bullahdelah, N.S.W. ( $K_2O$  content 5.7–9.5%). The ore was, until recently, shipped to Runcorn for the manufacture of alum, but this has been suspended, and in view of the shortage of potash the Commonwealth Government has examined the possibilities of meeting it by suitable treatment of alunite. Experiments were made to increase the solubility of the potash by a simple roasting process. In laboratory trials it was found that at 500° C. the alunite was dehydrated giving a mixture of potassium aluminium sulphate and alumina. Decomposition to potassium sulphate and alumina occurred at higher temperatures and at 700° C. this was complete in 1 hour. At this temperature 77.7% of the  $SO_3$  evolved was dissociated into  $SO_2$  and  $O_2$ . The calcined residue was leached with water and filter-pressed. On crystallisation a high grade salt containing more than 90%  $K_2SO_4$  was obtained. The insoluble residue was an impure alumina, that from Carrickalinga ore containing 92%  $Al_2O_3$  and that from Bullahdelah as low as 64%  $Al_2O_3$ . Even the latter is purer than natural bauxite so that the residue may possibly be suitable for the manufacture of aluminium. Trials with a simple hand-rabbed reverberatory furnace showed that the process worked satisfactorily at temperatures from 750° to 1000° C., but above that loss of potash occurred through the formation of potassium aluminate. The decomposition was accelerated by incorporating with the ore carbonaceous matter, especially molasses, which facilitates the escape of the gases. Alunite has also been successfully used as a fertiliser in South Australia by admixture with limestone before burning. Roasted alunite also is suggested as suitable for use as a fertiliser in the vicinity of the deposits and where conditions of transport allow, American experiments having shown its superiority over potassium sulphate (Skinner and Jackson, this J., 1913, 618).—H. J. II.

*Lime; Valuation of* — for various purposes. R. K. Meade. J. Ind. Eng. Chem., 1915, 10, 214–219.

QUICKLIMES are classified as high calcium, calcium,

magnesian, and high magnesian. Magnesian limes contain 10–25%  $MgO$  and high magnesian limes 25% as a minimum. The sum of calcium and magnesium oxides must be at least 90% for selected and 85% for unselected. Carbon dioxide should not exceed 3% and 5% respectively; silica, iron oxide, and alumina, 5% and 7.5%. For constructional purposes the value of a lime depends on its physical properties although these are influenced by chemical composition; the important properties are sand-carrying capacity, crushing strength, and tensile strength. For plastering, good colour and plasticity are required; magnesian limes are generally preferred. For the chemical industries the value depends on the percentage of calcium oxide; for many purposes the presence of magnesia is not objectionable, but for the sugar and caustic soda industries it is considered harmful. Lump lime should be sampled in large quantities (100 lb.) and divided by quartering after crushing to 1 in. size; a 15-lb. sample should be sent for analysis in an air-tight container. For the slaking test 5 lb. is carefully wetted and allowed to slake for 24 hours, then washed through a 20-mesh sieve without rubbing; the residue must not exceed 3% for selected and 5% for unselected. To test the constancy of volume, equal parts of hydrated lime and Portland cement are mixed with water to a workable paste and a pat is made about 3 ins. in diam. and  $\frac{1}{2}$  in. thick at the centre, tapering to a thin edge. This pat, when hardened on a glass plate for 24 hours in moist air and then exposed to steam above boiling water in a loosely closed vessel for 5 hours, should not show any deformation or cracking. The other methods employed are the same as those in use for testing cements. For chemical purposes the percentage of free lime is the most important factor. Several methods are in use, one of the oldest being to boil the ground sample with water and titrate quickly with hydrochloric acid in presence of phenolphthalein. The Scaife method is a modification of this in which the lime is boiled with water, cooled, and treated with less than the required quantity of acid; the residual lumps are broken up, the whole is transferred to a gauged flask and allowed to settle; titration is then completed with an aliquot portion of the clear liquid. Lunge's method of titrating with oxalic acid is well known, the sample being hydrated and prepared as a milk. The sucrose method has the advantage of giving a clear solution for titration; the sucrose solution must contain between 35 and 45% of sugar. The Solvay method requires a special flask in which the lime is caused to react with ammonium chloride and the clear liquid is withdrawn for titration in presence of methyl orange. A method is sometimes used in caustic soda works depending on the causticising of a solution of sodium carbonate.—J. F. B.

*Chromates and bichromates; Determination of* —. L. W. Winkler. Z. angew. Chem., 1918, 31, 46–48.

CHROMATES, or bichromates after they have been converted into chromate by boiling with calcium carbonate, and filtering, may be determined as barium chromate or silver chromate, the former being used in the presence of chlorides and the latter if the solution contains sulphate. As *barium chromate*, 100 c.c. of the solution containing about 0.2 gm. of alkali chromate is acidified with 1 c.c. of N/10 acetic acid, 1 gm. of sodium chloride is added (this makes the barium chromate granular and easy to filter), the solution is boiled, and treated, drop by drop, with 5 c.c. of 10% barium chloride solution. After 18 hours, the precipitate is collected, washed with 50 c.c. of cold water, dried for 3 hours at 132° C., and



weighed. If the precipitate is ignited before weighing it loses 0.25% in weight. The results obtained are accurate when dealing with a precipitate weighing about 0.3 grm., but with one-tenth of this quantity the weight found is about 1 mgrm. too low. Ammonium, potassium, calcium, and magnesium chlorides do not interfere, but nitrates, chlorates, and acetates increase the weight of the barium chromate precipitate. *As silver chromate.* 100 c.c. of the neutral alkali chromate solution is heated to boiling and treated with 5 c.c. of 10% silver nitrate solution. The precipitate is collected after 18 hours, washed with 50 c.c. of water saturated with silver chromate, dried for some hours at 132° C., and weighed. The precipitate should not be exposed to daylight. The presence of nitrates, chlorates, and acetates does not interfere, but sulphates cause the results to be too high, since some silver sulphate is precipitated with the silver chromate and cannot be removed by washing. The quantity of silver sulphate thus precipitated varies with the concentration of chromate and sulphate, and a table is given showing the corrections to be applied. (See also J. Chem. Soc., May, 1918.)—W. P. S.

*Spent oxide [from gas works]; Analysis of —.*  
Z. angew. Chem., 1918, 31, 45—46.

THE following procedure is recommended for the examination of spent oxide and other materials which have been used for the purification of gas. The substance is ground in a coffee mill and the moisture is determined by heating 25 grms. at 80° C. for about 4 hours or until the loss in weight between two successive weighings does not exceed 0.05 grm. To determine sulphur 10 grms. of the finely ground dried material is shaken with 75 c.c. of carbon bisulphide for some hours, a further 25 c.c. of carbon bisulphide is then added, the mixture is shaken, allowed to settle, and 50 c.c. of the clear solution is drawn off, and filtered. The filter is washed with carbon bisulphide, the filtrate and washings are evaporated in a weighed flask, the residue in the flask is dried at 100° C., and weighed. The quantity of sulphur found is calculated as a percentage of the original, moist material. Ferrocyanides are determined by shaking 10 grms. of the finely ground dry substance with 50 c.c. of 10% potassium hydroxide solution for about 12 hours, diluting the mixture to 255 c.c., and filtering; 100 c.c. of the filtrate is then added to 25 c.c. of hot ferric chloride solution (ferric chloride, 60 grms., concentrated hydrochloric acid, 200 c.c., and water to 1000 c.c.), the mixture is kept at 80° C. for some time, filtered, the precipitate washed with hot water, and the filter and precipitate then transferred to a beaker where they are stirred with 20 c.c. of 10% potassium hydroxide solution and a quantity of water until all blue colour has disappeared. The mixture is now washed into a 250 c.c. flask, diluted to the mark, shaken after the addition of 1 grm. of lead carbonate, and filtered; 100 c.c. of the filtrate is acidified with 5 c.c. of sulphuric acid (1:5), and titrated with copper sulphate solution (12 grms. per litre) which has been standardised against pure potassium ferrocyanide. Ferric chloride solution is used as an external indicator in the titration. Exactly the same quantities of reagents must be used in the standardisation of the copper sulphate solution as in titrating the test solution, and the conditions of the titration must be the same in each case.—W. P. S.

*Iron trisulphide.* W. Mecklenburg and V. Rodt.  
Z. anorg. Chem., 1918, 102, 130—148.

A KNOWLEDGE of the properties and conditions of existence of ferric sulphide is important in connection with the use of ferric oxide for purifying coal gas. Its formation by the prolonged

action of hydrogen sulphide on an aqueous suspension of freshly prepared ferric hydroxide in absence of air is described (see this J., 1917, S2). The trisulphide can be dehydrated without decomposition *in vacuo*, but when dry is pyrophoric if brought suddenly into contact with air. If spontaneous ignition is prevented, however, it is quite stable in air. By the action of excess of sodium sulphide solution on a solution of a ferric salt, or of excess of sodium polysulphide solution on one of a ferrous salt, a precipitate is formed having the composition  $\text{Fe}_2\text{S}_3 \cdot \text{Na}_2\text{S}$ . The alkali sulpho-ferrites form dilute, deep green colloidal aqueous solutions. (See also J. Chem. Soc., May, 1918.)

—E. H. R.

*Antimony tri-iodide; A new metastable form of —.*  
A. C. Vournasos. Comptes rend., 1918, 166, 526—528.

A YELLOW amorphous form of antimony tri-iodide may be prepared by cooling a hot saturated solution of the red crystalline variety in glycerol, from which it is deposited in small yellow globules; by heating the red form on a water-bath with glacial acetic acid and a little potassium acetate; or by heating a mixture of antimony trioxide and potassium iodide at 100° C. with an excess of anhydrous acetic acid. This yellow form melts at 172° C. and at this temperature is converted into the red crystalline form. The last-mentioned method may also be used for the preparation of arsenic, antimony, and bismuth tribromides and arsenic and bismuth tri-iodides. (See also J. Chem. Soc., May, 1918.)—W. G.

*Radium sulphate; Solubility of pure —.* S. C. Lind, J. E. Underwood, and C. F. Whittemore.  
J. Amer. Chem. Soc., 1918, 40, 465—472.

IN the recovery of radium from its ores, the radium is always precipitated at some stage with barium in the form of sulphate. Information as to the completeness with which the radium is removed from solution by this process has been sought by measurement of the solubility of pure radium sulphate in water and in sulphuric acid solutions. It was found that  $2.1 \times 10^{-8}$  grm.  $\text{RaSO}_4$  is dissolved by 1 c.c. of water at 25° C. The solubility is not affected by sulphuric acid up to a concentration of 50%. The solubility of radium sulphate is only about 1/100 of that of barium sulphate. This relationship does not account for the facts concerning the removal of radium from solution by precipitation with barium as sulphate, for the ratio of radium to barium in the precipitate is always the same as in the solution. (See also J. Chem. Soc., May, 1918.)

—H. M. D.

*Iodine; Action of sodium sulphide on — and the use of the reaction in analysis.* J. Ehrlich. Z. anal. Chem., 1918, 57, 21—22.

FREE iodine is dissolved by sodium sulphide solution with the formation of sodium iodide; sulphur is liberated by the reaction but soon dissolves again in the excess of sodium sulphide. To determine iodine in a waste product or residue containing an iodide and other substances, the iodine is liberated in the usual way with potassium permanganate, and dissolved in an excess of sodium sulphide solution; manganese sulphide, etc., is separated by filtration, the filtrate is treated with zinc sulphate to remove excess of sulphide, and the iodine is then precipitated as silver iodide.

—W. P. S.

*Chlorine; Detection of small quantities of — in iodine.* J. Pinkhof. Pharm. Weekblad, 1918, 55, 236.

THE presence of chlorine (between  $\frac{1}{2}$  and 2%) in



iodine can be detected by treatment with thiosulphate, and precipitation of the sulphate formed by addition of barium nitrate.—A. J. W.

*Argon; Industrial preparation of* —. G. Claude. *Comptes rend.*, 1918, 166, 492—494.

AN application of the process of re-liquefaction. The mixture of oxygen, argon, and nitrogen resulting from the fractional distillation of liquid air, suitably compressed, is liquefied in a bath of liquid oxygen in such a way that the most volatile constituent, the nitrogen, passes on. The liquid mixture of oxygen and argon is then distilled, the gases passing up a column down which trickles some of the same liquid. The oxygen condenses and the argon passes on. By this means there is obtained a gaseous mixture containing 75—80% of argon and only 1—2% of nitrogen, the remainder being oxygen, which may be removed by burning it with hydrogen.—W. G.

*Ammonia recovery at tar-distilling plants.* Woodward. See III.

*Alternating current electrolysis [of sodium thiosulphate] with mercury electrodes.* Weiser. See XI.

#### PATENTS.

*Silicic acid; Processes for making* —. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 113,769, Nov. 9, 1917. (Appl. No. 16,456 of 1917.)

SILICIC acid in the form of a light voluminous powder is obtained by the action of an aqueous solution of an ammonium salt on water-glass; hydrated ammonium silicate,  $\text{SiO}_2 \cdot \text{NH}_3 \cdot \text{H}_2\text{O}$ , is precipitated, and the resulting mixture is then heated to the boiling point when the precipitated silicate gives off ammonia and yields silicic acid. The mixture is filtered, the precipitate washed thoroughly, then dried at 100° to 150° C. The product containing some 15% of water is passed through a 20- to 40-mesh sieve to remove coarse particles. The hydrated ammonium silicate may also be filtered off directly, and after washing, heated to about 150° C. when ammonia and water are given off. The silicic acid obtained by this process is particularly suitable for use as a fuse filler.—H. M. D.

*Nitre-cake; Method for the utilisation of* —. J. Grossmann, Manchester. Eng. Pat. 114,180, Mar. 19, 1917. (Appl. No. 3933 of 1917.)

THE nitre-cake, finely ground, is mixed with finely ground sodium nitrate and heated at 150°—250° C., when the nitrate is decomposed almost quantitatively with the evolution of nitrous fumes. The proportion in which the substances are mixed will depend on the quantity of free sulphuric acid in the nitre-cake. The process may be carried on under ordinary, augmented, or diminished pressure, and air may be either excluded from or passed through the apparatus in which the decomposition takes place. If excluded from the decomposing vessel, the air may be mixed with the nitrous fumes subsequently, with formation of nitric and nitrous acids which may be used in the manufacture of sulphuric acid. The mixture may be placed on trays carried by trolleys which are introduced into the decomposing chamber at one end where the temperature is below 150° C. and discharged at the other end, the temperature of which is about 250° C.—H. M. D.

*Ammonium sulphate and Glauber's salt; Process for the production and separation of* —. H. M. Dawson, Leeds. Eng. Pat. 114,236, May 12, 1917. (Appl. No. 6767 of 1917.)

THE process relates to the separation of ammonium sulphate and Glauber's salt from the solution which is obtained by the absorption of ammonia in solutions of nitre-cake and other acid sodium sulphates. The concentration of the solution of ammonium and sodium sulphates is appropriately adjusted so that from 70 to 80 mols. of salt are present per 1000 mols. of water. The solution is then cooled to a sufficiently low temperature, when a large proportion of the contained sodium sulphate separates out in the form of Glauber's salt. In consequence of this crystallisation, the ratio of ammonium sulphate to sodium sulphate in the mother liquor is largely increased and when the mother liquor is evaporated at or about 100° C., pure ammonium sulphate separates out so long as the molecular ratio of ammonium sulphate to sodium sulphate in the hot mother liquor does not fall below about 2.7. When this stage is reached, the evaporation is stopped, and the residual liquor is suitably diluted and mixed with further liquor for treatment in the refrigerator. The presence of free acid in the solution from the saturator does not necessitate any essential modification in the cycle of processes, but it is advantageous to reduce the acidity as far as possible by the use of an efficient saturator. The proportion of free acid in the liquor may be further reduced by the addition of sodium carbonate or liquor ammonia before the concentration or adjusted to that value which is appropriate to the carrying out of the refrigeration process.—H. M. D.

*[Iron] sulphate crystals; Method of recovering* — [from pickling baths]. W. H. Sommer, Peoria, Ill., Assignor to The First Trust and Savings Co., Cleveland, Ohio, and W. E. Stone, Peoria, Ill. U.S. Pat. 1,256,068, Feb. 12, 1918. Date of appl., Sep. 20, 1915.

AFTER concentration by means of steam coils and air-agitation, the solution is allowed to settle, without cooling, to remove insoluble impurities, and then allowed to crystallise.—W. E. F. P.

*Cyanides; Process for the synthetic production of* — [by catalysis]. Process for the synthetic production of ammonia. P. A. Starke, Berkeley, Cal. U.S. Pats. (A) 1,256,272 and (B) 1,256,273, Feb. 12, 1918. Date of appl., Sep. 11, 1916.

(A) A MIXTURE of natural gas and ammonia is passed through a reactive mass heated to about 1000° C. The reactive mass is composed of suitable alkaline substances and a metal of the iron group, and is "devoid of carbon as a chemical reactive agent." (B) A mixture of natural gas and nitrogen (or atmospheric air) is treated as above at a temperature not higher than 800° C. and a pressure of not less than 7 atmospheres.—W. E. F. P.

*Alkali metals; Process of replacing* — in alkali-metal-carrying rocks and product thereof. H. W. Charlton, New York, Assignor to Kaolin Products Corporation. U.S. Pat. 1,256,295, Feb. 12, 1918. Date of appl., May 3, 1916.

FINELY divided sodium-potassium felspar is heated with milk of lime and calcium carbonate at a pressure of not less than 200 lb. per sq. in., to form a product consisting of hydrous calcium aluminium silicate associated with a small proportion of calcium carbonate.—W. E. F. P.



*Asbestos treatment; Process of — and product thereof.* H. W. Charlton, New York. U.S. Pat. 1,256,296, Feb. 12, 1918. Date of appl., June 28, 1917.

A PRODUCT consisting of asbestos wool, the fibres of which are uniformly coated with lime, is obtained by heating asbestos wool with milk of lime under pressure.—W. E. F. P.

*Barium sulphide; Purifying —.* W. E. Prisk and H. R. Harrison, Halifax, Nova Scotia. U.S. Pat. 1,256,593, Feb. 19, 1918. Date of appl., May 31, 1917.

A SOLUTION of the barium sulphide is filtered through granulated manganese dioxide and afterwards through granulated calcium carbonate.—H. M. D.

*Nitrogen compounds of metals; Process of producing —.* M. Sem, Assignor to Det Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank, Christiania, Norway. U.S. Pat. 1,256,935, Feb. 19, 1918. Date of appl., Nov. 21, 1916.

THE carbide of the metal, in the form of fine dust, is introduced with nitrogen into a chamber, the temperature of which is raised sufficiently to start the reaction by making use of the heat evolved in the reaction between carbide and nitrogen previously introduced.—H. M. D.

*Carbon for electrochemical, metallurgical, and other services; Manufacture of —.* T. W. S. Hutchins, Middlewich, Eng. Pat. 113,812, Mar. 2, 1917. (Appl. No. 3113 of 1917.)

GROUND pitch or tar, alone or mixed with ground coke, coal, retort carbon, etc., is fed into the upper end of a closed, heated, vertical fireclay tube until the latter is filled with the carbonised product. The furnace is then allowed to cool, and the product withdrawn from the bottom of the tube in the form of a rod. For the preparation of carbon tubes, a smaller fireclay tube or rod is fixed within the larger one and the charge fed into the annular space. The density of the product is increased by conducting the carbonisation under pressure; for this purpose, and the recovery of volatile products, the fireclay tube is provided at each end with a valved outlet pipe.—W. E. F. P.

*Sulphuric acid; Concentrating —.* J. W. Leitch, Huddersfield. U.S. Pats. 1,257,894 and 1,257,895, Feb. 26, 1918. Dates of appl., Dec. 14, 1915, and Oct. 11, 1917.

SEE Eng. Pat. 1495 of 1915; this J., 1916, 308.

*Ammonium nitrate; Process of manufacturing —.* R. Blom, Rjukan, Assignor to Norsk Hydro-Elektrisk Kvælstofaktieselskab, Christiania, Norway. U.S. Pat. 1,256,513, Feb. 19, 1918. Date of appl., July 20, 1916.

SEE Eng. Pat. 101,090 of 1916; this J., 1917, 1007.

*Ammonia; Process for producing —.* A. Classen, Aachen, Germany. U.S. Pat. 1,256,875, Feb. 19, 1918. Date of appl., Oct. 8, 1915.

SEE Eng. Pat. 14,055 of 1915; this J., 1917, 547.

*Aluminous compounds; Treatment of —.* H. Spence and W. B. Llewellyn, Assignors to P. Spence and Sons, Ltd., Manchester. U.S. Pat. 1,256,605, Feb. 19, 1918. Date of appl., Nov. 3, 1917.

SEE Eng. Pat. 109,998 of 1916; this J., 1917, 1177.

*Air; Apparatus for the liquefaction and rectification of —.* H. Filippo and P. Schoonenberg, Assignors to Naaml. Vennoots. Philips Metaal Gloeilampenfabriek, Eindhoven, Netherlands. U.S. Pat. 1,257,470, Feb. 26, 1918. Date of appl., June 3, 1916.

SEE Eng. Pat. 101,860 of 1916; this J., 1917, 32.

*Manufacture of lampblack.* Eng. Pat. 114,040. See XIII.

## VIII.—GLASS; CERAMICS.

*Glass; Tank-furnace for white — fired by gas from a coke-producer.* A. B. Roxburgh. Soc. Glass Tech., Mar. 20, 1918. Gas J., 1918, 141, 586—587.

A PRODUCER of the natural draught type is built up to the tank so as to make use of the sensible heat of the gas. It has an inclined grate of bars of cruciform cross-section capable of rotation on their bearings so as to loosen and discharge ash. However turned, each bar will form a channel which can be kept filled with water from a pipe arranged to drip upon an exposed end. The bar is thereby cooled and the steam necessary to prevent clinker formation is supplied to the producer. The gas outlet is placed at the side of the producer and below the level of the top of the fuel bed. The working depth of the fuel bed is thereby maintained constant so long as the outlet is covered, and the producer need only be charged once in four hours. The gas passes through a dust-collecting chamber to the tank furnace, whence the products of combustion are removed from the opposite end and conveyed under the tank through recuperators to preheat the air supply and then to the chimney. One cwt. of gas coke free from breeze is consumed per hour, the storage capacity of the producer being about 4 cwt.—H. J. H.

## PATENTS.

*Annealing [glass, porcelain, metals] and apparatus used therein.* A. Hilger, Ltd., and F. Twyman, London. Eng. Pat. 114,183, Mar. 19, 1917. (Appl. No. 3972 of 1917.)

A SAMPLE of the substance to be annealed, which may be glass, porcelain, or a metal, is formed into a rod, one end of which is inserted into a hole in a pillar and secured by means of a screw. A weight is attached to the other end of the rod, which is disposed horizontally. The apparatus is placed in the annealing chamber together with the articles to be annealed, and when the weight has sunk a certain distance it is known that the annealing process has reached a predetermined stage or is completed. The process can be carried out without measuring the temperature of the annealing chamber. The method is based on the fact that there is a relation between the disappearance of strain in a viscous substance and the deformation of the same substance under constant stress.—H. M. D.

*Glass-drawing furnace.* G. L. Catlin and D. Morrison, Port Allegany, Pa., Assignors to R. W. Hilton, Smethport, Pa. U.S. Pat. 1,255,983, Feb. 12, 1918. Date of appl., May 19, 1917.

A GLASS furnace with an open top is fitted with a top stone having a depending annular flange, angular in cross section and with its lower edge extending horizontally. This flange engages with a movable pot-bottom the opposite faces of which are formed with a central plane portion with a diameter approximately equal to that of the flange and having a bevelled margin extending at an

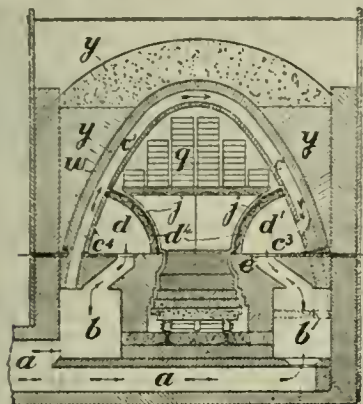
angle to the lower edge of the flange and engaging with it. Springs are provided to keep the pot-bottom and flange in contact. Means are provided for raising and lowering the pot-bottom and for rotating it when lowered.—A. B. S.

*Muffle leer [lehr].* H. M. Thompson, Washington, Pa. U.S. Pat. 1,257,150, Feb. 19, 1918. Date of appl., Oct. 1, 1917.

A MUFFLE chamber is superposed on a lehr chamber, with a horizontal division wall between. This wall consists of a number of spaced, parallel metal beams of inverted T-shape and plate-like tiles supported by adjacent beams. A number of baffle-walls resting on the beams divide the muffle chamber so as to produce a tortuous passage for the products of combustion.—A. B. S.

*Ovens or kilns for use in the manufacture of tiles, pottery and other ware and for other purposes.* J. H. Marlow, and Minton, Hollins, and Co., Stoke-on-Trent, Eng. Pat. 113,791, Dec. 4, 1916. (Appl. No. 17,366 of 1916.)

IN a tunnel oven, producer gas enters the oven through the duct, *a*, is mixed with hot air and burned in the combustion chambers, *b*, *b*<sup>1</sup>, the flames and products of combustion passing through the longitudinal flues, *d*, *d*<sup>1</sup>. Two further com-



bustion chambers (not shown) placed alongside *b*, *b*<sup>1</sup>, deliver their products into the space, *c*<sup>1</sup>, between the muffle arch, *c*<sup>1</sup>, and the outer arch, *w*, of the oven and thence into the flues, *d* or *d*<sup>1</sup>, respectively. Six such spaces are provided, the gases travelling in one direction through three of them and in the opposite direction through the other three so as to secure uniform heating. The curved sides, *d*<sup>1</sup>, of the flues, *d*, *d*<sup>1</sup>, are provided with grooves on their outer faces and are covered by curved plates, *j*, forming a series of chambers through which hot air is drawn from the tunnel and superheated before it is allowed to come into contact with the goods, *q*, to be heated. Alternatively, the longitudinal flues, *d*, *d*<sup>1</sup>, may have two sides of equal curvature, both of which are provided with air cavities, *j*. In the cooling zone of the kiln, the double arch, *w*, *c*<sup>1</sup>, is placed a little distance away from the oven wall and its inner wall, *c*<sup>1</sup>, is perforated, so that hot air may be drawn from the tunnel and through the space, *c*<sup>1</sup>. This arrangement ensures rapid cooling without "dunting." The fire-zone of the oven has a covering, *y*, of non-conducting material.—A. B. S.

[Ceramic] ware dryer and factory. A. B. Klay, Modesto, Cal. U.S. Pat. 1,256,237, Feb. 12, 1918. Date of appl., June 10, 1916.

SEVERAL superposed drying compartments are

arranged over a burning kiln in the basement of the factory, and vertical flues from the kiln pass through the compartments and serve as heat-radiators. Elevators are provided to convey the ware downwards through the successive drying compartments to the burning kiln and upwards for delivery.—A. B. S.

*Bricks and similar articles; Process of making*——. S. J. Hayde, Kansas City, Mo. U.S. Pat. 1,255,878, Feb. 12, 1918. Date of appl., July 3, 1917.

A RAW argillaceous material containing a lime-producing substance is burned for a short time at a temperature exceeding 1500° F. (815° C.), cooled slowly, treated with water so as to slake the lime, crushed, mixed with cement and water, and moulded so as to form bricks or similar articles.—A. B. S.

*Abrasives; Manufacture of artificial*——. A. T. Snodgrass, Manchester, Eng. Pat. 113,731, June 23, 1917. (Appl. No. 9023 of 1917.)

Two or three parts of finely powdered natural emery or corundum, raw bauxite, or other material rich in alumina, is mixed with one part of finely powdered glass ("flour" glass), moulded into balls, and burned in a plumbago crucible at a temperature not less than 1700° C. The molten mass is poured on to a cool surface or otherwise allowed to solidify.—A. B. S.

*Aluminous composition [abrasive] and method of preparing the same.* O. Hutchins, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,257,350, Feb. 26, 1918. Date of appl., Nov. 14, 1917.

A CRYSTALLINE aluminous abrasive is made by fusing a mixture of bauxite, barium oxide or a compound yielding it, and carbon in an electric furnace, the carbon being present in sufficient quantity to reduce the major portion of the iron, silicon, and titanite oxides present, but not the barium oxide.—A. B. S.

## IX.—BUILDING MATERIALS.

*Valuation of lime for various purposes.* Meade. See VII.

### PATENTS.

*Waterproof material for use in constructional purposes, as a substitute for leather, for street paving and the like.* D. Volant, Bordeaux, France. Eng. Pat. 111,485, Nov. 13, 1917. (Appl. No. 16,674 of 1917.) Under Int. Conv., Nov. 21, 1916.

A STRONG homogeneous waterproof material is composed of superposed sheets of paper each impregnated by immersion in a hot bath of tar or asphalt, a suitable number of sheets being applied to one another so as to produce a material of the desired thickness. The mass is then heated to a suitable temperature whilst being passed between rollers so as to compress the material into a single sheet. For use in street pavements, asphalt is preferable to tar as an impregnating material, and the sheets should be set on edge.—A. B. S.

*Waterproofed material [stone] and method of waterproofing.* E. R. Jump, Newton, Mass. U.S. Pat. 1,256,324, Feb. 12, 1918. Date of appl., May 2, 1917.

AN insoluble salt is precipitated on the surface of the particles of a mass of gypsum in order to make it waterproof.—A. B. S.



*Cement; Process of making* —. E. C. Eckel, Washington, D.C., Assignor to A. C. Spencer, Washington, D.C., and A. Cox, New York. U.S. Pat. 1,255,995, Feb. 12, 1918. Date of appl., Apr. 17, 1911. Renewed Jan. 3, 1918.

DIATOMACEOUS earth is added to a cement mixture containing iron and calcareous material so as to bring the total silica up to a desired ratio to the ferric oxide present. The clinker should contain an amount of combined silica more than twice that of the sesquioxides present.—A. B. S.

*Wood impregnation*. G. G. Carver, Assignor to The Jennison-Wright Co., Toledo, Ohio. U.S. Pat. 1,256,200, Feb. 12, 1918. Date of appl., May 20, 1915.

THE wood is submerged in a preserving fluid, and subjected to pressure, which is afterwards relieved and the excess of fluid removed. It is then immersed in an impregnating fluid under a lower pressure than that applied in the previous operation. Alternatively, the wood may be subjected to gas pressure and, without relieving this, may be treated in a bath of preservative substance under a still higher pressure. The surplus preservative is then removed and the wood subjected to a partial vacuum and afterwards impregnated by immersion in a suitable liquid under a lower pressure than that employed with the preservative fluids.—A. B. S.

*Impregnating wood; Apparatus for* —. J. H. Parsons, Assignor to W. H. Savery, Wilmington, Del. U.S. Pat. 1,257,923, Feb. 26, 1918. Date of appl., Nov. 11, 1916.

A CYLINDER is provided with a socket to receive one end of the log or piece to be treated, this socket being in communication with the atmosphere. The other end of the log is fitted into a socket attached to a piston which slides within the cylinder so as to clamp the log in the required position. The impregnating fluid is supplied under pressure to the interior of the cylinder.—A. B. S.

*Mineral wool; Means for making* —. W. Fay, St. Louis, Mo. U.S. Pat. 1,256,541, Feb. 19, 1918. Date of appl., Mar. 3, 1917.

A STREAM of mollen scoria is discharged from a cupola into a combined blast pipe and mixing chamber, and a blast of steam mixed with a dust-settling fluid is directed into it so as to form wool-fibres. The mixture passes into a chamber, in which the mineral wool fibres are collected, this chamber being provided with restricted outlets so as to maintain pressure therein.—A. B. S.

*Wood; Preservation of — and production of substances therefor*. W. P. Thompson, Liverpool. From P. C. Reilly, Indianapolis, Ind., U.S.A. Eng. Pat. 114,181, Mar. 19, 1917. (Appl. No. 3947 of 1917.)

SEE U.S. Pats. 1,220,001 and 1,230,782 of 1917; this J., 1917, 504, 866.

*[Cement] furnace structures; Rotary* —. R. Haddon, London. From Commercial Research Co., Long Island City, N.Y., U.S.A. Eng. Pat. 114,228, Apr. 24, 1917. (Appl. No. 5759 of 1917.)

SEE U.S. Pat. 1,238,394 of 1917; this J., 1917, 1130

*Process of asbestos treatment and product thereof*. U.S. Pat. 1,256,296. See VII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steels; Quenching and work-hardening of carbon* —. F. Cloup. Comptes rend., 1918, 166, 415—416.

THE heating curves of quenched or work-hardened

steels show an irregularity at 400° C. characterised by an evolution of heat. The change of state occurring at that point only proceeds slowly in either case, but it is irreversible, cooling curves in both cases being smooth and regular. For example, specimens of a steel containing 0.12% C were quenched from 1000° C. in water at 15° C. and cold-hammered respectively, and each was heated at 640° C. for 25 mins. Each specimen still showed the break in the heating curve during a second reheat, but after this had been continued for 12 hrs. at 600° C. transformation was complete and the heating curves showed no irregularity.—W. G.

*Tantalum; Estimation of — in its alloys with iron*. Travers. Comptes rend., 1918, 166, 494—495.

THE steel is treated with *aqua regia* and the tantalic acid is separated by two successive evaporations. In the case of ferro-tantalums the material is fused with anhydrous sodium sulphite and the mass is extracted with dilute acid. In either case the impure tantalic acid is fused with pure potassium hydroxide, the melt is extracted with water, and the filtrate acidified with very dilute sulphuric acid and boiled for one hour. The tantalic acid, contaminated with a little silica, is weighed and then heated in a current of hydrogen chloride at 900° C. This process removes the whole of the tantalic acid and leaves behind all the silica, which may be weighed and thus allowed for. If chromium is present a little sodium peroxide is added to the potassium hydroxide prior to the fusion. Tungsten, if present, is separated after the precipitation with dilute acid by washing the precipitate with a large excess of ammonia solution. (See also J. Chem. Soc., May, 1918.)—W. G.

*Platinum substitute [platinum-gold alloy]*. I. L. B. van der Marek. Pharm. Weekblad, 1918, 55, 149—151.

AN alloy containing about 11% Pt and 89% Au is being made under the trade name "Platino." Experiments show the alloy to be very resistant: it is, however, attacked by sulphuric acid containing nitric acid, but is more resistant to caustic potash than platinum. On heating in a smoky flame, it does not become brittle, as is the case with platinum. Its resistance to fused potassium nitrate obviates the necessity of using porcelain vessels for fusions. The cost is about one-third that of pure platinum.—F. W. A.

*Copper; Progress in the metallurgy of* —. H. C. H. Carpenter. J. Royal Soc. Arts, 1918, 66, 114—123, 128—137, 141—153.

A COPPER smelter was in operation near Keswick in 1580. The Mines Royal Society, which was constituted in that year, started smelting in Wales at Neath; 100 years later two other companies began operations at Melincrethyn and Taibach. The first smelters in the Swansea district were erected in 1717. The ores smelted in Wales were mined in Cornwall and Anglesey. During the 17th century the reverberatory furnace was evolved in Wales, all previous copper smelting having been carried out with charcoal in small shaft furnaces. The coal consumption in the Welsh process was over 18 tons per ton of metal produced. The English copper industry reached its zenith during the middle of the 19th century, but the arbitrary methods in buying ores and selling metal, pursued by the Associated Copper Smelters (the Welsh combiné) led to the introduction of smelting in Chile, which had already become the most important ore producer. In the decade 1861—70, Chile led the world's output; at the same time, mining operations at Calumet,



Mich., U.S.A., were greatly increased, while Spain, Portugal, and Australia became important producers. The Rio Tinto mine passed under English management in 1876. From 1880, the United States became the foremost copper producer, while Chile declined; Japan introduced up-to-date smelting processes. At the end of last century the United States produced more than 50% of the world's copper; the bulk of the output was derived from Arizona, Montana, Michigan, and Utah. The old Welsh, the Rio Tinto, and the pyritic processes are briefly described, as are also those used at Chuquicamata and Anaconda, including the following changes made at the latter plant in 1914—15. The slimes are treated in machines of the Minerals Separation type with 6—8 lb. of sulphuric acid, 2—3 lb. of kerosene sludge acid, and  $\frac{1}{2}$ —1 lb. of crude wood creosote per ton of feed. The latter assays 2.3—2.6% Cu, the flotation tailing 0.25%. By the adoption of flotation, the concentrator recovery has been raised from 76 to 95%. The flotation concentrate is filter-pressed, roasted, and smelted in reverberatories. The flue gases from the roasting-furnaces are treated by the Cottrell electrostatic process, which recovers 70 tons of dust per 24 hours from 1500 tons of concentrate. The new sand-leaching plant treats mill tailings as well as old dump material. This is roasted with salt and leached by downward percolation with a hot solution of salt and sulphuric acid. The liquors are precipitated with scrap-iron. The acid is made on the spot from the roaster gases. The process recovers 85% of the copper and 93.5% of the silver. The reverberatory smelting furnaces have been reconstructed and are now fired with coal-dust, whereby the output of each furnace has been more than doubled, while the fuel ratio, i.e., ratio of charge to coal, has increased from 4.2 : 1 to 6 : 1. A description of the coal-pulverising plant is also given.—W. R. S.

*Nitric acid and copper ore.* G. C. Westby. *Met. and Chem. Eng.*, 1918, 18, 290—296.

AN unsuccessful trial of a process of copper extraction from a sulphide ore is recorded. Pulverised ore was added at a carefully regulated rate to warm dilute nitric acid, the temperature being maintained by blowing in steam as required when the violence of the reaction had subsided. Agitation was effected by compressed air which also re-oxidised to nitrogen peroxide the nitric oxide evolved. The nitrous gases with excess of air were blown forward through a train of towers, where they were absorbed in water and nitric acid regenerated for use on fresh ore. Copper and iron were obtained as sulphates, and the solution was filtered and treated in the usual manner. Ten laboratory experiments showed an average recovery of copper of 88.9% and of nitric acid equal to 90.5%. The loss of nitric acid was due to imperfect recovery and also to loss in the liquors as nitrates of iron and copper. On a manufacturing scale the recovery of copper and nitric acid was much less satisfactory—67.2% and 75.7% respectively—and without an almost complete recovery of nitric acid the process is not feasible commercially.—H. J. H.

*Muntz metal (60 : 40 brass); Typical cases of the deterioration of — by selective corrosion.* H. S. Rawdon. U.S. Bureau of Standards, Technol. Paper 103, 1917. 28 pages.

By selective corrosion is meant that type of deterioration which is brought about by electrolytes (e.g., sea water) through the attack of certain microstructural constituents of the alloys leading, in the present instance, to "dezincification." Typical cases of such corrosion of Muntz metal

have been examined, including bolts and sheathing from the keels of sea-going boats, and condenser tubing. The paper is illustrated with numerous photomicrographs of the specimens described. Normally, the structure of Muntz metal consists of a matrix of  $\beta$  constituent, relatively rich in zinc, in which are embedded crystals of the  $\alpha$  constituent which is poorer in zinc. In all cases the  $\beta$  constituent is attacked first, the rate at which the  $\alpha$  constituent is attacked being variable and sometimes indefinitely delayed. The metal when thus attacked becomes red in appearance, like poor copper, and is very weak and porous. The micrographs show that the attack on the  $\beta$  phase is not a gradual one but is abrupt and complete, the line of demarcation between the attacked and unattacked portions being very sharp. Dezincification may also occur in a brass, but the method of attack is different. Samples which have been heat-treated have an outer layer of  $\alpha$ -brass from which excess of zinc has volatilised, and this layer serves as a protective coating for the  $\alpha$ - $\beta$  alloy beneath. The author's experiments do not show any relation between time or temperature of annealing of the metal and susceptibility to attack by salt solution. The character of the surface may be of importance, since at those points where specks of basic zinc chloride accumulate, corrosion is specially rapid. Other influences which may accelerate the rate of attack are contact with substances more electronegative than either  $\alpha$  or  $\beta$  constituent, an increase of temperature, or localised stresses. Whilst the results of the author's experiments suggest that highly localised stresses increase the susceptibility to attack, they are not regarded as conclusive.—E. H. R.

*Bronze; An investigation of unsound castings of Admiralty — (88 : 10 : 2); its cause and the remedy.* H. C. H. Carpenter and C. F. Elam. *Inst. of Metals*, March, 1918. [Advance copy.] 21 pages.

WHEN Admiralty bronze is poured at 1400° C., unsound castings are always obtained, due to the presence of blowholes. Pouring below 1100° C. very often causes unsoundness. The metal is always sound if left to cool in the crucible, even if overheated; this also applies to previously cast unsound metal. Hence the temperature of casting is the deciding factor. The blowholes are formed by the liberation of dissolved gases. These were collected *in vacuo* and analysed: they were found to consist of typical furnace gases, whether the castings were sound or unsound. No constant or sufficient difference in the volume of gas could be found to account for the presence or absence of blowholes. Pure copper heated *in vacuo* yields gas resembling that obtained from bronze, hence the gases which cause unsoundness are very probably derived from the copper itself, especially as they are most difficult to expel. The best way of avoiding unsound castings is to control the temperature before pouring, so that it is within the interval 1270°—1150° C.—W. R. S.

*Aluminium; Annealing of —.* R. J. Anderson. *Inst. of Metals*, Mar. 14, 1918. [Advance copy.] pp. 3 and 1 plate.

FIVE specimens of aluminium sheet (10 gauge) were subjected to chemical and microscopical examination. The iron content (0.74 to 0.90%) was higher than usual in American metal of good quality. Shore hardness numbers varied from 6 to 9, whereas the usual numbers for such sheet are 13—15 in the severely worked specimens and 4—5 in annealed specimens. The samples in question had probably been partly annealed after cold rolling. The metals were found to be almost structureless or with crystals deformed



in the direction of work. On annealing at 540° C. for 15 mins. followed by slow cooling a typical structure was developed. Excessive initial deformation appeared to retard crystal growth. (For the polishing and etching of aluminium see this J., 1918, 211 A.)—H. J. H.

*Aluminium-copper alloys; Constitution of the copper-rich*—. J. N. Greenwood. Inst. of Metals, March, 1918. [Advance copy.] 46 pages.

THE relation between the hardness and composition of alloys containing less than 16% Al was investigated by the Brinell and scleroscope methods, the former being more satisfactory on the whole. The hardness-composition curve of the  $\alpha$ -series of solid solutions (up to about 9.7% Al) was found to be linear. Hence the idea that the upper limit of this series of solid solutions is formed by the compound,  $\text{Cu}_4\text{Al}$ , cannot be maintained. The curve for the quenched  $\beta$ -series of solid solutions showed a minimum at 12.5% Al, which confirms the existence of  $\text{Cu}_3\text{Al}$ . In the case of alloys containing  $\alpha + \bar{c}$ ,  $\alpha + \beta$ , or  $\beta + \bar{c}$ , there is a marked deviation from the linear relationship between hardness and composition. Alloys with 8.7–13.3% Al reached a state of phase equilibrium after 20 days at 600° C. The results of a separate investigation of  $\beta$  will shortly be published.

—W. R. S.

*Aluminium-bronze; Die-casting of*—. H. Rix and H. Whitaker. Inst. of Metals, March, 1918. [Advance copy.] 9 pages.

THE advantages of aluminium as a die-casting metal are its low sp. gr., cheapness, and strength. Its chief drawbacks are high melting-point and shrinkage, weakness at high temperatures, and the tendency of the molten metal to attack iron. The high shrinkage can be reduced by alloying, and need not exceed about 1.4%. Aluminium-bronze containing about 10% Al does not give well-defined edges, but much better results are obtained if 1–4% Fe is added. The mechanical properties may be profoundly modified by heat treatment; the temperature of the molten metal and die should be known, and the rate of cooling of the casting standardised. The dies are made of close-grained cast iron, as hard as is consistent with good machining properties. From 5000 to 7000 castings can be made before the die shows signs of deterioration.—W. R. S.

*Metals; Velocity of dissolution of*— in acids. III. *Velocity of dissolution of alloys of zinc with arsenic, lead, cadmium, nickel, platinum, and gold*. M. Centnerszwer. Z. physik. Chem., 1918, 92, 563–580.

THE rate at which zinc dissolves in hydrochloric acid (compare this J., 1917, 292) is not increased when the metal is alloyed with small quantities of arsenic, lead, or cadmium. On the other hand, very small quantities (0.01%) of nickel, platinum, or gold produce a marked increase in the rate at which the zinc is attacked by acids. It seems probable that pure zinc will not react at all with hydrochloric or sulphuric acid if the concentration is less than 0.5–1 N. (See also J. Chem. Soc., May, 1918.)—H. M. D.

*Lead-tin-antimony alloys*. O. W. Ellis. Inst. of Metals, March, 1918. [Advance copy.] 4 pages.

THE general mechanical properties of alloys containing less than about 15% Sn were improved by the addition of antimony in quantities not exceeding 10%. An increase in the antimony content led to increased hardness, though there

appeared to be a region of maximum hardness in the vicinity of the 70:10:20 alloy. On the whole, the alloys were brittle and weak.—W. R. S.

*Alloys; Theoretical and experimental researches on metallic*—. N. Parravano. Annali Chim. Appl., 1918, 9, 1–86.

A RESUMÉ of the author's investigations on alloys, including theoretical studies on ternary and quaternary alloys. The systems which have been studied experimentally include Sn—As (this J., 1911, 1018), Sb—As (this J., 1912, 495), S<sup>b</sup>—Se, Bi—Se, Mn—Zn (this J., 1915, 286), Mn—Bi (this J., 1915, 802), Sb—K, Cu—Zn (this J., 1915, 86), Ni—Mn—Cu and Fe—Mn—Cu (this J., 1912, 1131), Fe—Ni—Mn (this J., 1912, 1081), Cu—Au—Ag and Ni—Cu—Au (this J., 1914, 261), Ni—Au—Ag (this J., 1913, 1158), Ni—Cu—Ag (this J., 1913, 1112), Ni—Cu—Pb (this J., 1914, 1212), Cu—Zn—Pb (this J., 1915, 86), Ag—Pb—Sn (this J., 1911, 1120), Cu—Sb—Bi, Pb—Sn—Cd—Bi (this J., 1912, 778), Fe—Ni—Mn—Cu (this J., 1913, 89), and Ni—Cu—Au—Ag (this J., 1914, 1092).

*Flotation; Effect of addition agents in*—. III. M. H. Thornberry and H. T. Mann. Met. and Chem. Eng., 1918, 18, 249–252.

THE experiments previously described (this J., 1918, 93 A, 153 A,) have been extended to include the use of chlorides. Chlorides of alkalis and alkaline earths, and hydrochloric acid reduce slightly the extraction and grade of concentrate. The effect of chlorides of the heavy metals is on the whole similar but more erratic. Silver chloride had a marked effect on the extraction when cresylic acid was used. Very detrimental effects on both extraction and grade of concentrate were noted with stannous chloride and uranium chloride, and in this respect cadmium chloride behaved similarly to the other salts of this metal. The froths were uniform and composed of small or medium-sized bubbles which broke up easily.—H. J. H.

*Slag viscosity tables; Blast furnace*—. A. L. Feild and P. H. Royster. Amer. Inst. Min. Eng., Feb. 18, 1918. Blast Furnace and Steel Plant, 1918, 6, 117–120.

THE method of measuring the viscosity of liquid slags already described (this J., 1918, 61 A) has been applied to typical blast-furnace slag mixtures. Provided that minor constituents are not present in abnormal quantities and that the MgO content does not exceed 8%, the viscosity was found to be a function of the relative percentages of lime, alumina, and silica. Tables have been constructed to cover the slag viscosities of such ternary mixtures over a wide range of variation of composition and temperature (U.S. Bureau of Mines Tech. Paper 187). In the present paper the application of these tables to blast-furnace problems is discussed. Low viscosity at low temperatures is favourable to fuel economy. High viscosity necessitates high working temperature and therefore favours the reduction of silica as in the manufacture of foundry iron. Thus high alumina content of slag favours the production of high-silicon pig. A long temperature range of the pasty stage conduces to lengthy contact of metal and slag and so favours desulphurisation in the basic pig process. The tables show that slight changes in composition sometimes cause a marked change in the viscosities of slags, emphasising the necessity for accurate adjustment of the burden and the chemical control of slags.—H. J. H.

*Spectroscopic detection of boron [in steel and in minerals]*. De Gramont. See XXIII.

## PATENTS.

*Steel; Manufacture of*—[in electric furnaces]. C. A. Keller, Paris. Eng. Pat. 113,635, Feb. 17, 1917. (Appl. No. 2391 of 1917.)

A QUANTITY of iron and steel turnings or similar light waste metal is subjected to desulphurising fusion in one or more electric furnaces to obtain a crude metal of approximately the desired composition, and this is transferred to a finishing furnace, which may be either a Martin furnace or an electric furnace. The melting furnace is kept full of turnings, mixed with a basic slag, and casting is effected at intervals. A proportion of carbon exceeding the final amount desired may be added to the metal in the melting furnace, so that the desired final content can be readily attained in the finishing furnace in which dephosphorisation is effected.—T. H. B.

*Metals [iron and steel]; Refining of*—. J. H. Beckett, Birkenhead. Eng. Pat. 113,834, Mar. 8, 1917. (Appl. No. 3414 of 1917.)

AIR is forced at a pressure of 20 to 80 lb. per sq. in. into the metal while in a furnace of the "mixer" type, through tuyères which pass down obliquely through the surface of the metal. The tuyères are protected by a fire-resisting medium or are cooled by water. The partial elimination of silicon, manganese, carbon, and phosphorus is thereby effected.—T. H. B.

*Iron sponge; Treatment of*—. A. Sinding-Larsen, Christiania, Norway. Eng. Pat. 114,025, Mar. 14, 1917. (Appl. No. 3722 of 1917.)

IRON sponge is treated with carbon monoxide at about 100° C., and at the same time subjected to a rubbing or disintegrating action; the vapours of iron carbonyl thus produced are brought into contact with a thin iron core forming the secondary of an electrical induction furnace, so heated that the iron carbonyl is decomposed and iron deposited on the core. The iron thus obtained is further carburised by heating at a higher temperature in presence of hydrocarbons. Nickel steel may be obtained by adding nickel carbonyl to the iron carbonyl before passing it into the induction furnace.—T. H. B.

*High speed steel; Process [and flux] for welding*—to other steel or iron. A. F. Beaulieu, Chicago, Ill. U.S. Pat. 1,256,429, Feb. 12, 1918. Date of appl., Dec. 21, 1915.

A FLUX for use in welding metals containing different percentages of carbon consists of sodium bicarbonate 40%, sodium carbonate 40%, sodium borate 10%, precipitated silica 5%, lithium carbonate 5%.—T. H. B.

*Magnetic iron ore; Separation of*—from its associated non-magnetic or feebly-magnetic minerals. D. M. Barringer, Wayne, Pa. U.S. Pat. 1,256,972, Feb. 19, 1918. Date of appl., May 10, 1911. Renewed July 17, 1917.

MAGNETIC material is separated from non-magnetic or less magnetic substances by conveying the mixed materials by water down an inclined trough of substantially non-magnetic material of high electrical conductivity, and subjecting the substances to the influence of a magnetic flux. The water separates the particles and reduces the friction of the same on the trough and on each other. The magnetic flux is created by an electromagnet, on the under side of the plate or trough, which is energised by an alternating current. The magnetic materials are diverted from their normal path of travel, passing towards and over the edges of the trough.—B. N.

*Composite bi-metallic articles [thermostatic metal]; Manufacture of*—. The British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 113,758, Sept. 18, 1917. (Appl. No. 13,406 of 1907.)

A BAR of "invar" (Ni 35.5%, C 0.18%, Mn 0.42%, Fe 63.9%) contained in a mould is heated to bright redness, and a flux (e.g., borax) applied to the surface; one end is then brought to a fusing temperature and brass melted upon it to alloy with the "invar." This "brassing" is continued until the entire upper surface of the bar has received a uniform layer of brass, about  $\frac{1}{8}$ -inch thick. While the brass layer is in the molten condition sufficient molten brass is poured into the mould to produce the desired thickness in the composition ingot, which is afterwards passed through suitable rolls and reduced to the required dimensions; lead-free brass is used.—T. H. B.

*Tungsten ingots and the like; Method of and apparatus for welding or sintering*—. M. J. Insull, Chicago, U.S.A. Eng. Pat. 113,835, Mar. 8, 1917. (Appl. No. 3422 of 1917.)

TUNGSTEN ingots or the like are sintered whilst subjected to a continual longitudinal pressure between electrodes, the ends of the latter being maintained perfectly parallel during longitudinal movement, so as to produce a straight bar or ingot which is held in position horizontally by the electrodes. A portion of the electrodes at least is composed of tungsten treated with carbon, and the electrodes, which are maintained under spring pressure, are of the same, preferably square, cross-section as the ingot. The electrode supports extend from or are supported by a stationary head or cover, which co-operates with a movable chamber to hold the ingot substantially horizontally between them, and the chamber is pressed by a device firmly against the cover to form a gas-tight chamber. One of the electrode supports is provided with a guide and guided portion, by which that electrode may be moved towards or away from the other whilst still maintaining parallelism of the electrode ends, a flexible conductor being provided and having off-set parallel portions connecting the guided portion of the electrode support with the guide portion.—B. N.

*Tungsten powder and the like; Compressed*—and method and apparatus for producing the same. M. J. Insull, Chicago, U.S.A. Eng. Pat. 113,836, Mar. 8, 1917. (Appl. No. 3423 of 1917.)

MALLEABLE tungsten articles are produced by first compressing tungsten powder to an unsintered form of a strength substantially equal to that of blackboard chalk and with sp. gr. about 17.25, by a pressure of from 135 to 160 tons per sq. in. and then relieving the pressure so that the compressed powder expands substantially equally in all directions. For this purpose a female die is formed of a movable lining seated in a die block, comprising four separate die members, two of which are preferably rigidly connected together, or two members may be formed with recesses in which the other two members engage, the block with the four die members forming five plane walls of a rectangular chamber with diverging walls in which the tungsten powder is compressed by the co-action of the male die. When the latter is withdrawn after compression, the slight lateral movement of the walls of the lining is such as to make the lateral expansion of the chamber about the same as the vertical enlargement.—B. N.



*Furnaces; Electric* — [for manufacture of tungsten powder]. M. J. Insull, Chicago, U.S.A. Eng. Pat. 113,839, Mar. 8, 1917. (Appl. No. 3426 of 1917.)

A FURNACE for the reduction of metallic oxides, particularly for the reduction of tungsten trioxide to metallic tungsten powder, is formed from a refractory silica tube, provided along part of its length only with a thin sheet nickel lining for protection from injurious chemicals, especially tungsten suboxides liberated during the reduction process. The tube is surrounded by a heating element, and a rapid current of hydrogen, preheated to a temperature substantially equal to that at which the material is maintained for reduction, is passed over the tungsten trioxide or other metallic oxide in the tube, preferably at a pressure exceeding that of the atmosphere. The hydrogen and metallic oxide are maintained at a substantially constant temperature throughout the length of the reducing portion of the furnace. The heating element for the hydrogen is arranged near the point where the gas enters the tube, and comprises a ribbon of conducting material, preferably nichrome, loosely wound upon a central support, each convolution being formed so as to engage the support and to extend away from the support substantially to the inner wall of the furnace. This form is secured by winding the element spirally about a flat mandrel, withdrawing the latter and permitting the element to uncoil, when it is afterwards secured to a central support within the furnace. A gas spreader is arranged near the point of entry so as to prevent the formation of stationary gas pockets about the heating element.—B. N.

*Gas-fired furnaces, oil-fired furnaces, and the like* [for heating and melting metals]. I. Hall, Birmingham. Eng. Pat. 114,177, Mar. 15, 1918. (Appl. Nos. 3746, Mar. 15, 1912, Mar. 19, and 12,201, Aug. 25, 1917.)

A FURNACE for melting metals, or heating bars, shells, etc., fired by means of one or more oil or gas burners, is provided in its upper part with a metallic conduit in which the air or mixture of air and fuel is preheated. This conduit is completely embedded in the refractory lining of the furnace in such a manner that a certain thickness of refractory material lies between the conduit and the heating chamber to protect the conduit from contact with the flame. The outlet from the conduit is connected to the gas or oil burners which heat the furnace. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 25,106 of 1906 and 103,232; this J., 1917, 200.)—J. H. P.

*Aluminium; Solder for* —. J. J. Aubertin, jun., St. Louis, Mo. U.S. Pat. 1,256,285, Feb. 12, 1918. Date of appl., Oct. 15, 1917.

The solder is composed of two parts of zinc to one of tin with a relatively small proportion of sal ammoniac (ammonium chloride).—T. H. B.

*Ores; Reduction of* —. S. Westberg and E. Edwin, Christiania, Norway. U.S. Pat. 1,256,623, Feb. 19, 1918. Date of appl., Aug. 26, 1916.

CARBONACEOUS material, and a gas containing combined oxygen, e.g., carbon dioxide and water vapour, are subjected to a high-tension electric arc, and the resulting carbon monoxide and hydrogen used to reduce an oxide ore.—T. H. B.

*Ore-concentration process.* B. H. Dosenbach, Butte, Mont. U.S. Pat. 1,257,329, Feb. 26, 1918. Date of appl., Nov. 16, 1917.

A MIXTURE of air with a "modifying agent" in

the form of a fog is introduced through a porous medium of fine texture into an ore pulp; certain mineral particles adhere to the bubbles formed as they rise through the pulp, forming a column of bubbles above the pulp.—T. H. B.

*[Hot-blast furnaces;] Operation of chequer-brick heaters* [for —]. A. Pföser, Achern, and O. Strack, Saarbrücken, Assignors to Gebr. Stumm, G.m.b.H., Neunkirchen, Germany. U.S. Pat. 1,257,524, Feb. 26, 1918. Date of appl., Aug. 26, 1913.

IN heating chequer-work brick heaters of the regenerative type for hot-blast furnaces, the transfer of heat from the hot gases to solid surfaces is promoted by violent agitation and increased velocity of the hot gases. This is obtained by introducing hot blast-furnace gas and air in quantities largely in excess of that normally passing through the heater to the stack, so that all particles of gas are brought into frictional contact with the walls of the chamber.—W. F. F.

*Precious metals; Process of extracting* — from ores containing alkaline-earth carbonates, etc. J. G. Kirchen and D. B. Bradner, Tonopah, Nev. U.S. Pat. 1,257,612, Feb. 26, 1918. Date of appl., Aug. 8, 1917.

ORE containing calcium carbonate and other alkaline-earth carbonates is roasted at a temperature exceeding 600° F. but less than 1200° F. (315°—650° C.) so that the carbonates are not decomposed to oxides, and the roasted ore is then subjected to cyanide treatment.—T. H. B.

*Iron sponge; Production of* — from iron ore. A. Sinding-Larsen, Vestre Aker, Norway. U.S. Pat. 1,256,939, Feb. 19, 1918. Date of appl., Mar. 21, 1917.

SEE Eng. Pat. 113,351 of 1917; this J., 1918, 184A.

*[Zinc] sulphate liquors; Process for the electrolysis and treatment of* —. E. J. Hunt, Oldbury, and W. T. Gidden, Warley, Assignors to Chance and Hunt, Ltd., Oldbury. Re-issue 14,436, Feb. 26, 1918, of U.S. Pat. 1,107,309, Aug. 18, 1914. Date of appl., Sept. 21, 1917.

SEE Eng. Pat. 11,634 of 1913; this J., 1914, 692.

*Zinc; Process of extracting* — from its ores. E. A. Johansson, Trollhättan, Sweden, Assignor to Soc. Anon. Metallurgique Proc. De Laval, Brussels. U.S. Pat. 1,256,802, Feb. 19, 1918. Date of appl., Mar. 22, 1912.

SEE Fr. Pat. 441,697 of 1912; this J., 1912, 931.

*Manufacture of carbon for electrochemical, metallurgical, and other services.* Eng. Pat. 113,812. See VII.

*Method of recovering [iron] sulphate crystals* [from pickling baths]. U.S. Pat. 1,256,068. See VII.

*Annealing [glass, porcelain, metals] and apparatus used therein.* Eng. Pat. 114,183. See VIII.

*Means for making mineral wool.* U.S. Pat. 1,256,541. See IX.

*Electric resistance element.* U.S. Pat. 1,257,272. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Alternating current electrolysis* [of sodium thiosulphate] with mercury electrodes. H. B. Weiser. J. Phys. Chem., 1918, 22, 78—94.

WHEN an alternating current is passed through a solution of sodium thiosulphate between mercury

electrodes, the electrodes are corroded and mercuric sulphide is precipitated. The yield of sulphide decreases as the frequency of alternation increases. Similar effects are obtained with other metal electrodes, but with mercury the effects are distinguished from those with solid metals in that reproducible results are obtainable. The electrolytic yield increases with the concentration of the thiosulphate solution, provided that this is not large enough to produce a film of sulphide which cuts down the corrosion very considerably, and also with the current density. Rise of temperature also causes a slight increase in the electrolytic yield. The experiments were made with a current alternating 7200 times per minute. (See also J. Chem. Soc., May, 1918.)—H. M. D.

#### PATENTS.

*Furnaces; Control and regulation of electric* —. Brown, Boveri, and Co., Ltd., and A. Gray, London. Eng. Pat. 113,827, Mar. 6, 1917. (Appl. No. 3318 of 1917.)

THE furnace is controlled by automatic variation of the length of the arc, a motor-generator combination being so arranged that the power supplied to the motor is proportional to the variation in the predetermined current input of the furnace, and regulation being effected without making or breaking any of the controlled circuits during the period of automatic regulation. The direct current motor is supplied with current from a separately excited direct current generator, the current being controlled by an automatic regulator having a rotating field, influenced by any variation in the working current, and an additional spring control. A variable non-inductive resistance, in parallel or in series with the electro-magnetic field system of the automatic regulator, is provided for readjusting the current input to any desired value. A number of electrodes may be operated by a single motor responsive to the variations in the average current taken by the furnace.—B. N.

*Furnace; Electric heating* —. *Method of heating*. E. E. Walker and J. W. Cox, Erie, Pa. U.S. Pats. (A) 1,256,958 and (B) 1,256,959, Feb. 19, 1918. Dates of appl., (A) Jan. 2, 1917. Renewed July 11, 1917, (B) Feb. 15, 1917.

(A) AN electric heating furnace comprises a heat-diffusing electrode, which is liquid under the heat of the arc, a supporting floor for the material to be treated, and an enclosing wall including a space of greater area than the electrode, the wall being in such a position that it receives and deflects the heat from the liquid electrode. A second electrode is arranged to act with the liquid electrode for completing the arc. (B) An electric arc is formed, and the heat taken up, diffused, and communicated through the medium of a liquid electrode, which is melted by the heat of the arc, the matter to be heated being independent of the liquid electrode.—B. N.

*Electrical contacts [of tungsten or tungsten alloys]*. M. J. Insull, Chicago, U.S.A. Eng. Pat. 113,838, Mar. 8, 1917. (Appl. No. 3425 of 1917.)

AN electrical make and break contact, of pure tungsten or tungsten alloyed with other rare metals such as molybdenum, is made by cutting a disc of metal from the end of a rod which is drawn, or which has the grain thereof running lengthwise of the rod, the grain being thus perpendicular to the contact face.—B. N.

*Resistance element; Electric* —. G. M. Laird, Assignor to L. B. Lincoln, Chicago, Ill. U.S. Pat. 1,257,272, Feb. 19, 1918. Date of appl., May 19, 1916.

AN element formed of an alloy of nickel, cobalt, and silicon, containing at least 75% Ni.—B. N.

*Primary cells; Galvanic* —. R. Schuster, Puppling, Germany. Eng. Pat. 114,012, Mar. 12, 1917. (Appl. No. 3584 of 1917.)

A TWO-FLUID cell contains zinc in caustic alkali, and carbon in an aqueous solution of potassium or sodium bichromate or permanganate to which is added a large excess of concentrated sulphuric acid.—B. N.

*Electrolytic apparatus*. H. T. Shriver, West Orange, N.J. U.S. Pat. 1,256,067, Feb. 12, 1918. Date of appl., Jan. 20, 1916.

AN electrolytic cell of the filter-press type comprises a number of flat plates held face to face, the plates being recessed to form a cell between adjacent faces, with a diaphragm between. Each plate is provided with a U-shaped duct formed in the walls, one arm being relatively short and connected to the recess, and the other relatively long and opening to the outside of the plate. A constantly running supply of diluent is supplied to the long arm at a faster rate than that of exhaustion from the cell, the long arm being so arranged that the excess overflows after a predetermined level has been reached, thus keeping the liquid at a constant pressure.—B. N.

*Electrolytic cell*. T. G. Hitt, Seattle, Wash. U.S. Pat. 1,256,319, Feb. 12, 1918. Date of appl., Oct. 26, 1917.

THE receptacle containing the electrolyte is provided with a cover from which boxes hang side by side, vertical grooves in the adjacent faces registering together, and registering with grooves in the cover. Carbons pass through the apertures made by the grooves. Each box is provided with a vertical slot containing anode material, and wires connect the electrodes with a source of electricity, the connections with the anode material being within the boxes, which are sealed to protect the connections from electrolytic action.—B. N.

[*Electric battery plate separators.*] *Composition of matter*. J. Becker, Chicago, Ill. U.S. Pat. 1,256,861, Feb. 19, 1918. Date of appl., Nov. 27, 1916.

SEPARATORS for battery plates are formed from a pasty mixture of powdered volcanic ash (50 parts), powdered calcium sulphate (40 parts), and sulphuric acid, sp. gr. 1.285—1.357 (50 parts), in conjunction with a base of asbestos fibre (1 part).—B. N.

*Manufacture of carbon for electrochemical, metallurgical, and other services*. Eng. Pat. 113,812. See VII.

*Electric furnace [for the manufacture of tungsten powder]*. Eng. Pat. 113,839. See X.

*Process of refining sugar*. U.S. Pat. 1,256,758. See XVII.

## XII.—FATS; OILS; WAXES.

*Sesamé oil reaction; Modified* —. F. Weehuizen. Pharm. Weekblad, 1918, 55, 77—79.

THE work of Van Ekenstein and Blanksma (this J., 1909, 536) showed that  $\beta$ -hydroxy- $\alpha$ -methylfurfural, and not furfural, is formed by the action of hydrochloric acid on hexoses; in a later paper Blanksma (this J., 1910, 833) states that the product is  $\omega$ -hydroxymethylfurfural, the aldehyde



corresponding to 2-hydroxymethylfuran-5-carboxylic acid. The substitution of furfural for the sugar in Baudouin's reaction for sesamé oil is therefore incorrect. This reaction is carried out by shaking a small amount of solid or liquid levulose (sucrose may be used) with 3–5 c.c. of a fully saturated alcoholic solution of hydrochloric acid for about 1 min., adding an equal volume of the oil under examination, and again shaking vigorously for about 1 min. A 5% solution of sesamé oil in olive oil gave a purple alcoholic layer on allowing to stand for 5 mins.: unadulterated samples gave a light red or greenish colour.

—F. W. A.

*Soap solutions. III.* V. Lenher and G. M. Bishop. *J. Phys. Chem.*, 1918, 22, 95–98.

COMPARATIVE experiments in which *N*/10 sodium oleate solution was filtered through layers of animal charcoal, willow charcoal, and Ceylon graphite respectively show that the adsorption of the soap by the three forms of carbon diminishes in the order in which these are written. (See also *J. Chem. Soc.*, May, 1918.)—H. M. D.

*Glycerol; Estimation of — by Wagenaar's method.* J. H. M. Beckers and I. M. Kolthoff. *Pharm. Weekblad*, 1918, 55, 272–281.

WAGENAAR'S method of estimating glycerol gives results dependent on the composition and temperature of the liquid. Methyl alcohol is without influence on the reaction, but the presence of ethyl alcohol, halides, nitrates, and to a less degree sulphates, is deleterious. With fats, it is best to saponify with methyl-alcoholic potash, and liberate the fatty acids with the minimum quantity of sulphuric acid.—A. J. W.

*Glycerin; Estimation of the amount of water in —.* I. M. Kolthoff. *Pharm. Weekblad*, 1918, 55, 301–307.

AN account of a method of estimating the percentage of water in glycerin by observing the temperature range of complete miscibility of mixtures of the sample and aniline. A table showing the corresponding percentages of water is given.

—A. J. W.

*Fats and fatty acids from petroleum.* Moore and Egloff. See IIA.

#### PATENTS.

[*Hydrogenation of oils;*] *Process of making metallic catalysts [for —].* W. D. Richardson, Chicago, Ill. U.S. Pat. 1,257,396, Feb. 26, 1918. Date of appl., Nov. 11, 1912.

A PERMANENT catalyst, consisting of an electrically disintegrated colloidal metal in a fluid fatty mass, is obtained by producing an electric arc between two metallic, *e.g.*, nickel, electrodes submerged in a liquid or semi-liquid fatty substance, until from 1 to 20% of the metal has been disintegrated, stirring being maintained the while to diffuse the disintegrated metal throughout the mass.

—A. DE W.

*Hydrogenating oils or fats; Process of —.* W. D. Richardson, Assignor to Swift and Co., Chicago, Ill. U.S. Pat. 1,257,397, Feb. 26, 1918. Date of appl., Nov. 11, 1912.

A METAL is electrically disintegrated in a fluid medium, *e.g.*, water, the fluid medium removed,

and the disintegrated metal added to the oily or fatty material, which is then treated with hydrogen.

—A. DE W.

[*Hydrogenation of oils;*] *Process for making catalysts [for —].* W. D. Richardson, Assignor to Swift and Co., Chicago, Ill. U.S. Pat. 1,257,531, Feb. 26, 1918. Date of appl., May 6, 1915.

A COLLOIDAL metallic catalytic material is obtained by electrically disintegrating thick electrodes comprising nickel in a liquid medium, *e.g.*, water, the finely-divided precipitate containing nickel being collected, freed from liquid medium, and then mixed with oil.—A. DE W.

*Method and apparatus for removing oils, fats, and greases from cloths.* U.S. Pat. 1,256,356. See V.

*Process for making drying oils from mineral oils.* U.S. Pat. 1,256,535. See XIII.

*Process of making drying oils.* U.S. Pat. 1,256,536. See XIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### PATENTS.

*Titanium compounds; Manufacture of pigments from —.* Det Norske Aktieselskab for Elektrokemisk Industri, Christiania, Norway. Eng. Pat. 104,885, Mar. 15, 1917. (Appl. No. 3815 of 1917.) Under Ind. Conv., Mar. 8, 1916.

TITANIUM hydrates (metatitanic or orthotitanic acid) containing undesirable impurities of an acid character, *e.g.*, basic titanium sulphate, are washed with water until neutral and then treated in the moist condition with a soluble compound of a metal, *e.g.*, barium chloride, forming a neutral insoluble sulphate not injurious to the titanic acid as a pigment, in an amount necessary to combine with the sulphuric acid of such basic sulphate. The impurity may also be removed by first treating the impure titanic acid with a base such as soda in quantity insufficient to neutralise all the sulphuric acid of the basic titanic sulphate present, removing the soluble sulphate formed, and converting the residual sulphuric acid into insoluble sulphate as above, or by using excess of base and subsequently removing the excess with acid.—A. DE W.

*Lamp black; Manufacture of —.* J. J. Shedlock, Little Bentley, Essex. Eng. Pat. 114,040, Mar. 21, 1917. (Appl. No. 4127 of 1917.)

A VEGETABLE or mineral fat or oil is heated and then discharged through a perforated pipe on to the heated bed-plate of a chamber, where the vapour is brought into immediate contact with jets of heated air, the flow of oil or fat and heated air being so regulated that on ignition of the vapours the hydrogen constituent only of the oil or fat is oxidised. Means are provided for withdrawing the liberated carbon constituent and collecting it by subjecting it to the action of sprays of any suitable liquid wherein it is absorbed, *e.g.*, of hydrocarbon oils or tar, when required to be adapted for the manufacture of carbon rods or plates, or of water, when required for use as a pigment, etc.—A. DE W.

*Zinc-lead pigment [; Process and apparatus for making —].* J. A. Singmaster and F. G. Breyer, Palmerton, Pa., Assignors to New Jersey Zinc Co., New York. U.S. Pat. 1,257,136, Feb. 19, 1918. Date of appl., Oct. 26, 1917.

A ZINC-LEAD pigment containing 65%—97% of zinc oxide and possessing a covering power, opacity, flow, and whiteness equal to that of "XX brand of American process zinc oxide" is obtained by volatilising at a high temperature the zinc and lead from an oxidised ore and subjecting the same to a regulated sulphating and oxidising atmosphere, so that the lead is converted into sulphate to the required degree without materially sulphating the zinc or unduly maintaining the fume at an elevated temperature.—A. DE W.

*Drying oils from mineral oils; Process of making —.* W. Dyer, Portland, Oreg. U.S. Pat. 1,256,535, Feb. 19, 1918. Date of appl., Nov. 7, 1916.

A MIXTURE of 10—20 parts of non-drying mineral oil, which has been freed from gasoline and kerosene, and one part of alcohol is boiled for a sufficient length of time to convert the mineral oil into a drying oil.—A. DE W.

*Drying oils; Process of making —.* W. and W. E. Dyer, Portland, Oreg. U.S. Pat. 1,256,536, Feb. 19, 1918. Date of appl., Apr. 12, 1917.

A "NON-DRYING" or "semi-drying" fatty oil is mixed with a relatively smaller bulk of alcohol, and the mixture boiled until an oil of good drying properties is obtained.—A. DE W.

*Paint; Thinned — and process of making paint-thinner [from mineral oils].* W. and W. E. Dyer, Portland, Oreg. U.S. Pat. 1,256,537, Feb. 19, 1918. Date of appl., June 7, 1917.

A PAINT thinner is obtained by boiling a mixture of about 9—19 vols. of mineral oil having a gravity about that of kerosene, and one vol. of alcohol for about half an hour. The thinner is claimed to have "drying properties," to be capable of hastening the drying of paints containing drying oils, and to possess a heavier "body" than kerosene, gasoline, or turpentine.—A. DE W.

*Varnish; Manufacture of —.* R. Weeber, Bärn, Austria. U.S. Pat. 1,257,562, Feb. 26, 1918. Date of appl., Feb. 19, 1914.

A SEMI-DRYING oil, such as train oil, is treated in a comparatively cold state with a lye to saponify the free fatty acids, the soap thus formed removed, and the oil then polymerised to a varnish with the aid of siccatives.—A. DE W.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Vulcanisation without sulphur by Ostromyslenski's method.* E. Bunschoten. Chem. Weekblad, 1918, 15, 257—268.

AN investigation of the mechanical properties of the products obtained by Ostromyslenski's method (this J., 1916, 59). The use of raw rubber and nitrobenzene or *m*-dinitrobenzene, with or without *p*-nitrosodimethylaniline or acetaldehyde-ammonia as catalyst, gives unsatisfactory results, as stated by Stevens (this J., 1917, 107). The process is much assisted by addition of lead oxide, and tables are given showing the influence of time, temperature, and proportion of materials on the mechanical properties of the products, as determined by means of Schopper's dynamometer.

—A. J. W.

#### PATENTS.

*Rubber; Process of reclaiming —.* H. R. Murdock, Naugatuck, Conn., Assignor to Rubber Regenerating Co. U.S. Pat. 1,257,292, Feb. 19, 1918. Date of appl., Oct. 8, 1915.

THE fibrous material in waste rubber is destroyed by treatment with carbohydrates other than cellulose, e.g., starch, the products of hydrolysis of which, under the conditions of devulcanisation, effect its decomposition into a brittle substance.  
E. W. L.

*Rubber compound and method of producing the same.* F. J. Gleason, Walpole, Mass., Assignor to Standard Woven Fabric Co., Framingham, Mass. U.S. Pat. 1,275,698, Feb. 26, 1918. Date of appl., Nov. 20, 1915.

COTTON and ground, vulcanised rubber are milled together until they have become thoroughly mixed, and the cotton is in a hard, flaky condition; the mixture is then ground, until the rubber is pulverised and the cotton is in a finely divided and fluffy condition, and used as an ingredient of a rubber mixing.—E. W. L.

*Rubber and the like; Machinery for [kneading] —.* S. C. Davidson, Belfast. U.S. Pat. 1,256,664, Feb. 19, 1918. Date of appl., June 7, 1915.

SEE Eng. Pats. 22,490 of 1914 and 1420, 1421, and 7168 of 1915; this J., 1916, 59.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### PATENTS.

*Tanning fish skins; Process of —.* K. Bendixen, Assignor to Skindfabriken Unicum, K. Bendixen, P. Bendixen and Co., Copenhagen. U.S. Pat. 1,256,974, Feb. 19, 1918. Date of appl. May 7, 1917.

SEE Fr. Pat. 440,092 of 1912; this J., 1912, 738.

*Hides, skins, or the like; Process for softening —.* O. Röhm, Darmstadt, Germany. U.S. Pat. 1,257,402, Feb. 26, 1918. Date of appl., Aug. 27, 1915.

SEE Ger. Pat. 288,095 of 1914; this J., 1916, 372.

*Waterproof material for use in constructional purposes, as a substitute for leather, for street paving and the like.* Eng. Pat. 111,485. See IX.

#### XVI.—SOILS; FERTILISERS.

*Soils; Water extractions of — as criteria of their crop-producing power.* J. S. Burd. J. Agric. Res., 1918, 12, 297—309.

THE author has determined the amounts of water-soluble nitrate, phosphate, and basic ions (potassium, calcium, magnesium) in good, medium, and poor soils, in the cropped and the uncropped state, and also the amounts of these constituents in a crop of barley produced on these soils. The results indicate that there is always present in soils, in a condition permitting ready solution in water, enough of the more important chemical elements to supply the immediate needs of the crop. This supply never entirely disappears in soils which are at all productive, but it would appear that a plant may be unable to satisfy its requirements if the concen-



tration with reference to the individual ions falls below certain minima. Further the optimum concentration probably varies with the soil according to the physico-chemical system present in the soil solution. There are certain characters which indicate the composition of the soil and its productivity, but these involve three variables and it is not possible to give an exact correlation between productivity and the concentrations of these three variables. To obtain a precise measure of soil fertility the soil must be studied concurrently in the cropped and the uncropped condition and under strict control.—W. G.

*Soil extract; Effect of season and crop growth in modifying the —.* G. R. Stewart. J. Agric. Res., 1918, 12, 311—368.

THIRTEEN soils, belonging to two different types, silty clay loams and fine sandy clay loams, were used and the water-soluble nutrients in them were periodically determined during two seasons. In the first season all the soils were cropped with barley, but in the second season comparisons were made between cropped and uncropped duplicates. Marked differences were observed between the contents of nitrates, calcium, magnesium, and potassium present in the water extracts from the cropped and uncropped soils, but similar differences were not shown by phosphates. The phosphate content of different soils varied considerably, but for a given soil it was practically constant in both the cropped and the uncropped plot. During the growing season the concentrations of nutrients in eight of the thirteen cropped soils were practically the same, although striking differences were obtained in the uncropped soils. The three poorest soils yielded the smallest amounts of water-soluble nutrients and the smallest differences between the cropped and uncropped duplicates. The results of the investigation indicate that large amounts of water-soluble nutrients are developed by cultivation, fallowing, and biennial cropping.—W. G.

*Soil solution; The freezing-point method as an index of variations in the — due to season and crop growth.* D. R. Hoagland. J. Agric. Res., 1918, 12, 369—395.

THE results obtained by the freezing-point method of Bouyoucos and McCool (Mich. Agr. Exp. Stat. Tech. Bull. No. 24, 1916) on the soils used by Stewart (see preceding abstract) confirm his results obtained by the extraction method as to the variation in the concentration of the soil solution. This concentration may vary greatly for the same soil under different conditions, such as season, leaching, incubation, or treatment with carbon dioxide. The growth of a crop markedly diminishes the concentration of the soil solution, this effect being still evident eight months after the crop has been removed. The results indicate that the soil solutions should not be considered as saturated, and under conditions favourable to crop growth they were found to be very dilute, particularly at the height of the growing season.—W. G.

*Carbonic acid gas in relation to soil acidity changes.* H. A. Noyes and L. Yoder. Soil Sci., 1918, 5, 151—161.

SOIL kept at one-half its water-holding capacity in a greenhouse, either with or without a crop, increased its acidity. The increase in acidity of cropped soil was modified by applications of calcium carbonate, and varied with different fertiliser applications. Carbon dioxide passed through a cropped soil, treated with lime alone or lime plus fertiliser, increased the acidity of the soil.

The experimental results indicate that soil acidity is largely the result of hydrolytic mass action phenomena.—W. G.

*Bacteria of certain Wisconsin soils; Influence of carbonates of magnesium and calcium on —.* H. L. Fulmer. J. Agric. Res., 1918, 12, 463—504.

THE three soils used were an acid and a neutral silt loam and an acid sand. In all cases the application of calcium or magnesium carbonate caused an increase in the number of bacteria in the soil, whether sterilised or unsterilised, a greater increase being obtained in the acid soils than in the neutral soil. Magnesium carbonate was more effective than calcium carbonate in this respect. Ammonification and nitrification were both benefited by the application of these carbonates, but the increase in ammonifying power was not always parallel with the increase in the total number of bacteria in the soil. The data as a whole indicate that magnesium carbonate is superior to calcium carbonate in stimulating the reproduction of bacteria in these three soils, and that, in general, smaller applications of either carbonate give better results than do the heavier applications.—W. G.

*Ammonium phosphate; Some availability studies with — and its chemical and biological effects upon the soil.* F. E. Allison. Soil Sci., 1918, 5, 1—80.

THE commercial ammonium phosphate used was manufactured by the American Cyanamid Company by the following process. Ground rock phosphate was mixed with sufficient sulphuric acid to liberate the whole of the phosphoric acid, which was filtered off. Ammonia, generated by the action of steam on calcium cyanamide under pressure, was bubbled through the phosphoric acid solution until di-ammonium phosphate was formed. Sufficient phosphoric acid was added to convert this into the mono-ammonium salt, and the solution was evaporated to dryness, giving commercial "Ammono-Phos." This material contained 13.5%  $\text{NH}_3$  and 43%  $\text{P}_2\text{O}_5$ , of which 96.5% was either water- or citrate-soluble. This was compared as a nitrogenous fertiliser with ammonium sulphate, dried blood, and cottonseed meal, and the average nitrogen recovery for six crops on Norfolk sand, ammonium phosphate coming first, was respectively 65.88, 61.10, 43.74, and 41.19%, and on a Sasafra loam 48.46, 50.42, 42.51, 36.49%. The crop yields were about in the same ratio as the nitrogen recoveries, except that under very acid conditions ammonium sulphate frequently showed a higher recovery of nitrogen but a much smaller crop than ammonium phosphate. Ammonium phosphate produced a smaller increase in soil acidity than did ammonium sulphate. There was very little difference in the availability of the phosphorus in ammonium phosphate, acid phosphate (superphosphate), or basic slag. Ammonium phosphate showed about the same toxicity to germination and early growth as ammonium sulphate and less than sodium nitrate or ammonium chloride when applied to soils in high concentrations. Of the crops examined, corn (maize), buckwheat, barley, wheat, and oats were resistant to large applications of fertilisers, while vetches, rape, and cowpeas were relatively susceptible. As a fertiliser ammonium phosphate is in general of the same value as an equivalent amount of nitrogen as ammonium sulphate and phosphorus as superphosphate. (See also J. Chem. Soc., May, 1918).—W. G.

*Ammonium sulphate; Significance of the sulphur in — applied to certain soils.* C. B. Lipman and W. F. Gericke. Soil Sci., 1918, 5, 81—86.

THE addition of sulphur, in the form of sulphuric



acid or sodium sulphate and to a lesser extent in the form of flowers of sulphur, to sodium or calcium nitrate or dried blood resulted in these fertilisers giving nearly as good a crop of barley grown on Oakley blow sand in pots, as did ammonium sulphate alone. The sulphur compounds applied without the nitrogenous fertiliser gave no increase over the control pots. (See also J. Chem. Soc., May, 1918.)—W. G.

*Ammonium sulphate; Effect of — in nutrient solution on the growth of soya beans in sand cultures* M. I. Wolkoff. Soil Sci., 1918, 5, 123—150.

AMMONIUM sulphate, when substituted for potassium nitrate in Tottingham's series of nutrient solutions (mixtures of  $\text{KH}_2\text{PO}_4$ ,  $\text{KNO}_3$ ,  $\text{Ca}(\text{NO}_3)_2$ , and  $\text{MgSO}_4$ ) in sand cultures, gave a better yield of soya beans in certain salt proportions than in the corresponding salt proportions with potassium nitrate, but caused considerable injury when added in excess. The foliage of the plants in the ammonium sulphate series had a greener colour than in the other series. There is a very close relation between the yield of tops, yield of roots, and the total transpiration of the plants. The amount of water required to produce 1 gm. of dry matter of soya bean tops was less in the ammonium sulphate series than in the other series. —W. G.

*Phosphates; Toxicity of monobasic — towards soybeans grown in soil and solution cultures* J. W. Shive. Soil Sci., 1918, 5, 87—122.

THE monobasic phosphates examined were those of potassium, sodium, ammonium, magnesium, and calcium, and the plants were grown (a) in soil cultures to which the salts were added singly in solutions having osmotic concentration values varying from 0.5 to 7 atmos; (b) in soil cultures to which the phosphates were applied in conjunction with a complete fertiliser in solutions with varying salt proportions but with total osmotic concentration values of 2.5 atmos; (c) the mixed solutions employed in the preparation of the soil cultures were also used for water cultures without alteration. The results from series (a) show that, from the point of view of yield, the growth of soya bean tops was injuriously affected by each of the five phosphates, specific injury to the plants being identical in every case and caused when the solution applied had osmotic concentration values above 1 atmos. Arranged in order of increasing toxicity the phosphates are  $\text{KH}_2\text{PO}_4$ ,  $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ ,  $\text{MgH}_2(\text{PO}_4)_2$ ,  $\text{NaH}_2\text{PO}_4$ ,  $\text{CaH}_2(\text{PO}_4)_2$ . The phosphates also caused pronounced injury when used in conjunction with a complete fertiliser, but not to a sufficient degree to kill the plants. In these cases the degree of injury was not determined solely by the total phosphate content of the medium but was also related to the relative proportions of the constituent salts. In the solution cultures the phosphates arranged in order of increasing toxicity are  $\text{NaH}_2\text{PO}_4$ ,  $(\text{NH}_4)_2\text{H}_2\text{PO}_4$ ,  $\text{KH}_2\text{PO}_4$ ,  $\text{MgH}_2(\text{PO}_4)_2$ ,  $\text{CaH}_2(\text{PO}_4)_2$ . The acid content of the solutions bears a definite relation to the specific injury sustained by the plants grown in them. The higher the acid content the greater is the degree of injury suffered by the plants. The relative salt proportions, however, play a very important rôle in either accentuating or diminishing the injury when the acidity of the solutions is sufficiently high to produce it.—W. G.

*Potash in fertiliser materials; Study of the De Roodt method for the determination of —* T. E. Keitt and H. E. Shiver. J. Ind. Eng. Chem., 1918, 10, 219—222.

In the Lindo-Gladding method (Chem. News, 1881,

44, 77, 86, 97, 129; U.S. Dept. Agric., Div. of Chem. Bull., 1885, 7, 38) the whole of the potash present in water soluble form is not accounted for and the tedious separation of the alkaline-earth bases is liable to be accompanied by occlusion of potash. The method of De Roodt (this J., 1895, 599), as now modified, is much simpler and avoids the use of platinum evaporating dishes, while the degree of accuracy is eminently satisfactory. Ten grms. of the sample is placed in a 500 c.c. flask and boiled with 300 c.c. of water for 30 mins., then diluted to the mark. Fifty c.c. of the clear solution is evaporated to dryness in a porcelain dish with 3—5 c.c. of nitric acid to destroy organic matter and the residue is treated with hot water and excess of hydrochloric acid. The solution is again evaporated to dryness and the residue treated with hot water and several drops of hydrochloric acid, and sufficient platinic chloride is added to the solution to precipitate all the potash present. The precipitate is covered with acidulated alcohol and allowed to stand for 15—20 mins., in which time all the iron, aluminium, and magnesium will dissolve; the precipitate is filtered off, washed until the filtrate is colourless, then washed well with ammonium chloride solution saturated with potassium platinochloride; this washing should be thorough, since the accuracy of the result largely depends on it. Lastly the precipitate is washed with 95% alcohol to remove the ammonium chloride, dried, and weighed. The preparation of the acidulated alcohol is as follows:—to each 1000 c.c. of 95% alcohol, 75 c.c. of concentrated hydrochloric acid is added, then dry hydrogen chloride is passed into the mixture until 1 c.c. of the alcohol neutralises 2.25 c.c. of N/1 alkali, using phenolphthalein as indicator. Potassium platinochloride is soluble in this alcohol to the extent of about one part in 60,000 at the ordinary temperature. This quick and simple method has been thoroughly tested and is much more accurate than the Lindo-Gladding method.—J. F. B.

*Water hyacinth (Eichornia crassipes): its value as a fertiliser.* R. S. Finlow and K. McLean. Agric. Res. Inst. Pusa. Bull. No. 71, 1917. 16 pages.

THE whole fresh green plant of the water hyacinth contains on the average: moisture 95.5%, organic matter 3.5% (containing N 0.04%), ash 1.0%. The average composition of the ash is  $\text{K}_2\text{O}$  28.7%,  $\text{Na}_2\text{O}$  1.8%,  $\text{CaO}$  12.8%,  $\text{Cl}$  21.0%,  $\text{P}_2\text{O}_5$  7.0%. The material if well-rotted has about the same composition as farmyard manure except that it is much richer in potash. If the fresh green plant is stacked immediately for rotting, a very serious loss of fertilising ingredients may take place in the liquid exuding from the heap. It is preferable, therefore, to dry the plant for several days before stacking it. Field tests have shown that water hyacinth either in the rotted state or as ash is a valuable manure on laterite soils of the old alluvium in Bengal.—W. G.

*Germination; Influence of acids on —* L. Maquenne and E. Demoussy. Comptes rend., 1918, 166, 547—552.

MINERAL acids, as such, are amongst the substances most injurious to germination, but their action is modified by the addition of salts, or by the presence of the salts that the acid may form by its action on the teguments of the seeds or on the glass of the containing vessel (compare this J., 1917, 932; 1918, 132 A). Calcium appears to be one of the most powerful antitoxic substances in this respect. (See also J. Chem. Soc., May, 1918.)—W. G.

*Alunite deposits of Australia and their utilisation.* See VII.



## PATENTS.

*Soil; Method of and apparatus for the treatment of —.* R. E. C. L. Guinness, London, and E. R. Richards, Harpenden. Eng. Pat. 113,860, Mar. 21, 1917. (Appl. No. 4123 of 1917.)

In a process for the heat treatment of soil to be cultivated, a predetermined thickness of the soil is thoroughly broken up and the fragments simultaneously heated to 80°–100° C. The apparatus consists of a self-propelled or other carriage provided with a flame or radiant heat producer, mechanism for rousing, agitating, stirring, or conveying the broken soil, and a motor and speed-changing device for actuating this mechanism—for example, a ploughshare mounted to deliver the soil to a rotary cutter, and thence to an agitating chamber (a rotating cylinder fitted with rakes) wherein the heating is effected.

W. E. F. P.

*Phosphatic manure; Manufacture of a —.* S. Crook and F. W. Gilbertson, Pontardawe, Glam. Eng. Pat. 113,648, Feb. 26, 1917. (Appl. No. 2819 of 1917.)

A MIXTURE of finely divided mineral phosphate with one or more alkali bisulphates, preferably nitre-cake, is made into a stiff, non-setting paste with water, then dried and ground for use.

W. E. F. P.

*Fertilising material; Process for preparing —.* T. A. Dungan, Chicago, Ill. U.S. Pat. 1,257,591, Feb. 26, 1918. Date of appl., Apr. 3, 1917.

STERILISED organic material is inoculated with aerobic bacteria, which are permitted to develop while air is forced continuously through the mass.—J. F. B.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar factories; Theory and practice in the design of multiple evaporators for —.* A. L. Webre. J. Ind. Eng. Chem., 1918, 10, 191–198.

CERTAIN points in the engineering design of the evaporators are discussed. The coefficient of heat transmission from the steam to the heating surface varies with the temperature; this is illustrated by a curve. The presence of non-condensable gases in the steam has a marked retarding effect, particularly if sufficient means are not adopted for ensuring complete agitation of the steam; moreover, under reduced pressure, any gases leaking in occupy a larger volume. Means should be provided for the proper venting of the steam belts into a large header connected to the vapour space of the last effect; accumulations of condensed water in this header must be prevented. In regard to heat transmission considered from the liquor side, the circulation of the liquor from the top of the steam drum to the bottom, as distinguished from vigorous agitation due to the escape of vapours up the tubes, has no beneficial effect. There is a hydrostatic increase of pressure due to the column of liquid, which raises the boiling point in the lower part of the apparatus, and it is detrimental to work at a higher level than is necessitated by the conditions prevailing. The accumulation of scale in the tubes is most detrimental; frequent periodical cleaning by boiling out, first with caustic soda and then with dilute hydrochloric acid, and if necessary by mechanical scrapers, is the rule. Rapid circulation impedes the incrustation of mechanically suspended matter but not of chemical deposits. The total drop of temperature between that of steam at 5 lb. pressure to vapour under 26 ins. vacuum is not equally divided between the four effects, but increases

towards the later ones. Various factors determine this rule. The coefficient of heat transmission falls from one effect to the next; the boiling point, compared with that of water under the same pressure, rises as the liquor becomes more concentrated; the effect of the hydrostatic head of liquid increases with the increase of density in the successive effects, so that the pressure indicated by the vacuum gauge for the vapour at the surface of the liquid does not represent the average pressure through the length of the tube; the increase in viscosity of the liquor also diminishes the available temperature-drop, according to imperfectly defined laws, to an extent approximately equal to that due to rise of boiling point. Combining the temperature losses due to boiling point, static head, and viscosity with the normal working drop determined by the available coefficient of heat transmission, the total temperature difference of 102° F. (57° C.) is divided between the four stages in the proportions of 15°, 18°, 23°, and 46° F. (8°, 10°, 13°, and 26° C.) respectively. A table is given showing the destination and amount of heat entering and leaving each stage and the amount of water evaporated for a given set of conditions. From this table the distribution of heating surface, at an average evaporation of 5½ lb. per sq. ft. per hour, is calculated, and it is found that in standard practice the second, third, and fourth vessels may be equally dimensioned, according to their mean requirements, and the first may be given double the heating surface of the others. The steam pipes, exhaust pipes, and vapour pipes should be dimensioned for a velocity of 100 ft. per sec., except in the case of the last effect, where, on account of the low density of the steam, 200 ft. per sec. is permissible. Loss by frothing is very rare; the best means for preventing it is to maintain a high vacuum; grease or oil is sometimes used. Loss by spraying is often serious; the mean height of projection of particles of liquid can be calculated from the volume and velocity of the vapour generated. Thus it is found that spraying may be very serious in the fourth vessel and perhaps also in the third. Sudden generation of vapour up the tubes should be avoided by distributing the admission of liquor over a wide area by a perforated coil or other device. Baffle-plates arranged in the vapour space above the liquor are also advantageous.

—J. F. B.

*Ash of cane and beet sugar products; Determination of the —.* J. P. Ogilvie and J. H. Lindfield. Intern. Sugar J., 1918, 20, 114–117.

SCHWEIZER (Archief Suikerind. Ned.-Indie, 1916, 24, 214–216) observed that the customary deduction of 10% for the conversion of the sulphated to the carbonated or so-called "true ash" is far from correct in the case of Java molasses. The authors corroborate this and give the results of determinations made with a number of cane and beet sugars and molasses from different countries. The sulphated ash was found in the usual manner, using 6 grms. of the sample and 1.5 c.c. of concentrated sulphuric acid; while the carbonated ash was ascertained by the method of carbonising below red heat, dissolving out the alkali salts, and incinerating the carbonised mass, the ash obtained and the residue from the evaporation of the solution of the alkali salts being heated to a low temperature after the addition of a little ammonium carbonate. Considerable differences in the correction necessary for converting the sulphated ash to the carbonated ash were found, even in products of the same class. In the case of beet sugars it varied from 12 to 25 but was never as low as 10%; in that of beet molasses from 12 to 18; and in that of cane molasses from 14 to 21; but with cane sugars the values were very irregular, fluctuating



between 6 and 26%. Varying factors are to be expected with cane sugars, owing to the presence in them of greater or less amounts of insoluble inorganic matter, which is partly sand, and partly salts thrown out of solution during evaporation and boiling and retained during curing in the centrifugals. Beet sugars, on the other hand, and beet and cane molasses contain a smaller and more constant quantity of insoluble ash. From the point of view of the refiner it is only the ash of the soluble matter that is of importance, and if this constituent were determined, in place of the total ash, a more exact indication of the value of refining sugars would be obtained. Determinations were made of the ash in sugars also by direct incineration followed by carbonating, as still practised by some chemists, but this procedure, owing evidently to the volatilisation of alkali salts, was found to yield appreciably lower results than either of the other two methods used. —J. P. O.

*Molasses; Notes on the analysis of* —. H. S. Walker. *J. Ind. Eng. Chem.*, 1918, 10, 198—202.

DISCREPANCIES in the Clerget sucrose determinations in waste molasses have been investigated. It would appear that the method of clarification with basic lead acetate solution gives results which are too high by 0.5—0.7% of sucrose, on account of the large volume occupied by the lead precipitate. Clarification with dry basic lead acetate, after the solution has been made up to a measured volume, gives figures which are much closer to the true sucrose value but which are apt to be slightly low on account of dilution, especially if an excess of lead be used in clarifying. Artificial mixtures have been made up with known quantities of pure sucrose, invert sugar, and the product obtained from waste molasses after precipitating with lead, washing, and decomposing the lead precipitate by hydrogen sulphide. With these reconstructed molasses of known sucrose content, a method has been devised to avoid the error due to dilution in the dry basic lead acetate method. Twice the normal weight of molasses (52 grms.) is dissolved in water and made up to 300 c.c. The solution is clarified with 15—20 grms. of dry basic lead acetate and a few grms. of dry sand, and filtered. To 75 c.c. of the filtrate in a 100 c.c. flask, 20 c.c. of a solution containing 100 grms. of phosphoric acid per litre is added, and the mixture is made up to 100 c.c. If necessary, about 0.5 gm. of zinc dust may be added immediately before filtration to improve the colour. The direct polarisation is taken in a 400 mm. tube. Another portion of 75 c.c. of the original filtrate is treated with 2 c.c. of dilute hydrochloric acid (1 vol. of concentrated acid to 1 vol. of water) to neutralise the alkalinity, the liquid is heated to 65°—70° C., and 10 c.c. of the dilute hydrochloric acid is added. After 15 mins. the liquid is cooled, made up to 100 c.c., treated with zinc dust, and filtered. The divisor 142.1—0.54 is used for the Clerget formula. By this method the error due to the volume of the lead phosphate precipitate compensates for the error of dilution caused by any excess of lead originally dissolved. The amount of phosphoric acid prescribed is the largest which can be used without danger of inversion; its effect on the rotation of invert sugar is not quite equal to that of the hydrochloric acid in the Clerget solution, but the difference is negligible. —J. F. B.

#### PATENTS.

*Sugar; Process of refining* —. R. R. Williams, Manila, Philippine Islands. U.S. Pat. 1,256,758, Feb. 19, 1918. Date of appl., Jan. 26, 1914. Renewed Aug. 29, 1917.

A SOLUTION of sugar is subjected to the electro-

lytic action of a direct current of electricity, whereby the impurities which are susceptible to electrolytic action are caused to pass through porous partitions into adjacent compartments, in which the electrodes are immersed in a stream of fresh water which carries the impurities away.—J. F. B.

*Molasses; Recovery of sugar from* —. F. Tiemann, Berlin. U.S. Pat. 1,256,952, Feb. 19, 1918. Date of appl., June 25, 1911.

SEE Ger. Pats. 279,719, 279,720, and 288,411; this J., 1915, 371; 1916, 375.

#### XVIII.—FERMENTATION INDUSTRIES.

*Worts and beers; Physico-chemical investigations of* —. W. Wöllmer. *Z. ges. Brauw.*, 1917, 40, 387.

DETERMINATIONS of hydron concentration in worts of low specific gravity used for the preparation of war-beers, gave values much lower than those for worts of normal gravity correspondingly diluted with distilled water. In thin brewery worts the acid-reacting malt constituents are not only abnormally diluted but also largely neutralised owing to the relative preponderance of the salts of the brewing water. The low hydron concentration of thin worts must affect the transformation of hop-resins (cp. this J., 1918, 218 A) and the activity of the yeast. The deficiency is not made good during fermentation, for the hydron concentration of fermented war-beer was found to be abnormally low.—J. H. L.

*Wort and beer; Bacteria [B. termo] in* —. P. Petit. *Brasserie et Malterie*, 1917, 7, 273—277.

THE author describes the effects of infection of worts by the organisms known collectively as *Bacterium termo*. Such infection very commonly occurs, especially in hot weather, as the result of contact of the wort with infected utensils or germ-laden air. Although not usually considered very dangerous, since they are suppressed during fermentation and therefore not found in the finished beer, these organisms may give rise to defects more or less serious. The best known of these is the bad odour which they produce in worts and which may even persist in the beer; but if present in large numbers they may weaken the yeast and thus retard fermentation, and give rise to difficulties of filtration and clarification in beers predisposed to these defects. It is probable that many cases of infection and haze in beers are indirectly due to infection of the wort by *B. termo*, which although completely eliminated during fermentation, renders the beer abnormally subject to other disorders. Serious infection of wort with *B. termo* is most easily detected during the later stages of the primary fermentation, the organisms being then dead but still in suspension, sometimes in sufficient numbers to cause opalescence. Practical measures for tracing the infection to its source are discussed.—J. H. L.

#### PATENTS.

*Yeast; Process for pressing — and apparatus for use therein*. E. A. Meyer, Bristol. Eng. Pat. 113,628. (Appl. Nos. 17,338, Dec. 2, 1916, and 8804, June 19, 1917.)

THE yeast is deposited on the surface of a drum revolving in a tank containing the liquid suspension of yeast; the air is exhausted from the drum and a knife is adjusted so that it skims off only the dry outer layer of the deposited yeast, leaving the lower moist layer on the drum to prevent the breaking of the vacuum. This effect is also assisted by providing, below the knife and in close



proximity to it, a perforated pipe through which a liquid containing yeast is constantly sprayed over the surface of the drum.—J. F. B.

*Minim-alcoholic beverages: Method of preparing* —. E. Gudeman, Chicago, Ill. U.S. Pat. 1,256,894, Feb. 19, 1918. Date of appl., June 22, 1908.

THE beer charge is subjected in an open vessel to the action of free steam in copious and regulated quantity to agitate the charge and heat it to somewhat below the boiling point, the supply of steam being such that the major portion of it is forced through the charge to drive off the alcohol, while a small portion condenses to maintain the volume of the charge practically constant.—J. F. B.

*Extract having the flavour and aroma of malt, process of producing same, and beverage therefrom.* M. and L. Wallerstein, New York. Eng. Pat. 107,367, Jan. 17, 1917. (Appl. No. 882 of 1917.) Under Int. Conv., June 21, 1916.

SEE U.S. Pats. 1,214,729 and 1,214,730 of 1917; this J., 1917, 400.

## XIXA.—FOODS.

### PATENTS.

*Food products [potatoes]: Method of preparing pre-cooked* —. Condensed Food Co., Philadelphia, Pa., U.S.A. Eng. Pat. 113,900, June 28, 1917. (Appl. No. 9196 of 1917.) Under Int. Conv., Apr. 21, 1917.

PEELED potatoes are cooked for 20 to 40 minutes by steam at 220° to 230° F. (104° to 110° C.) and about 15 lb. pressure, then cut into thin shreds ("riced"), which are dried while still hot in a current of air at about 180° F. (82° C.). This drying operation requires from 25 mins. to 2 hours. The product keeps indefinitely in cardboard packages. The process may also be applied to sweet potatoes, carrots, and other vegetables.—W. P. S.

*Food; Means for preserving* —. L. L. Kahn, San Francisco, Cal. U.S. Pat. 1,256,495, Feb. 12, 1918. Date of appl., Aug. 18, 1917.

A TIN for containing preserved foods is provided with a celluloid lining which completely encloses the food and prevents the latter from coming into contact with the metal.—W. P. S.

*Drying apparatus [for fruit].* G. D. Parker, Riverside, Cal. U.S. Pat. 1,256,825, Feb. 19, 1918. Date of appl., June 27, 1917.

THE dryer comprises a housing open at the bottom, through which air is forced in a downward direction. Inside the housing is a series of superposed horizontal endless conveyors. The fruit is introduced at the top and is carried by the conveyors from one end of the housing to the other alternately in opposite directions in successively lower horizontal planes.—J. H. P.

## XIXB.—WATER PURIFICATION; SANITATION.

### PATENTS.

*Suspended matter; Means for maintaining — in liquids in a state of suspension without precipitation [e.g., in water softening].* A. Pendlebury, Bradford. Eng. Pat. 114,016, Mar. 13, 1917. (Appl. No. 3645 of 1917.)

AN insoluble substance, such as lime, is mixed with water to a required density by means of

agitators in two mixing tanks placed side by side on the ground level. To the tanks is fitted a common suction pipe with a valve to each tank, so that one tank can be filled whilst the other is being emptied. This pipe is connected to a centrifugal pump capable of delivering about double the quantity of liquid required. The liquid is delivered into a tank at a higher level from which measured quantities are taken for addition to the water or other liquid to be treated. The upper tank has a semi-circular bottom and the pressure of the pump keeps the mixture in motion and prevents any settling. Surplus liquid overflows and falls back into the mixing tanks.—J. H. P.

*Filter-body [for removing germs from liquids].* G. H. Seitz and F. Schmittthener, Assignors to Seitz-Werke T. und G. Seitz, Kreuznach, Germany. U.S. Pat. 1,256,171, Feb. 12, 1918. Date of appl., Mar. 10, 1917.

A PASTE consisting of infusorial earth, flax, cotton, and water containing formaldehyde is dried, formed into a suitable shape, and sterilised.—W. P. S.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Storax; Examination of* —. L. van Itallie and H. J. Lemkes. Pharm. Weekblad, 1918, 55, 142—149.

THE determination of the cinnamic acid content is preferred to the acid or saponification values; the acid occurs in storax in the free state and as the cinnamyl, phenylpropyl, ethyl, and ricinoleic esters. The results of the examination of nine samples of storax are given. The free cinnamic acid cannot be determined from the acid value, which includes acids insoluble in water; moreover, if hydrolysis occurs, a high acid value results. Titration of the aqueous extract with alkali gives a much lower content of cinnamic acid than the following bromine method, which is recommended by the authors. 1 gm. of the sample is boiled with 20 c.c. of N/2 alcoholic potassium hydroxide for 1 hour under a reflux condenser, the mixture distilled on a water-bath, and the residue dissolved in 25 c.c. of water. The solution is extracted with 20 c.c. of ether, and the aqueous layer, together with two 5 c.c. portions of water used for washing the ether layer, diluted to about 950 c.c., treated with 10 c.c. of dilute sulphuric acid, made up to 1000 c.c., and filtered. 100 c.c. of the filtrate is treated with 10 c.c. of N/10 potassium bromate, 1 gm. of potassium bromide, and 5 c.c. of sulphuric acid; after 15 mins., 1 gm. of potassium iodide is added, and then, after a further 5 mins., the liberated iodine is titrated with thio-sulphate; 1 c.c. of N/10 KBrO<sub>3</sub> is equivalent to 7.4 mgrms. of cinnamic acid.—F. W. A.

### PATENT.

*Process of effecting organic chemical reactions.* Eng. Pat. 110,547. See I.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Laws of thermochemical processes and of photochemical processes.* Trautz. See I.

*Blue and brown print paper: characteristics, tests, and specifications.* Veitch and others. See V.

## XXII.—EXPLOSIVES; MATCHES.

### PATENT.

*Processes for making silicic acid.* Eng. Pat. 113,769. See VII.

## XXIII.—ANALYSIS.

*Analysis; A new process of quantitative —.*  
A. B. P. Leime. *Comptes rend.*, 1918, 166,  
465—467.

A DESCRIPTION of a spectrographic method for  
quantitative analysis. (See also *J. Chem. Soc.*,  
May, 1918.)—W. G.

*Boron [e.g., in steel and minerals]; Spectroscopic  
detection of —.* A. de Gramont. *Comptes  
rend.*, 1918, 166, 477—480.

USING the condensed spark, boron may be readily  
detected in steels, when present in small amounts,  
by the ray  $\lambda$  3151.2 and the doublet  $\lambda$  2497.82 and  
2496.87. The same method may be employed  
for its detection in minerals after previous fusion  
with alkali carbonates. (See also *J. Chem. Soc.*,  
May, 1918.)—W. G.

*Calorimeter design; Thermal leakage and —.*  
W. P. White. *J. Amer. Chem. Soc.*, 1918, 40,  
379—393.

THE influence of the air space round the calorimeter  
on the thermal leakage is considered with par-  
ticular reference to the width of the air gap, the  
size of the calorimeter, and the use of adiabatic  
methods. The rate of heat interchange is approxi-  
mately proportional to the difference of tempera-  
ture between the calorimeter and its environment,  
except in so far as such interchange is caused by  
evaporation or convection. For ordinary calori-  
metric conditions the effect of convection is  
more nearly proportional to the square of the  
temperature difference. The convection effect  
diminishes rapidly as the width of the surrounding  
air gap decreases, and this reduction can be advan-  
tageously carried so far that the total thermal  
leakage is mainly due to conduction, which in-  
creases approximately in inverse proportion to the  
width of the air space. Calculation shows that  
gaps of 10 to 17 mm. are most suitable for ordinary  
calorimeters. In the case of larger calorimeters,  
the width of the air space may be increased beyond  
these limits without detriment to the influence of  
convection on the thermal leakage. Conduction  
may be reduced by the use of very thin reflecting  
shields, the use of which does not increase the  
convection effect. Evaporation must be reduced  
as far as possible by suitable design of the calori-  
meter, and attention must be given to thermometer  
lag in correcting the actual readings. (See also  
*J. Chem. Soc.*, May, 1918.)—H. M. D.

*Determination of moisture in coke.* Fieldner and  
Selvig. See IIA.

*Testing natural gas for gasoline.* Oberfell. See IIA.

*Limits of accuracy of Holleman's method of deter-  
mining the composition of a mixture of two or  
three isomers. [Application to the nitranilines.]*  
Nichols. See III.

*Distinguishing manila from all other "hard" rope  
fibres.* Swett. See V.

*Valuation of lime for various purposes.* Meade.  
See VII.

*Analysis of spent oxide [from gas works].* See VII.

*Action of sodium sulphide on iodine and the use of  
the reaction in analysis.* Ehrlich. See VII.

*Detection of small quantities of chlorine in iodine.*  
Pinkhof. See VII.

*Estimation of lanthanum in its alloys with iron.*  
Travers. See X.

*Platinum substitute [platinum-gold alloy].* Van der  
Marck. See X.

*Modified sesame oil reaction.* Weehuizen. See XII.

*Estimation of glycerol by Wagenaar's method.*  
Beckers and Kolthoff. See XII.

*Estimation of the amount of water in glycerin.*  
Kolthoff. See XII.

*Study of the De Roode method for the determination of  
polash in fertiliser materials.* Keitt and Shiver.  
See XVI.

*Determination of the ash of cane and beet sugar  
products.* Ogilvie and Lindfield. See XVII.

*Notes on the analysis of molasses.* Walker.  
See XVII.

*Examination of storax.* Van Itallie and Lemkes.  
See XX.

## PATENT.

*Thermostats; Construction of —.* A. Lundie,  
Farnham. Eng. Pat. 114,231, Apr. 27, 1917.  
(Appl. No. 5983 of 1917.)

## Patent List.

The dates given in this list are, in the case of Applications for  
Patents, those of application, and in the case of Complete Specifi-  
cations accepted, those of the Official Journals in which the  
acceptance is announced. Complete Specifications thus advertised  
as accepted are open to inspection at the Patent Office immediately,  
and to opposition within two months of the date given.

## I.—GENERAL; PLANT; MACHINERY.

## APPLICATIONS.

Adt Co. Drying apparatus. 6357. Apr. 15.  
(U.S., July 17, 1917.)

Alexander. Apparatus or kilns for drying granular,  
pulverulent, and pasty materials. 6612, 6613, and  
6614. Apr. 18.

Bowman. Evaporating apparatus. 6420.  
Apr. 16.

Dent and United Alkali Co. 6598. See II.

Hardinge. 7059. See X.

Marks (Soc. Anon. Ital. Ansaldo & Co.). 6361.  
See II.

Morison. Condensing plant. 6338. Apr. 15.

Müller. Manufacture of colloidal solutions.  
6791. Apr. 22. (Ger., Mar. 3, 1917.)

Newell and Co., and Woodhouse. Grinding-  
mills. 6960. Apr. 25.

Roggen and Scheffers. Kilns. 6444. Apr. 16.  
(Fr., Apr. 26, 1917.)

Smallwood. Furnaces. 6804 and 6805. Apr. 23.

Soc. l'Air Liquide. Liberation of a gas from  
mixtures containing it. 7082. Apr. 26. (Fr.,  
Nov. 17, 1917.)

Sturgeon. Centrifugal separators. 6408. Apr. 16.

Sulman. Stillheads or dephlegmators. 6674.  
Apr. 19.

## COMPLETE SPECIFICATIONS ACCEPTED.

16,372 (1916). Burchenal. Filters. (104,673.)  
May 1.

5432 (1917). Fay. Anti-priming devices or  
baffle plates for evaporators, etc. (114,888.) May 1.

6093 (1917). Gaunt and others. See X.

6913 (1917). Carroll. Separating liquids of  
different specific gravity insoluble or difficultly  
soluble in each other. (114,921.) May 1.

1025 (1918). Davidson and Holmes and Co.  
Apparatus for bringing liquids and gases, vapours,  
fumes, and the like into intimate contact. (115,006.)  
May 1.

2888 (1918). Sadezky. Production of a drying  
medium. (114,810.) Apr. 24.



## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### APPLICATIONS.

Anderson. Production of oil fuel. 6499. Apr. 17.  
 Bancroft and Hansford. Retorts, and methods of working them. 7131. Apr. 27.  
 Barrow and Scott. Suction-gas producers. 6523. Apr. 17.  
 Boake, Roberts, and Co., and others. 7039. See XX.  
 Dent, and United Alkali Co. Apparatus for distributing liquids in gas-scrubbers, etc. 6598. Apr. 18.  
 Doyle. Raising and drying peat. 6801. Apr. 23.  
 Edmonds. Compounds for treating fuel. 6925. Apr. 24.  
 Harger and Helps. 6752. See III.  
 Langwell, and Power-Gas Corporation. Treatment of peat. 6777. Apr. 22.  
 Marks (Cleveland Trust Co.). Distilling petroleum, etc. 6921. Apr. 24.  
 Marks (Soc. Anon. Ital. Ansaldo & Co.). Production of gases from water or oil. 6361. Apr. 15.  
 Parsons. Treatment of carbon and its combinations with other elements. 6833. Apr. 23.  
 Taikoo Sugar Refining Co. (Obrembski and Resker). Production of decolorising-carbon. 6427. Apr. 16.  
 Wellington. Distillation of coal, etc. 6731. Apr. 20.  
 Wilton. 6375. See III.  
 Wood. Drying fuel supplied to furnaces, etc. 6779. Apr. 22.

### COMPLETE SPECIFICATIONS ACCEPTED.

54 (1917). Oltmans. Obtaining lower from higher carburets of hydrogen. (108,451.) May 1.  
 1534 (1917). Drakes, Ltd., and Drake. Regenerative retort settings. (114,859.) May 1.  
 5096 (1917). Recovery of sulphate of ammonia from producer gases. (114,658.) Apr. 24.  
 8269 (1917). Porteous and Allen. Coke ovens of the horizontal or approximately-horizontal flue type. (114,936.) May 1.  
 8337 (1917). Perkin and West. Destructive distillation of carbonaceous materials. (114,937.) May 1.  
 13,694 (1917). Yeadon and Whitaker. System of carbonising coal. (114,971.) May 1.

## III.—TAR AND TAR PRODUCTS.

### APPLICATIONS.

Anderson. Process of obtaining oil from tar or pitch. 7026. Apr. 26.  
 Archer. Tar stills. 6667. Apr. 19.  
 Harger and Helps. Manufacture of liquid fuel from dehydrated tar. 6752. Apr. 22.  
 Klein. Dehydration of tar. 7032. Apr. 26.  
 Wilton. Treatment of tar for improving its value for fuel purposes and for separation of products. 6375. Apr. 15.

### COMPLETE SPECIFICATION ACCEPTED.

3609 (1917). Ransford (Cassella und Co.). Manufacture of chlorinated products of xylene and new compounds and dyestuffs therefrom. (114,645.) Apr. 24.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATIONS.

Morton, Thomson, and Morton Sundom Fabrics, Ltd. Manufacture of anthracene derivative colouring matters. 6916 and 6917. Apr. 24.

Morton, Dandridge, and Morton Sundom Fabrics, Ltd. Production of colouring matters. 7009. Apr. 25.

### COMPLETE SPECIFICATION ACCEPTED.

3609 (1917). Ransford (Cassella und Co.). See III

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### APPLICATIONS.

Borzykowshi. Production of articles from viscose. 6346. Apr. 15. (U.S., May 29, 1917.)  
 Gallon and Johnston. Pulp-making. 6528. Apr. 17.  
 Orrell. Manufacture of paper, etc. 6473. Apr. 17.  
 Robbins. 7111. See XII.  
 Sutherland and others. 6909. See XIII.  
 Ziman. Treatment of kapok fibre to render it antiseptic and fireproof. 6969. Apr. 25.

### COMPLETE SPECIFICATION ACCEPTED.

9666 (1917). Heberlein und Co. Process for imparting new properties to cotton fabrics. (108,671.) Apr. 24.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### APPLICATION.

Lord and Lord. Machines for dyeing, washing, scouring, sizing, bleaching, and mercerising yarn in hank form. 7109. Apr. 27.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATION.

Ashcroft. Manufacture of nitrogen with or without other constituents. 6382. Apr. 15.  
 Ashcroft. Manufacture of nitrogen and its compounds, removal of oxygen from mixtures of gases, and obtaining useful products by oxidation and deoxidation. 6383. Apr. 15.  
 Ashcroft. Manufacture of oxides or salts of tungsten or of metallic tungsten. 6456. Apr. 17.  
 Ashcroft. Manufacture of oxides or salts of metals, or of metals, or of non-metals, from ores, minerals, residues, etc. 6457. Apr. 17.  
 Ashcroft. Manufacture of aluminium or its oxides or salts. 6548. Apr. 17.  
 Ashcroft. Manufacture of silicon chloride and removal of silica from fused melts. 6459. Apr. 17.  
 Ashcroft. Heat treatment of potash rocks with salts for extraction of potash. 6669. Apr. 19.  
 Crosland. 6874. See IX.  
 Fisher Chemical Engineering Co., and others. 6597. See XII.  
 Langwell, and Power-Gas Corporation. Manufacture of salts of fatty acids, particularly acetates. 6778. Apr. 22.  
 Norsk Hydro-Elektrisk Kvaestofaktieselskab. Manufacture of nitric acid. 6769. Apr. 22. (Norway, May 8, 1917.)  
 Pease. Manufacture of alkaline cyanide and of ammonia therefrom. 6450. Apr. 16.  
 Ridoni, and Soc. Talcio e Grafitti Val Chisone. Manufacture of hydrofluoric acid. 6525. Apr. 17. (Ital., Apr. 19, 1917.)  
 Ridoni, and Soc. Talcio e Grafitti Val Chisone. Purification of natural graphites. 6837. Apr. 23. (Ital., May 5, 1917.)

### COMPLETE SPECIFICATIONS ACCEPTED.

5096 (1917). Mellor. See II.  
 5125 (1917). Maxted and Smith. Manufacture of nitrogen and of mixtures of nitrogen and hydrogen. (114,663.) Apr. 24.

5559 (1917). Armstrong. Powders for generating caustic alkalis. (114,896.) May 1.  
 6596 (1917). Comment. Preparation of anhydrous zinc sulphide. (114,407.) Apr. 24.  
 7737 (1917). Ilayhurst. Condensers for acids. (114,706.) Apr. 24.  
 8224 (1917). Gladysz. Manufacture of mixtures of sodium and potassium bitartrates. (107,020.) Apr. 24.  
 9171 (1917). Kaltenbach. Sulphurous anhydride. (107,589.) Apr. 24.  
 10,435 (1917). Ashcroft. Removal of soda from felspars or natural or artificial silicates or aluminates and manufacture of caustic potash. (114,743.) Apr. 24.  
 11,535 (1917). Bloxam (Zellstoffabrik Waldhof). *See X.*  
 13,721 (1917). Aktiebolaget Carlit. Manufacture of ammonium perchlorate. (110,544.) Apr. 24.  
 13,883 (1917). Statham. *See XI.*

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Haddan (Corning Glass Works). Manufacture of glass for absorbing ultra-violet radiations. 6398 and 6399. Apr. 15.  
 Hamblet. Admixtures for refractory clays. 6568. Apr. 18.  
 Ionides. Pottery-burning, etc. 6663. Apr. 19.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5441 (1917). Longstaff and Geairns. Manufacture of briquettes, silica, plastic, gainster, common fire, and like bricks. (114,676.) Apr. 24.  
 13,963 (1917). Elektro-Osmose A.-G. Manufacture of ceramic and like ware. (113,777.) Apr. 24.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Crosland. Kiln or furnace for calcining limestone, cement, etc. 6874. Apr. 24.  
 Ramsden. Acid-proof rock asphalt. 7101. Apr. 27.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3317 (1917). Linden. Concrete or cement substances. (114,641.) Apr. 24.  
 5420 (1917). Jolnerkevitch. Impregnation of wood, etc. (114,886.) May 1.  
 5441 (1917). Longstaff and Geairns. *See VIII.*  
 18,380 (1917). Deckers. Manufacture of cement from blast furnace slag. (114,794.) Apr. 24.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Ashcroft. Manufacture of chemically-reactive alloys. 6381 and 6967. Apr. 15 and 25.  
 Ashcroft. 6546, 6547, and 6548. *See VII.*  
 Brazil, Straker, and Co., and Shepherd. Electrodeposition of iron. 7074. Apr. 26.  
 Crocker and Crocker. Increasing production of zinc dust from spelter retorts. 7025. Apr. 26.  
 Elmore. Extraction of lead and zinc from their ores. 6668. Apr. 19.  
 Harding. Conical mills for grinding ores, etc. 7059. Apr. 26.  
 Industrial Inventions, Ltd., and Perry. Production of malleable iron castings. 7049. Apr. 26.  
 Procter. Aluminium solder. 7128. Apr. 27.  
 Prosser. Ore or metal concentrators. 6751. Apr. 22.

Quinan. Production of metals by reduction of oxides. 6832. Apr. 23.

Sumpter, and Vickers, Ltd. Surface-hardening steel plates, etc., and iron and malleable iron castings. 6537. Apr. 17.

Wade (Blaw-Knox Co.). Open-hearth furnaces, etc. 7000. Apr. 25.

#### COMPLETE SPECIFICATIONS ACCEPTED.

6093 (1917). Gaunt, Brookfield, and Tylor and Sons. Crucible and like furnaces. (114,684.) Apr. 24.  
 11,464 (1917). Harbord. Furnaces for roasting ores. (114,953.) May 1.  
 11,498 (1917). Jones. Production of spelter. (112,923.) Apr. 24.  
 11,523 (1917). Metals Disintegrating Co. Process for reducing metals to a finely-divided state. (109,258.) Apr. 24.  
 11,535 (1917). Bloxam (Zellstoffabrik Waldhof). Roasting pyrites, ores, etc., in shelf-burners. (114,954.) May 1.  
 14,286 (1917). Lockwood. Electrodeposition of base metals. (114,976.) May 1.  
 18,380 (1917). Deckers. *See IX.*

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Berry. Working of electric furnaces, and electrode used therein. 6678. Apr. 19.  
 Bosch A.-G. Electrodes for spark discharges. 6459. Apr. 16. (Ger. Apr. 16, 1917.)  
 Brazil, Straker, and Co., and Shepherd. 7074. *See X.*  
 Etchells, Greaves, and Watson and Co. Electric furnaces. 6563. Apr. 18.  
 Lawton. Electric furnaces. 6660 and 6661. Apr. 19.  
 Leitner. Electric cells. 6432. Apr. 16.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5442 (1916). Foley. Electric induction furnaces. (114,853.) May 1.  
 13,883 (1917). Statham. Electrolytic cells especially for electrolysis of salt or similar material. (114,974.) May 1.  
 14,286 (1917). Lockwood. *See X.*  
 14,932 (1917). Dixon. Electric furnaces. (111,103.) Apr. 24.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Co-operative Wholesale Society, and Martin. Manufacture of cocoa butter. 6760. Apr. 22.  
 Fisher Chemical Engineering Co., Hinchley, and Simon. Apparatus for extracting oils, wax, india-rubber, sulphur, etc., soluble in organic solvents. 6597. Apr. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5242 and 5243 (1917). Young. Apparatus and method for rapidly ageing soap. (114,878 and 114,879.) May 1.  
 17,683 (1917). Johnson. Separation of fats, oils, or the like from liquids. (114,789.) Apr. 24.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Imray (Soc. Chem. Industry in Basle). Manufacture of weatherproof cement for paints. 7069. Apr. 26.  
 Robbins. Preparation of dope for treating fabrics for aircraft. 7111. Apr. 27.  
 Sutherland, and Wall Paper Manufacturers Ltd. Varnishes or dopes for fabrics of aeroplanes, etc. 6909. Apr. 24.



## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

- Banbury. Machine for treating rubber, etc. 6564. Apr. 18. (U.S., Apr. 20, 1917.)  
 Fisher Chemical Engineering Co., and others. 6597. *See* XII.  
 Gorell. Application of rubber coatings to articles. 6665. Apr. 19.

## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

- Marshall. Production of cake or powder glue from meat and/or slaughterhouse offal. 6800. Apr. 23.  
 Miller. Leather substitute for boots, etc. 6393. Apr. 15.  
 Wright. Tanning machinery. 6686. Apr. 20.

## COMPLETE SPECIFICATION ACCEPTED.

- 1665 (1918). Vulcan Engineering Co., and Dickson. Machine for facilitating the handling and treatment of hides, skins, etc., in tanning and like operations. (115,009.) May 1.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

- Marks (Soc. Anon. Ital. Ansaldo & Co.). Production of fertilisers. 6445. Apr. 16.  
 Marks (Soc. Anon. Ital. Ansaldo & Co.). Production of superphosphate minerals. 7058. Apr. 26.

## COMPLETE SPECIFICATION ACCEPTED.

- 5062 (1917). Howorth (Norsk Hydro-Elektrisk Kvaelfstofaktieselskab. Manufacture of a phosphate fertiliser containing nitrogen. (114,873.) May 1.

## XVII.—SUGARS; STARCHES; GUMS.

## APPLICATION.

- Taikoo Sugar Refining Co. 6427. *See* II.

## COMPLETE SPECIFICATION ACCEPTED.

- 7175 (1917). Hayashi, Emura, and Kwanto Sansho Kabushiki-Kaisha. Material for clarifying sugar solutions and means for regenerating the material after use. (108,303.) May 1.

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATION.

- Desborough and others. 6729. *See* XX.

## COMPLETE SPECIFICATION ACCEPTED.

- 14,041 (1914). Barbet et Fils et Cie. Rectification of fermented musts, methyl alcohol, and other liquids. Apr. 24.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

- Co-operative Wholesale Society, and Martin. 6760. *See* XII.  
 Croft. Beverage. 6404. Apr. 16.

- Graham. Bread, and process of producing same. 6971. Apr. 25.

- Grousseau and Viconge. Treatment of cocoa and waste products of chocolate manufacture. 7094. Apr. 27. (Fr., June 11, 1917.)

- Jones, and Jones and Attwood. Purification of sewage, etc. 7104. Apr. 27.

- Kemp and Spencer. Dried eggs in powder form. 6502. Apr. 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 7275 (1917). Stamp. Apparatus for drying and deodorising nightsoil, slaughterhouse refuse, etc. (114,700.) Apr. 24.

- 7583 (1917). Giraud. Manufacture of aerated water. (108,852.) May 1.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

- Belart. Continuous process of manufacturing chloroform. 7106. Apr. 27.

- Blanc. Manufacture of aromatic aldehydes. 7119. Apr. 27. (Fr., Apr. 27, 1917.)

- Boake, Roberts, and Co., and Durrans. Production of alkylating agents. 6583. Apr. 18.

- Boake, Roberts and Co., Durrans, and Perkin. Manufacture of ethylene. 7039. Apr. 26.

- Boake, Roberts and Co., and Durrans. Manufacture of esters. 7040. Apr. 26.

- Crosfield and Sons, and Hilditch. Manufacture of acetaldehyde. 6980. Apr. 25.

- Desborough, Reilly, and Thaysen. Manufacture of lactic acid and its salts, etc. 6729. Apr. 20.

- National Federation of Associated Paint, Colour, and Varnish Manufacturers of the United Kingdom, and others. Production of organic compounds, etc. 6734. Apr. 20.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 14,041 (1914). Barbet et Fils et Cie. *See* XVIII.

- 5669 (1917). Newton (Bayer und Co.). Manufacture of pharmaceutical products. (114,680.) Apr. 24.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

- Agnew, Renwick, and Ilford, Ltd. High-temperature development of photographic materials. 6723. Apr. 20.

- Eagle. Kinematograph films. 6959. Apr. 25.

- Smith. Production of screens for half-tone and like photographic printing-surfaces. 6435. Apr. 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 4429 (1917). Wade. Kinematograph films. (114,871.) May 1.

- 7961 (1917). Crosland and Crosland. Preparation of sensitised paper or like flexible material for use in obtaining direct X-ray photographs or radiograms. (114,933.) May 1.

## I.—GENERAL ; PLANT ; MACHINERY.

*Evaporation and drying ; Some general aspects of* — H. K. Moore, Amer. Inst. Chem. Eng., Dec. 7, 1917. Met. and Chem. Eng., 1918, 18, 128—133, 186—192.

DIRECT evaporation under atmospheric pressure or in a vacuum is used where: the amount of liquor to be evaporated is so small that it would not pay to have a multiple effect; the boiling points are very high; the liquor deposits an encrusting material on concentration; where crystals of a certain size are desired; the material to be evaporated is corrosive, or decomposes at a high temperature; the object is to recover a volatile constituent or to obtain artificially a low temperature or a sudden chill as in refrigeration processes; the liquor to be evaporated is already a concentrated solution and the product is separated as a dry film, powder, or mass of crystals. Direct vacuum evaporation (single effect) shows very little economy of steam over evaporation under atmospheric pressure and none at all if the vacuum has to be produced by steam power. Multiple-effect evaporation is used where the cost of fuel is high and the quantity of liquor to be evaporated is large. Air drying or gas drying depends upon the property of partially saturated air or gas of taking up more vapour, and it may be profitably employed in drying at a comparatively low temperature such materials as pulp, paper, cloth etc., which are continuously entering and leaving the apparatus; in drying at low temperatures substances of which the internal structure must be preserved; where the final product must be obtained in a fine state of division; in drying such substances as are not affected by heat but are sticky or poor conductors of heat. The heat necessary to evaporate the water may be supplied by the air or gas itself or may be applied directly. Drying by means of radiant heat is used when it is necessary to concentrate substances that require a high temperature and when there is no suitable material of which to construct ordinary apparatus. Mechanical drying by the use of filter and hydraulic presses, and centrifugal machines, and refrigerating machinery for drying gases are also briefly discussed. Several tables are given and the method of using them to solve evaporation and drying problems explained. The subject of heat conductivity is considered, and it is pointed out that the heat conductivity tables of physicists are of little use in practice, as the rate of heat exchange is largely increased when ebullition becomes violent owing to the rapid motion of the liquid. The phenomena in an evaporator tube, methods of calculating temperature differences, types of multiple-effect evaporators, and the relative advantages of forward and backward flow are discussed and illustrated by charts.—W. H. C.

## PATENTS.

*Distillation or evaporation of liquids [tar, petroleum, etc.]* J. L. Major, Hull. Eng. Pat. 114,353, Apr. 26, 1917. (Appl. No. 5915 of 1917.)

THE liquid to be distilled is fed into an externally heated still through a number of pipes which extend from near the bottom of the still to the level of the surface of the liquid. The liquid in passing upwards through the pipes becomes heated and overflows from the upper ends of the pipes on to the surface of the main body of liquid in the still, the constituents of lower boiling point being vaporised before the liquid mixes with the main body. An annular tray with serrated edges

may be provided into which the pipes discharge and from the serrations of which the liquid overflows.—W. H. C.

*Liquids and gases ; Apparatus for effecting intimate contact between* — Kirkham, Hulett, and Chandler, Ltd., and S. Hersey, London. Eng. Pat. 114,370, June 16, 1917. (Appl. No. 8697 of 1917.)

IN apparatus for intimately mixing liquids and gases, of the type described in Eng. Pats. 18,129 of 1908, 23,813 of 1911, and 104,261 (this J., 1909, 873; 1912, 801, and 1917, 445), in which the liquid is projected centrifugally against the walls of a vessel through which the gas is passed, the liquid which rebounds from the walls is sprayed again. Curved pick-up pipes dip into the liquid contained in the vessel and deliver it to a rotating perforated tray, and an additional annular perforated tray is carried by the main tray outside and at a slightly lower level, so as to receive liquid which rebounds from the walls. The auxiliary tray may also be provided with scoop pipes dipping into the liquid, and alternatively its rim may be serrated. In another form, the auxiliary tray may be replaced by a series of radial projecting horizontal plates each having an upturned perforated serrated or like periphery, with or without a scoop pipe. The following edge of each plate may be upturned so as to act as a fan.—W. F. F.

*Refrigeration : Method of* — F. G. Keyes, Boston, Mass., Assignor to National Automatic Refrigerator Co. U.S. Pat. 1,258,017, Mar. 5, 1918. Date of appl., Jan. 27, 1917.

A MIXTURE of an absorbent, a solvent, and a refrigerant is distilled so that the solvent and refrigerant pass into the refrigerating chamber in proportions comprising substantially a critical boiling mixture. The resulting mixture is vaporised in the refrigerating chamber and re-absorbed by the absorbent in the same proportions. (See also U.S. Pat. 1,251,538; this J., 1918, 142 A.) —W. H. C.

*Dryers ; Evacuator for cylindrical* — C. L. St. Clair, Oregon City, Oreg., and J. H. Hoffman, Dansville, N.Y. U.S. Pat. 1,258,055, Mar. 5, 1918. Date of appl., Aug. 10, 1917.

A HORIZONTAL cylindrical dryer is provided with helical ribs on its inner surface, and a narrow chamber forming one end is divided by a spiral partition having several convolutions. A number of openings are provided around the circumference of the narrow chamber, each of which is connected with the end of one of the helical ribs. The narrow chamber is provided with an axial drain pipe.—W. F. F.

*Heat of liquids ; Apparatus for utilising the* — E. N. Trump, Syracuse, N.Y., Assignor to The Solvay Process Co., Solvay, N.Y. U.S. Pat. 1,258,165, Mar. 5, 1918. Date of appl., Mar. 7, 1916.

THE liquid is caused to flow under control through a series of connected chambers, each of which is connected with a separate compartment of an apparatus in which progressively lower steam pressures are maintained.—W. H. C.

*Mixing and agitating mechanism\* for extractors, etc.* O. Z. Howard, New York, Assignor to The Diamond Match Co., Chicago, Ill. U.S. Pat. 1,258,334, Mar. 5, 1918. Date of appl., Sep. 14, 1916.

THE agitating device is carried on a central vertical screwed shaft by which it is rotated and at the same time moved vertically upwards or



downwards. Means are provided for automatically arresting and reversing the motion of the shaft at each limit of the vertical movement.

—W. H. C.

*Heater; Solar* ——. D. A. Harrison, Los Angeles, Cal. U.S. Pat. 1,258,405, Mar. 5, 1918. Date of appl., July 24, 1915.

Two headers for supply and discharge of fluid are connected by thin slanting tubes, the upper header sloping from its centre towards both ends in the direction of the opposite header. A grooved reflector is arranged behind the tubes, each tube fitting within but slightly spaced from the walls of the groove. A sheet transparent to heat rays is arranged to cover the other side of the tubes, and another reflector extends over the upper header. Means are thus provided to direct the sun's rays on to the tubes and upper header. The tubes are contracted at their outlet ends in the upper header.—W. F. F.

*Gases; Apparatus for use in preparing solutions of* ——. G. Braam, Rotterdam, Netherlands. Eng. Pat. 111,858, Dec. 4, 1917. (Appl. No. 17,974 of 1917). Under Int. Conv., July 25, 1916.

SEE U.S. Pat. 1,256,291 of 1918; this J., 1918, 198 A.

*Waterproofing and rendering impervious to dust, bags, sacks, and the like; Method of* ——. J. Gadsden, Melbourne, Australia. U.S. Pat. 1,258,104, Mar. 5, 1918. Date of appl., July 6, 1917.

SEE Eng. Pat. 109,252 of 1917; this J., 1918, 45 A.

*Vessels or apparatus for use in concentration of sulphuric or other acids.* Eng. Pat. 114,072. See VII.

*Apparatus for concentrating solutions.* U.S. Pat. 1,258,168. See VII.

*Evaporating apparatus.* U.S. Pats. 1,258,562 and 1,258,563. See XVIII.

*Process of desiccating liquids.* U.S. Pat. 1,258,318. See XIXa.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Oridisable constituents of* ——. Part I. J. I. Graham and J. Hill. Inst. Min. Eng., Sept. 14, 1917. Trans. N. Eng. Inst. Min. Eng., 1918, 68, 37—54.

PREVIOUSLY published experiments on the extraction of coal with pyridine have been made at the boiling point of the solvent, without exclusion of air. These conditions favour retention, chemical or physical, of solvent and absorption of oxygen by the coal or its constituents. Extraction experiments have been made by the authors, on coal from the Barnsley Soft seam at 40° C., under diminished pressure (60 mm. mercury), and in an atmosphere of nitrogen. A yield of 10—15 % of extract was obtained after removal of solvent by distillation *in vacuo* and by washing with hydrochloric acid. The extract was a brown powder fusing on heating, partly soluble in caustic soda, insoluble in ammonia; 35—40 % of it was soluble in chloroform. The residue was black, coke-like, and retained pyridine tenaciously. The capacity for absorbing oxygen of the extract, residue, and the original coal was determined by an adaptation of methods previously described (see this J., 1914, 1000), at 30° and 90° C., and in presence of air and an atmosphere containing 90 % of oxygen. The absorption by the extract was negligibly small, whereas the residue displayed an avidity for oxygen almost equal to

that of the original coal. The so-called resinic constituent is thus not responsible for the spontaneous combustion of coal as commonly supposed, but rather the insoluble or cellulosic bodies. The extract, unlike those obtained by Wheeler (see this J., 1913, 969), was inert towards a photographic plate while the residue was strongly active, but this activity seems to be unconnected with the oxidisability. This activity is destroyed when the coal is coked.—H. J. II.

*Intensive toluol production. II. Proposed improvements in the concentrating and refining process.* Lichtenthaeler. See III.

### PATENTS.

*Coke ovens; Regenerators for* ——. E. C. R. Marks, London. From Soc. Franco-Belge de Fours à Coke, Brussels. Eng. Pat. 114,222, Apr. 13, 1917. (Appl. No. 5255 of 1917.)

Two sets of regenerators are arranged longitudinally below a battery of coke ovens, one set being heated by waste gases while the other set is heating the combustible gases. Each set comprises a central gallery divided transversely by partitions to form a row of regenerator chambers. These are filled with chequer brickwork and communicate alternately with two longitudinal flues which are arranged on each side of and parallel to the central gallery. The lower parts of the flues may also be filled with chequer brickwork, forming auxiliary regenerators, one of which may be outside the furnace setting. One of the flues may be wholly filled with chequer brickwork, and an auxiliary flue provided outside the furnace setting.—W. F. F.

*Gas wash boxes.* F. Thuman, London. From C. S. Chrisman, West Chester, Pa., U.S.A. Eng. Pat. 114,397, Nov. 7, 1917. (Appl. No. 16,290 of 1917.)

THE gas-inlet pipe dipping into the liquid in a wash-box is provided with a rim having a more extensive periphery, by making re-entrant spaces in the wall of the pipe. There may be two such re-entrant spaces at right angles, extending across the inlet pipe, so that in effect the pipe is divided into four branches, sector-shaped in cross-section, and separated by liquid spaces.—W. F. F.

*Hydrocarbons; Art of and means for cracking* ——. J. W. Coast, jun., Tulsa, Okla., Assignor to the Process Co., U.S. Pats. 1,258,190 and 1,258,191, Mar. 5, 1918. Date of appl., Mar. 22, 1917.

AT the bottom of a cracking still supported on a heating chamber, is a layer of fusible material so heavy that coke-forming substances float on its surface, a skimming device forcing them to a point above one of the end walls of the heating chamber. The contents of the still are maintained under pressure.—L. A. C.

*Petroleum hydrocarbons; Apparatus for cracking* ——. J. S. Cosden and J. W. Coast, jun., Tulsa, Okla., Assignors to The Process Co. U.S. Pat. 1,258,196, Mar. 5, 1918. Date of appl., Dec. 26, 1916.

SUPERHEATED vapours from a cracking chamber pass through a series of reflux condensers, the first of which is a supplementary cracking chamber into which a relatively cool hydrocarbon is sprayed. The vapours pass into another of the series into which steam is discharged. The pressure throughout the apparatus is greater than three atmospheres.—L. A. C.

*Distillation or evaporation of liquids [petroleum].* Eng. Pat. 111,353. See I.

*Apparatus for determining the quantity of carbon dioxide in flue-gases.* U.S. Pat. 1,260,020. See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION ; HEATING ; LIGHTING.

### PATENTS.

[*Peat and*] *pitch*; *Carbonisation of*—. W. Anderson, Helensburgh, Dumbarton. Eng. Pat. 114,371, Nov. 21, 1917. (Appl. No. 8789 of 1917.)

WET peat, 100 parts, and ground coal tar pitch, 20 parts, are mixed together and passed through a pulping machine, and then formed into blocks. The blocks are dried in a stove in a current of hot air, and are then suitable for carbonisation in a retort.—W. F. F.

*Electrodes for searchlights*. E. A. Sperry, Brooklyn, N.Y., U.S.A. Eng. Pat. 102,971, Dec. 22, 1916. (Appl. No. 18,411 of 1916.) Under Int. Conv., Dec. 22, 1915.

THE positive electrode consists of an outer shell of highly refractory pure carbon with an inner core of volatile material, *e.g.*, about 40–60 % of cerium or other rare earth fluoride or the like, mixed with carbon. The luminosity of the flame is increased and the formation of a crater facilitated. The flame has a high resistance and an anode drop of about 13 volts. The current density in the positive electrode is such that the flame is caused to issue from the tip of the electrode or mouth of the crater. The negative electrode may be provided with a core containing carbon and an alkali chlorate or borate so as to control the negative flame.—W. F. F.

## III.—TAR AND TAR PRODUCTS.

*Toluol production ; Intensive* —. II. *Proposed improvements in the concentrating and refining process*. F. E. Lichtenthaler, Met. and Chem. Eng., 1918, 18, 195–202. (See also this J., 1918, 174 A.)

THE present method of concentrating the toluol into crude toluol in intermittent stills and then washing and refining, is condemned as causing loss of toluol and waste of chemicals. Continuous concentration of the toluol into one fraction in a double-column continuous concentrator provided with an internal reflux system is advocated. The crude light oil is separated into crude benzol, crude toluol, and solvent naphtha. The crude toluol is then washed with sulphuric acid, water, caustic soda, and again with water, and rectified in an intermittent column still provided with direct and indirect steam heating and an internal reflux system. Automatic soda dissolvers are described and the room for improvement in the chemical washing process mentioned. Central concentration and refining stations to which the crude light oil obtained at the gas works might be sent for working up are advocated.—W. H. C.

### PATENTS.

*Distilling coal tar or the like ; Process and apparatus for* —. W. P. Thompson, Liverpool. From P. C. Reilly, Indianapolis, Ind., U.S.A. Eng. Pat. 114,453, Mar. 19, 1917. (Appl. No. 3948 of 1917.)

SEE U.S. Pat. 1,230,782 of 1917 ; this J., 1917, 866.

*Hydrocarbons ; Treatment of* — [to produce pitch]. J. Rosen, Assignor to Soc. des Combustibles Industriels, Paris. U.S. Pat. 1,259,674, Mar. 19, 1918. Date of appl., Feb. 3, 1913.

SEE Eng. Pat. 28,973 of 1912 ; this J., 1914, 246.

*Distillation or evaporation of liquids [tar, etc.]*. Eng. Pat. 114,353. See I.

*Carbonisation of [peat and] pitch*. Eng. Pat. 114,371. See IIb.

*Nitro compounds and process of making same from tar-oils*. U.S. Pat. 1,258,587. See XXII.

## IV.—COLOURING MATTERS AND DYES.

*Selenomethylene Blue*. P. Karrer, Ber., 1918, 51, 190–192.

TRUE Selenomethylene Blue bromide (3,6-tetramethyldiaminophenazselenonium bromide) is prepared by treating selenodiphenylamine with bromine in glacial acetic acid and triturating the phenazselenonium perbromide so formed with an alcoholic solution of dimethylamine. It has the same properties as ordinary Methylene Blue. (See also J. Chem. Soc., 1918, i., 237.)—J. C. W.

### PATENTS.

*Disazo dyestuffs for wool ; Manufacture of* —. O. Imray, London. From Soc. of Chem. Industry in Basle, Switzerland. Eng. Pat. 114,339, Mar. 27, 1917. (Appl. No. 4426 of 1917.)

SEE U.S. Pat. 1,233,742 of 1917 ; this J., 1917, 1002.

*Chromium compounds of azo dyestuffs and process of making same*. G. Engi and C. Jagerspacher, Assignors to Soc. of Chem. Industry in Basle, Switzerland. U.S. Pat. 1,259,499, Mar. 19, 1918. Date of appl., Nov. 29, 1915.

SEE Eng. Pat. 15,456 of 1915 ; this J., 1916, 1255.

*Mercury compounds of the acridine series and process of making same*. U.S. Pat. 1,259,517. See XX.

## V.—FIBRES ; TEXTILES ; CELLULOSE ; PAPER.

*Fibres ; Electrification of* —. S. A. Shorter. J. Textile Inst., 1918, 9, 6–10.

WOOL is positively charged by most materials with which it is likely to come into contact in the course of manufacture. Certain other materials, however, possess the property of electrifying wool with a negative charge; among these may be mentioned asbestos, vitreous silica, certain kinds of glass and porcelain, and certain kinds of leather, such as buff and wash leathers and raw hide, which are of the class of "oil leathers." Ordinary leathers electrify wool positively. Thus it has been proved experimentally that wool issuing from the front rollers of a French drawing frame had a strong positive charge, but after subsequently passing through the buff leather rubbing rollers, this charge was almost completely neutralised. For this reason some form of "oil leather" would be the most suitable foundation for the card clothing, while on the other hand, cotton-rubber cloth, which is frequently used, would be very objectionable from an electrical point of view. Experiments in the drawing and spinning processes showed that, under the ordinary conditions of draft, there was little or no electrification after passing between the back rollers but a strong electrification after the front rollers; when the back rollers were driven as fast as the front rollers, so that there was no draft, the electrification after the back rollers was strongly pronounced. Hence increase of speed increases electrification because the electricity is then generated faster than it can be discharged by conductivity. An untwisted sliver emerges from the front rollers far more highly electrified than a twisted sliver, owing to the wider and closer contact between the wool and the rollers and the greater lateral displacement of fibre over fibre. When both front rollers were covered with wash leather there was still a positive though diminished



charge on the wool, although the leather generates negative electricity on the fibre. Hence the lateral motion under pressure of one fibre over another develops more positive electricity than the negative electricity generated by the contact between the wool and the rollers. Negative electricity is also lost by escape through contact with the front rollers; this might be prevented by constructing them of insulating material. Increase of pressure increases electrification. The development of electricity in the finishing process is caused mainly by contact with wood and metal; it may be considerably diminished by the substitution of other materials, such as vitreous silica, porcelain, or glass. Covering the rollers with cloth is sometimes effective, according to the nature of the materials. Investigations on the influence of humidity showed that for room temperatures of 60°–70° F. (16°–21·5° C.), a difference of 5°–6° F. (2·8°–3·3° C.) between the dry and wet bulbs gave a very favourable decrease in the electrification; this is due more to condensation of a film of moisture on the rollers than to absorption of excessive humidity by the wool.—J. F. B.

*Wool containing vegetable matter; Carbonisation of —.* W. Harrison. J. Textile Inst., 1918, 9, 10–13.

IN the carbonisation process the loss of strength of the wool varies with the conditions of treatment. It is much more pronounced when the wool is heated at 100° C. while saturated with the dilute sulphuric acid, than when it is rapidly dried first and then heated under the same conditions. With acid at a concentration of 50 grms. per litre, little or no tendering occurs below 60° C. and if the acidified wool be first dried, it may be heated for 2 hours at 100° C. with very little tendering. With acid at 100 grms. per litre, tendering is produced during drying and subsequent heating. When tendering has been caused by drying wool with the acid in it, the wool may regain strength after washing and neutralising. This is due to the partial gelatinisation of the wool fibre by the concentrated acid and its subsequent coagulation when the acid is removed. In practice, the goods should be neutralised as soon as possible after carbonising. It is not desirable to employ an acid of more than 5% strength: the impregnated wool should be dried below 170° F. (77° C.) before heating further for the carbonisation of the vegetable matter; this last process should be as short as possible consistent with the destruction of the vegetable impurities. From experiments made to determine the consumption of acid, it is estimated that 100 lb. of air-dry wool containing 20% of vegetable matter, impregnated with 5% acid (6½° Tw.), would absorb about 10 gallons of liquid, after hydro-extracting, equal to 5 lb. of sulphuric acid. Of this, the quantity absorbed by the vegetable matter (1 lb. of acid) would be lost during crushing and "willowing." If the wool is washed in 400 galls. of water, 2·4 lb. of acid would be removed, leaving 1·6 lb. to be neutralised, with a consumption of 2 lb. of soda ash. It is noted that darkening in colour proceeds almost parallel to the degree of tendering of the wool.—J. F. B.

#### PATENTS.

*Flax and other fibres: Method and apparatus for retting —.* N. T. Bacon, Peace Dale, R.I. U.S. Pat. 1,258,083, Mar. 5, 1918. Date of appl., Apr. 26, 1917.

THE process of water-retting of flax is regulated by locally checking the retting action by bubbling gas through selected portions of the retting water.—J. F. B.

*Esparto or the like; Removing — from stationary digesters and the like used in paper-making.* W. M. Wallace, T. Marshall, and A. Brown, Denny, Stirling, and Bertrams, Ltd., Edinburgh. Eng. Pat. 114,348, Apr. 10, 1917. (Appl. No. 4991 of 1917.) Addition to Eng. Pat. 104,578, Apr. 16, 1916 (this J., 1917, 450).

As a modification of the original method for breaking up and discharging digested material from digesters, drainage vats, and other receptacles by the hydraulic force of a jet of water, a movable nozzle having a swivel or universal joint or a flexible portion, and operated in all directions by means of a lever and rod mechanism, is inserted through the charging hole of the digester when the lid is off. The material disintegrated by the force of the water is discharged through an opening in the bottom of the digester, and a pipe passing through an orifice in the false bottom connects the upper surface of this with the discharge orifice. This passage is kept clear by a suitably disposed water-jet nozzle fixed inside the boiler and connected to the water supply when the lid is opened.—J. F. B.

*Paper pulp; Process and apparatus for treating —.* G. C. Howard, Tacoma, Wash. U.S. Pat. 1,258,568, Mar. 5, 1918. Date of appl., May 29, 1916.

PAPER pulp, e.g., a mixture of wood-pulp and water, is introduced into a vat and caused to flow within the influence of a pair of rapidly rotating rollers, whereby the surface friction of the rollers draws a portion of the pulp and a reduced amount of water between the rolls, causing it to rise and float upon the remainder. The height of the liquid is adjusted so that the rolls are submerged, and means are provided to prevent the flow of the heavier pulp material beyond the roll and for discharging the withdrawn pulp and water and the remainder of the water and pulp by separate outlets.—J. F. B.

*Paper; Process and apparatus for regenerating old —.* H. Wade, London. From Soc. Union Française des Papeteries, Lyon, France. Eng. Pat. 114,257, Aug. 16, 1917. (Appl. No. 11,784 of 1917.)

THE material is triturated in the presence of water in a vat provided with stirrers, the blades of which are inclined relatively to their plane of rotation and are arranged to overlap during rotation, so that the blades of one stirrer engage with the angular spaces of the blades of the other. The bottom of the vat is provided with projecting teeth on which the paper is torn or triturated by the stirrers. A pump withdraws pulp from the bottom of the vat and delivers it into a bucket or trough situated over the top of the vat and containing a grooved roller which rubs the pulp against grooves in the bottom of the bucket and then discharges it back into the vat. At the end of the operation the pulp is transferred to the upper end of an inclined wire screen on which jets of water are directed in a uniform manner, under constant head, from an upper tank to wash the impurities through the screen as the pulp travels down it.—J. F. B.

*Paper; Process of making —.* W. H. Good-enough, Camas, Wash. U.S. Pat. 1,257,996, Mar. 5, 1918. Date of appl., Aug. 29, 1917.

IN the process of making news-print paper, 2 lb. of "sal soda" (sodium carbonate) is added to 1500 lb. of paper stock.—J. F. B.

*Cotton and other fibre-bearing seeds; Delinting of ginned —, and the segregation and collection of the detached fibres. Defibration of the decorticated hulls of cotton and other fibre-bearing seeds, and segregation and collection of the detached fibres.* E. C. de Segundo, London. Eng. Pats. 114,435 and 114,450, Feb. 28, 1917. (Appl. Nos. 10,753 of 1916 and 2975 of 1917.)

*Cellulose acetate; Manufacture of —.* J. Koetschet, Lyon, Assignor to Soc. Chim. Usines du Rhône, Paris, France. U.S. Pat. 1,258,913, Mar. 12, 1918. Date of appl., June 18, 1914.

SEE Fr. Pat. 473,399 of 1914; this J., 1915, 488.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Tendering of bleached (but badly scoured) vegetable yarns; Investigation of the cause of —.* S. H. Higgins. J. Soc. Dyers and Col., 1918, 35—43.

FROM experiments on the washing of cream yarn goods and on the retention of chlorine and acid by cotton and linen, it is concluded that the deterioration of imperfectly bleached textile fabrics during storing is due to mineral acids which have not been completely washed out of the goods, and that the chlorine reaction of chemicked and washed cloth is due to free chlorine; no evidence was obtained that chloramines play any part in this deterioration, as suggested by Cross and Bevan (this J., 1908, 260; compare Cross, J. Soc. Dyers and Col., April, 1918). The yellowing of bleached goods on storing or on steaming is ascribed to the darkening of the melted wax, and can be prevented only by good scouring.

—F. W. A.

*Dyeings fast to light; The production of —.* K. Gebhard. Sealed communication, June 12, 1910. J. Soc. Dyers and Col., 1918, 34, 74—76. (Compare this J., 1910, 691.)

THREE methods are discussed whereby the fading of dyestuffs may be prevented. (1) Dislocation of the original equilibrium between the oxygen in solution and the ions of the water (the perhydroxyl ions). This equilibrium



is influenced by all other bodies in the system. When it is ascertained what kind of influence takes place under the action of light the equilibrium can be established in a direction favourable to the durability of the dyestuff by a suitable concentration of the substances present or the introduction of new substances. The equilibrium is altered to the disadvantage of the perhydroxyl ions by increasing the  $H^+$  ion concentration in aqueous solutions of acid dyestuffs. This is effective with wool but not with cotton, as the latter reacts easily with  $H^+$  and  $OH^+$  and removes them, thus facilitating the generation of the unfavourable  $OOH$  ions. (2) Preventing or impeding the reaction between the dyestuff and the perhydroxyl ions. The facility of reaction may be decreased by converting the dyestuff into the least reactive state, e.g., by making it difficultly soluble. This is realised in the case of vat dyestuffs, colour lakes, and after-treatment with copper sulphate. Another method consists in diverting the reactivity of the perhydroxyl ions into other channels, e.g., by the addition of other substances more easily oxidisable than the dyestuff. Only those substances are useful which are capable of being automatically regenerated; this regeneration should be brought about by the light itself. Such catalytic reducing agents are cerium

sulphate or calcium citrate. The enzyme catalase is a substance of this order but is not practically available. (3) Transformation of the dyestuff peroxide into more stable compounds. Dyestuffs might conceivably be used which possess the property of splitting off the elements of water from the labile dyestuff-peroxyhydrates and forming the more stable dyestuff-peroxides. This dehydration may be promoted by the introduction of other substances into the system. It may also be possible to replace the peroxide oxygen in the dyestuff by other radicals which make it more stable, e.g., by reciprocal decomposition of a dyestuff-peroxide with an inorganic or organic peroxide; or the radical may be introduced directly where the dissociated oxygen attaches itself during the auto-oxidation. Such a combination is perhaps formed when dyeing reduced hydroxyanthraquinones on chrome mordants. In some cases the fibre itself may serve as a suitable radical. Fastness to light may be increased by excluding the unfavourable influence of moisture, e.g., by a layer of oil or wax, but the method has certain definite limitations.—J. F. B.

### PATENTS.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,258,203, (B) 1,258,542, (C) 1,258,543, (D) 1,258,544, (E) 1,258,545, (F) 1,258,546, Mar. 5, 1918. Dates of appl. (A) Dec. 5, 1916, (B) and (C) Dec. 22, 1916, (D) Dec. 29, 1916, (E) Jan. 11, 1917, (F) Jan. 31, 1917.

(A) THE dyeing machine comprises a receptacle in which is a fibre chamber formed by the walls of the receptacle, a perforated bottom plate having openings diverging upwards, and a perforated top plate having openings diverging downwards; means are provided for moving the top plate relatively to the bottom plate, so as to compress the fibre, and for holding it in the desired position. Below the fibre chamber is a second chamber having a perforated bottom plate with openings diverging upwards and tubes also diverging upwards integral with and opening into each of the openings of the bottom of the fibre chamber. Alternatively, a fourth perforated plate with openings diverging downwards may be spaced beneath the third plate, the tubes then connecting the openings of the third and fourth plates. A circulating device is provided for forcing liquid through the entire system. A small testing device similar and proportionate to the large one may be arranged externally, the small device being connected to the second chamber of the large one, so that the liquid passes through both machines in parallel. (B) Below the second or liquid supply chamber, a series of tapering rods converging downwards are arranged and means are provided for heating the liquid. (C) The openings in the plates are of pyramidal form and the tube chamber is provided with pyramidal tubes. (D) The openings in the plates are elongated, and below the liquid supply chamber is a plate-carrying chamber converging downwards, containing a series of plates diverging upwards, and having tapered edges. (E) The fixed plate of the fibre chamber is constructed of a series of thin blades attached to the wall of the receptacle and a reservoir is inserted in the external pump circuit. (F) A series of members converging upwards is provided above the movable top plate of the fibre chamber.—J. F. B.

*Cotton fabrics; Treatment of —.* O. Klausner, Assignor to Akt.-Ges. Cilander, Herisau, Switzerland. U.S. Pat. 1,258,225, Mar. 5, 1918. Date of appl., Aug. 12, 1916.

SEE Eng. Pat. 103,432 of 1916; this J., 1917, 289.



## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Boric acid; Ignition of* —. C. R. Bagshaw. Analyst, 1918, 43, 138—139.

To ascertain whether boric acid could be determined in boric acid powder and boric acid ointment by igniting the sample and weighing the residual boron trioxide, a number of experiments were carried out. Boric acid ointment known to contain 10% of the acid yielded 5.11% of boron trioxide, equivalent to 9.06% of boric acid; this residue of boron trioxide decreased in weight continuously on further heating. Boric acid itself should yield 56.4% of boron trioxide on ignition, but the amount found by the author varied from 52.6 to 55.1% according to the conditions under which the substance was heated.

—W. P. S.

*Argon; Preparation of* — in the laboratory. M. Bodenstein and L. Wachenheim. Ber., 1918, 51, 265—270.

ARGON is recovered from commercial oxygen, made from the air, by combustion with hydrogen. The process described is an adaptation of Ger. Pat. 295,572 (see Fr. Pat. 473,985; this J., 1915, 717) to laboratory requirements. (See further, J. Chem. Soc., 1918, ii., 166.)—J. C. W.

*Influence of calcium sulphate on the action of water on iron.* Medinger. See X.

*Gasometric determination of combined carbon dioxide.* Mestrezat. See XIXb.

*Determination of potash.* Blount. See XXIII.

*Detection and determination of bromine ions by means of fuchsin-sulphuric acid reagent.* Denigès and Chelle. See XXIII.

*New general method of determining iodine in inorganic and organic compounds.* Tarugi. See XXIII.

### PATENTS.

*Sulphuric or other acids; Vessels or apparatus for use in the concentration of* —. S. J. Tungay and G. B. Haughton, London. Eng. Pat. 114,072, May 23, 1917. (Appl. No. 7418 of 1917.)

In the concentration of sulphuric or other acid, the liquid flows over square or oblong trays arranged in cascade and constructed of acid-proof metal, passive iron, lead, fused quartz, pottery, glass, or the like. The trays are provided with longitudinal or transverse channels or corrugations to increase the heating surface and prevent deposition of solid matter. (Reference has been directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4391 of 1875, 18,751 of 1890, and 9514 and 13,927 of 1893; this J., 1891, 240; 1893, 690; and 1894, 636.)—W. F. F.

*Acetic acid from acetic aldehyde; Manufacture of* —. H. Dreyfus, Basle, Switzerland. Eng. Pat. 108,459, Apr. 23, 1917. (Appl. No. 5683 of 1917.) Under Int. Conv., Apr. 27, 1916.

In the oxidation of acetaldehyde to acetic acid by air or oxygen as described in Eng. Pat. 105,065 of 1917 (this J., 1918, 180 A), the best temperature is found to be between 150° and 250° C., preferably between 150° and 200° C. Suitable catalysts are: Platinum, copper and its compounds, chromium compounds, iron, uranium

oxide, vanadium oxide, cerium oxide, etc. Although catalysts facilitate the reaction, they are not necessary for complete oxidation.—L. A. C.

*Concentrating [phosphoric acid] solutions; Apparatus for* —. W. J. Wayte, New York. U.S. Pat. 1,258,168, Mar. 5, 1918. Date of appl., Feb. 20, 1917.

A SHALLOW tank divided into compartments and provided with wheels is supported below a fixed vapour hood so that it may be raised into contact with the lower part of the hood or lowered on to a truck and wheeled aside. Heating coils supported from the fixed hood depend into the tank, one into each compartment, when it is in its raised position. The apparatus is intended more especially for the concentration of a solution of phosphoric acid.—W. H. C.

*Kelp char, etc.; Process of and apparatus for treating* — [to obtain alkali salts]. (A) F. Van D. Crusier, Oswego, N.Y., and (B) F. Van D. Crusier, Oswego, and O. Z. Howard, New York, Assignors to The Diamond Match Co., Chicago. Ill. U.S. Pats. (A) 1,258,320 and (B) 1,258,321, Mar. 5, 1918. Dates of appl., Aug. 10 and Sept. 14, 1916.

AT the bottom of each of a series of vats placed at different levels is a valved discharge opening leading to the vat below and also lateral outlets covered by strainers. The kelp char is extracted in the top vat by extraction liquor from the succeeding vats, the solid matter being allowed to settle and the liquor drawn off for concentration; a further quantity of liquor is then added and the material flushed into the second vat, this process being repeated down the series until extraction is complete.—L. A. C.

*Alkali chlorides; Process for the electrolysis of* —. R. van Hasselt, The Hague, Netherlands. U.S. Pat. 1,259,683, Mar. 19, 1918. Date of appl., Oct. 12, 1916.

SEE Eng. Pat. 101,440 of 1916; this J., 1916, 1109.

*Preparation of anhydrous sulphide of zinc.* Eng. Pat. 106,489. See XIII.

## VIII.—GLASS; CERAMICS.

*Glass; Analysis of* —. L. Ronnet. Ann. Falsif., 1918, 11, 26—31.

To determine silica, iron, aluminium, manganese, calcium, magnesium, potassium, and sodium in glass, the author employs mainly the usual methods of analysis. Iron and aluminium are precipitated together by a basic precipitation and then separated from one another by dissolving in hydrochloric acid and boiling with sodium thio-sulphate, the iron being converted into a soluble double thiosulphate of iron and sodium, whilst the alumina is precipitated together with sulphur. Potassium and sodium are weighed together as their chlorides and the potassium is then determined by the perchlorate method. (See also J. Chem. Soc., June, 1918.)—W. P. S.

### PATENTS.

*Glass-gathering mechanism.* The Westlake European Machine Co., Assignees of A. Kadow, Toledo, Ohio, U.S.A. Eng. Pat. 112,613, June 18, 1917. (Appl. No. 8758 of 1917.) Under Int. Conv., Jan. 11, 1917.

*Glass ware; Manufacture of hollow* — and machinery therefor. T. B. Kitson and G. O. Tague, Leeds. Eng. Pat. 114,477, Apr. 5, 1917. (Appl. No. 4927 of 1917.)

## IX.—BUILDING MATERIALS.

## PATENTS.

*Stones; Manufacture of metal-coated artificial* ——. H. Welte, Znaim, Austria. U.S. Pat. 1,258,282, Mar. 5, 1918. Date of appl., Nov. 10, 1915.

SEE Eng. Pat. 17,862 of 1915; this J., 1917, 139.

*Mortar; Manufacture of* ——. J. Joachim, Berlin, Assignor to Mortelwerk Joliet Ges. m. b. H., Waidmannslust, Germany. U.S. Pat. 1,259,241, Mar. 12, 1918. Date of appl., Aug. 5, 1913.

SEE Fr. Pat. 461,129 of 1913; this J., 1914, 259.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steel ingots; Defects in* ——. J. N. Kilby. Iron and Steel Inst., May, 1918. [Advance copy.] 21 pages.

EXPERIENCE shows that the variations in casting temperatures are not as great as usually supposed, and that the speed of pouring has a greater influence on the cracking of ingots. The teeming time for a 7200 lb. ingot is given as six minutes; if it be less than three minutes 80% of the ingots will show cracks on rolling. The addition of 8 to 10% of lime (or 6 to 8% of magnesia) to acid open-hearth slags is recommended as giving a reduced content of ferrous oxide, thereby enabling the slag to remove more extraneous matter present in the steel. The amount of manganese remaining in the metal is proportional to the lime content of the slag.—C. A. K.

*Steel; Effects of cold working on the elastic properties of* ——. J. A. Van den Broek. Iron and Steel Inst., May, 1918. [Advance copy.] 41 pages.

MILD steel was subjected to cold stretching, compression, and twisting, and afterwards tested with reference to its elastic properties. It was found, contrary to accepted opinion, that cold stretching does not lower the elastic limit in compression, and the effects of cold working are probably a function of the strains and are independent of the stresses involved in the process. When a mild steel is cold worked and properly heat treated, the elastic limit may be raised more than 100% when tested in the same direction as the applied strain. Tempering at 100° C. to 300° C., or ageing the cold-worked steel, tends to improve the elastic properties. Application of heat merely accelerates the effect of time. Cold working breaks up the coarser crystalline structure and planes of weakness, with formation of amorphous steel, and after ageing, the elastic limit is not reached until a greater stress than that at which cold working is discontinued is attained. The beneficial effect of cold working is permanent.—C. A. K.

*Pearlite [in steel]; Effect of cold work on the divorce of* ——. J. H. Whiteley. Iron and Steel Inst., May, 1918. [Advance copy.] 9 pages.

By annealing cold-worked pearlitic steels a little below the critical point (690° C.), the values for combined carbon as given by the Eggertz colour test are considerably increased as compared with the unstrained metal. At 500° C. pearlite showed a tendency to break up, and at 680° C. the divorce was completed in less than two hours, the cementite then occurring in rounded particles uniformly distributed. The pearlite in an unstrained steel was practically unaltered. The granular structure of the cementite is similar to the "granular pearlite" in tempered steels.—C. A. K.

*Steel; Constitution and occurrence of non-metallic inclusions in* ——. A. McCance. Iron and Steel Inst., May, 1918. [Advance copy.] 48 pages.

ATTENTION is drawn to the important part that non-metallic inclusions play as a source of weakness in stressed materials. Ziegler's suggestion that non-metallic inclusions are soluble in steel is not supported and their practical importance is concerned with their existence as suspensions. In consequence of this they do not obey the laws of segregation governing other elements in solution in liquid steel, but their ultimate position is determined by secondary influences such as convection currents or local movements of crystallisation. The probability of particles of 0.001 cm. diam. rising to the surface of an ingot is practically nil, but coalescence occurs during the solidification of the last portions of metal, the suspensoids thereby being crowded together. Consequently the average size of particles found in the solid ingot is always larger than that of the particles occurring in the liquid steel. The opinion is expressed that the prevention of sulphur red-shortness by manganese is due to manganese sulphide being insoluble and therefore precipitating the sulphur out of solution in the steel. In commercial steels sulphur inclusions may be composed of manganese sulphide but not of iron sulphide even in solid solution. The eutectic of MnO—SiO<sub>2</sub> solidifies at about 1180° C. with a composition of 41% SiO<sub>2</sub> and 59% MnO. As most of the silicates isolated from steels have approximately the same percentage of silica it is concluded that they belong to the most easily fusible class, alumina and iron oxide making up deficiency in manganese oxide. Consideration of the system iron oxide-silica indicates that there is probably a physical equilibrium between Fe<sub>2</sub>O<sub>3</sub>, FeO, and SiO<sub>2</sub>, independent of their chemical grouping. High working temperatures are advocated to minimise inclusion dangers on both physical and chemical grounds. Inclusions of an adventitious nature are chiefly chips of refractory material with which the steel comes in contact. Manganese silicate shows a selective solubility for the alumina in firebrick inclusions, and the interaction between firebrick and steels containing manganese has a distinct bearing on the wear of ladle nozzles and runners conveying steel to the moulds. Hydrofluoric acid may be used as a substitute for Stead's cupric reagent (this J., 1915, 616) for the identification of phosphorus, especially in cast samples.—C. A. K.

*Steel; Inclusions in — and ferrite lines.* J. E. Stead. Iron and Steel Inst., May, 1918. [Advance copy.] 6 pages.

EXPERIMENTS were made to determine whether or not non-metallic inclusions in steel induce ferrite to crystallise around them. In no case did quartz, magnesia, lime, basic open-hearth slag, cyanonitride of titanium, or manganese sulphide inclusions, artificially introduced into a steel bar, lead to development of a ferrite envelope, when the bar was heated to and slowly cooled from about 1200° C. Fluorspar proved an exception in the case of 0.16 and 0.31% carbon steels, and mill scale and slags containing iron oxide wholly or partially decarburised the adjacent metal. Hermetically sealed cavities made in steel did not induce formation of ferrite. The influence of phosphorus was shown by similarly heating and cooling two forged steels, containing 0.07 and 0.3% P respectively. Before heating both showed white ferrite bands or lines round the non-metallic inclusions, but after the slow cooling from 1200° C. these were absent, due to the concentration of the phosphorus in the inclusions being reduced by inter-diffusion at that temperature (see following abstract). Pure slag particles,



free from phosphorus, were not surrounded by a ferrite layer, and the presence of such ferrite is associated with a high phosphorus inclusion.

—C. A. K.

*Iron, carbon, and phosphorus.* J. E. Stead. *Iron and Steel Inst.*, May, 1918. [Advance copy.] 24 pages.

THE diffusion of phosphorus, in solid solution, in iron has been noted in a cast alloy containing about 3% C and 0.3% P. In the original specimen the phosphorus was associated with ferrite. When the steel was heated to 960° C. and quenched in water, it was found that carbon had diffused into the ferrite of lower phosphorus content, but on slow cooling the carbon returned to the parts still lower in phosphorus. When heated to 1200° C. and slowly cooled, the phosphorus interdiffused and became more evenly distributed and on cooling no separation was effected. It is suggested, as the solution pressure of carbon increases rapidly with increase in temperature, and as iron phosphide is mobile at a high temperature, that if an alloy containing phosphorus is carburised at a high temperature, the entrance of carbon into solid solution would increase the internal tension and drive the phosphorus into less carburised areas. Concentration of phosphorus in these areas would proceed until the tension of the phosphide in solid solution balanced the tension of carbide in solid solution at any given temperature, and if that temperature exceeded the formation point of the liquid ternary eutectic Fe—C—P, the internal tension of the high phosphide ferrite would become so great that the phosphide would be forced out of solid solution, forming a eutectic liquid. This temperature is given as 1200° to 1300° C., from trial on an alloy containing 0.5% P, an abnormal figure for a commercial steel. In a tentative investigation to determine the critical point of solution of iron phosphide in iron, it was found that solution of phosphide in phosphorus-free iron had occurred at 700° C., but in iron practically saturated with phosphide in solution the temperature is higher. Conversely, observations on the temperature at which iron phosphide ( $\text{Fe}_3\text{P}$ ) falls out of solid solution shows that the amount present is a vital factor. Methods for the detection of phosphorus in solid solution are given. (See also this J., 1915, 616.)—C. A. K.

*Hypo-eutectic steel; Transformation of martensite in —.* M. Matweiff. *Rev. Soc. Russe de Mét.*, 1916, 1., 149—155. *Rev. Mét.*, 1918, 15, Ext., 44—48.

OBSERVATIONS of the micro-structure of hypo-eutectic steel containing C 0.26—0.30%, Mn 0.60—0.90%, Si 0.2—0.4%, S less than 0.035%, and P less than 0.05%, were made after hardening and tempering under different conditions. A cylindrical specimen was heated so that the temperature varied from the melting point at one end to a comparatively low temperature at the other end, and the specimen was then quenched in cold water so that the hardness varied from a maximum at one end to a minimum at the other. Micrographs are shown, taken at five zones along the specimen measuring from the hardened end, and these show (1) the presence of martensite in large needles, (2) martensite in small needles, (3) that the pearlite has been converted into martensite but the ferrite has not been dissolved in the latter, (4) the simultaneous presence of pearlite, ferrite, and martensite, and (5) the presence of pearlite and ferrite only, indicating a soft metal. The effect of tempering, at various temperatures, specimens hardened as above was then observed. When the reheating temperature was below 300° C. no change took place, but above

300° C. zones (1) and (2) showed the presence of sorbite, zones (3) and (4) showed the presence of sorbite in place of amorphous martensite, and zone (5) showed no change. Tempering at 500°—600° C. showed no important changes, but at 750° C. zone (1) showed deposits of free ferrite on the surface of the grains of martensite, indicating overheating, and zones (2) and (3) showed the texture due to normal heating. It is concluded that steel hardened at a very high temperature would show little strength and ductility owing to the coarse martensite structure, and that the breaking strength increases to a maximum as the hardening temperature is reduced to about 800° C. When the steel is subsequently tempered, the breaking strength increases with rise of the temperature of reheating.—W. F. F.

*Manganese [in steel]; Volumetric determination of — by means of sodium arsenite.* F. Ibbotson. *Chem. News*, 1918, 117, 157—158.

ATTENTION is directed to a source of error in the determination of manganese in steel when the metal is dissolved in nitric acid, the manganese oxidised to permanganate, and the latter titrated with sodium arsenite solution. In the presence of nitric acid, the arsenite solution has a reducing value about 33% more than it has when used for titrating a permanganate solution free from nitric acid. Reliable results are obtained, however, if the arsenite solution is standardised against permanganate under the conditions obtaining in the actual determination, i.e., in the presence of nitric acid, etc.—W. P. S.

*Nickel-chrome steel; Allotropic transformations and microstructure of —.* A. M. Masloff. *Rev. Soc. Russe de Mét.*, 1915, 1., 495—515. *Rev. Mét.*, 1918, 15, Ext., 37—44.

EXPERIMENTS were made on samples of nickel-chrome steel to determine separately the effect on the micro-structure of the maximum temperature of heating, rate of heating and cooling, and variations in the proportions of nickel and chromium. The specimens tested contained approximately C 0.5%, Mn 0.3%, Si 0.25%, P 0.03%, while the proportions of chromium and nickel were varied as desired from 0.92% to 2.03% and 1.07% to 5.53% respectively. The influence of the maximum temperature of heating on the transformation or recalcence point observed during subsequent cooling was shown to be practically negligible if the rate of cooling was not greatly varied, and the influence of the rate of heating was also negligible. Variation in the rate of cooling had considerable effect. When the rate of cooling between 750° and 550° C. varies from 1° to 32° C. per minute, the steel shows the same transformation point as if chromium were absent, and contains troostite and pearlite. The transformation point falls as the rate of cooling increases, and becomes less clearly marked, while the specimen shows the structure of pure troostite changing with the most rapid cooling to martensite. The presence of nickel was found to reduce the temperature at which the solid solution of carbon in iron decomposes, while chromium was found to have a smaller but opposite effect. Chromium, however, retards the decomposition of this solid solution. Steel containing a small amount of nickel but sufficient chromium may therefore be hardened by cooling in air.—W. F. F.

*High-speed steel; Researches on —.* M. Yatskevitch. *Rev. Mét.*, 1918, 15, 65—112.

EXPERIMENTS were made to determine the effect of varying heat treatment upon the transformation points, the general form of the heating and cooling curves, the micro-structure, and the hardness of a sample of high-speed steel. The



sample contained W 18.20%, Cr 3.00%, C 0.62%, V 0.95%, Si 0.27%, Mn 0.38%, S 0.025%, and P 0.025%. The heating and cooling curves were obtained by the Saladin-Le Chatelier apparatus, in which a neutral body was employed in addition to the sample, the two registering thermocouples being connected differentially according to the Roberts-Austen method so that differential curves were obtained. The effect of the rate of cooling was also observed in three series of experiments in which the cooling took place (1) within the heating tube, (2) in the air, and (3) in a current created by a fan. Series of heating curves are shown in which the maximum temperature of the specimen varies from 700° C. to 1050° C. and these show changes of curvature at about 400°–500° C. and points of inflexion at 773° C. and 856° C., attributed to three distinct exothermic reactions. The corresponding cooling curves show in general (1) only a single point of inflexion or transformation point, which falls as the maximum temperature increases, except that with the higher maxima a second lower point of inflexion appears at about 500° C.; (2) points of inflexion similar to (1) except that the lower point of inflexion varies from 250° to 370° C. and appears with the lower maximum temperatures, and (3) (rapid cooling) similar curves with more strongly marked lower point of inflexion, the upper point disappearing with the higher maximum temperatures. These results show the importance of the time factor in the hardening and tempering of high-speed steels. Corresponding curves are given in which the maximum temperature is above the melting point, and also comparative curves showing the variation in hardness with temperature. A number of micrographs are given showing the effect on hardness of varying heat treatment, and varying rates of cooling. The presence in the steel of various combinations of the constituent elements can be shown by suitable choice of etching reagents. The higher maximum temperatures show the increased production of particles which resist the action of reagents.—W. F. F.

*Cobalt steel; Determination of cobalt and nickel in* —. W. R. Schoeller and A. R. Powell. *Iron and Steel Inst.*, May, 1918. [Advance copy.] 5 pages.

THE method is based on the separation of hexamine cobaltous and hexamine nickelous iodides (this J., 1917, 720), the process being modified by the previous removal of iron and chromium. 2 grms. of drillings is dissolved in 30 c.c. each of hydrochloric and nitric acids, and evaporated to dryness. 2 c.c. of sulphuric acid (1:1), a little hydrochloric acid, and 25 c.c. of water are added, and the cooled solution titrated with 20% sodium carbonate solution, using a thiocyanate solution as a spot indicator. When the red colour grows faint, the solution is made up to 200 c.c. and filtered. 100 c.c. of the filtered solution is heated to boiling with 1–2 c.c. of nitric acid, and treated with 25 c.c. of bromine water and an excess of caustic soda. The precipitate contains all the cobalt and nickel and traces of iron, chromium, and manganese. It is filtered off, dissolved in hydrochloric acid, and the solution evaporated with a few c.c. of nitric acid, and treated with a saturated solution of 1 gm. of tartaric acid and 50 c.c. of ammonia (sp. gr. 0.88), followed by a saturated solution of 4 grms. of potassium iodide. The iodides of cobalt and nickel, after washing with ammoniacal potassium iodide solution, are dissolved in 10 c.c. of hydrochloric acid (1:1) with addition of a little sodium sulphite, the solution treated with 5 c.c. of 25% ammonium phosphate solution, and the cobalt precipitated by addition of ammonia until the blue amorphous precipitate becomes pink and crystalline. The

cobalt-ammonium phosphate is titrated with *N/5* sulphuric or nitric acid until the precipitate disappears (see this J., 1916, 656). Manganese if present is precipitated with the cobalt. It is determined in the liquid, after the titration, by the persulphate method, and a correction applied. 1 c.c. of *N/5* acid = 0.0059 gm. Co. The filtrate from the cobalt-ammonium phosphate is cooled and titrated with a freshly standardised solution containing 10 grms. of sodium cyanide, 2 grms. of caustic soda, and 1 gm. of silver nitrate, until the silver iodide precipitate at first produced redissolves.—C. A. K.

*Calcium sulphate; Influence of — on the action of water on iron.* P. Medinger. *Ber.*, 1918, 51, 270–271.

THE fact that gas and water pipes suffer corrosion particularly severely when they lie in heavy clays containing calcium sulphate is due to the depression of the ionisation of the bicarbonates and consequent liberation of more hydrogen ions from the carbonic acid. This influence of the sulphate on a solution containing calcium bicarbonate and free carbonic acid may be demonstrated by means of litmus.—J. C. W.

[*Gold amalgamation.*] *Discussion on mercury losses.* J. Chem., Met., Min. Soc., S. Africa, 1918, 18, 173–180.

H. RUSDEN stated that free mercury exists in black sand in the globular and floured forms. The globular mercury can be recovered from the tube mill discharge in a Batea pan. In the subsequent cyanide treatment a portion of the mercury is dissolved, and precipitated by zinc, a zinc-gold amalgam being sometimes formed. The average recovery of mercury from black sand is 2 lb. per ton.

H. A. White explained that coarseness of pulp as well as the mineral composition (especially the presence of chlorite, talc, clay, and certain sulphides) may have a considerable influence upon the mercury consumption. He suggested leaching black sand residues with sodium sulphide and precipitating the solution with aluminium powder. If the supply of mercury should become inadequate, it might become necessary to adopt blankets, and cyanide the finely ground coarser metal thus caught. A few experiments bearing upon the adhesion of gold to grease tables were made on the mill discharge stream. By removing the grease and concentrate every two hours, the recovery was 28% of that obtained by amalgamation. Better results might be expected from a continuous grease belt system. In another test, a plate was divided into three equal longitudinal strips: the middle one was covered with canvas, the outside ones with blanketing. In 24 hours the recovery on canvas was 70, and that on the blankets 40%, of the recovery by amalgamation.

F. Wartenweiler stated that, by the use of Batea pans, the mercury content of black sand had been reduced from 0.45–0.75 to 0.18%. Gold precipitates taken from extractor boxes assayed 0.15–0.25% Hg.—W. R. S.

[*Gold-bearing concentrates; Cyaniding versus smelting* [of —]. A. E. Drucker. *Eng. and Min. J.*, 1918, 105, 454.

STATISTICS are given showing the importance of cyaniding raw gold-bearing concentrates on the spot. Fine grinding in cyanide solution to pass a 200-mesh screen is necessary for an extraction of 90–95% of the gold within 24–48 hours. The raw concentrates obtained at Maibong, Korea, were treated by fine grinding in a cyanide solution, air agitation, decantation, and filter pressing, at a minimum cost of \$4.47 per ton and a gold



extraction of 91.2%. The concentrates were formerly shipped to America for smelting at a total cost for marketing of over \$30 per ton.—W. F. F.

*Chalcopyrite; Flotation of — in chalcopyrite-pyrrhotite ores of S. Oregon.* W. H. Coghill. Tech. Paper 182, U.S. Bureau of Mines.

The separation of these sulphide ores, which have nearly the same density, is difficult by ordinary methods of ore dressing. Flotation experiments gave favourable results. The ore was ground to pass a 200-mesh sieve. The oil used ("Flotco No. 1") gave a steep surface tension curve and was characterised by high solubility. The water was neutral. The extraction of copper attained was 78.7% and a concentration ratio of 12:1, while the product contained 27.4% of copper. The gold which is present in the chalcopyrite, passed into the tailings in the process, but seems to be easily recoverable by amalgamation. An insoluble oil is to be avoided as also the introduction of iron from the crushing plant.—H. J. H.

*Copper minerals; Determination of — in partly oxidised ores.* C. E. Van Barneveld and E. S. Leaver. U.S. Bureau of Mines. Eng. and Min. J., 1918, 105, 552—555.

It is important in ore flotation to be able to distinguish between copper present as sulphide and oxide minerals. Extraction by 5% cold sulphuric acid does not effect a quantitative separation of oxidised compounds. Metallic iron from the crushing machinery is dissolved and causes deposition of metallic copper. Only one-half of the copper in cuprite passes into solution, while some chalcopyrite, bornite, and chalcocite are also dissolved. Neither does a 5% solution of ammonia give satisfactory results. The sulphides are dissolved to a slight extent, while the formation of gelatinous silica interferes with the solution of oxidised minerals. These are also defects of Hunt and Thurston's method (Colorado School of Mines, May, 1917, 7, 157) based on the extraction of the oxidised minerals with alkaline sodium tartrate and alkaline ammonium sulphate. Extraction with dilute sulphurous acid is considered to give the most correct results:—2 grms. of ore, pulped to pass a 100- or 150-mesh sieve, is shaken with a solution containing 3% of  $\text{SO}_2$  for one-half to two hours. The insoluble residue is filtered off and washed with dilute sulphurous acid, and the filtrate then contains all the copper present as oxide, carbonate, or silicate in the ore. After acidification with nitric acid the copper can be estimated electrolytically. Sulphide minerals are not affected. Metallic iron—up to 3%—is rapidly dissolved and has no influence if excess of sulphurous acid is present. No interference is caused by the formation of gelatinous silica and ores of any grade can be dealt with. Metallic copper must be determined separately.—H. J. H.

*Sulphur [copper sulphide] and copper oxide determination.* C. G. Maier. Eng. and Min. J., 1918, 105, 372—373.

In a method of determining accurately the proportions of copper sulphide and "oxide" or acid-soluble copper in a sample of copper ore, especially for use in flotation processes, 1 to 3 grms. of the ore is heated to 80°—90° C. with 50 c.c. of 4% sulphuric acid for 30—45 minutes. The solution is cooled and 4 to 5 c.c. of clean mercury added and mixed thoroughly with the residue, so that it amalgamates with the metallic copper present. The solution is decanted and the ore washed out of the beaker, leaving the mercury, which contains all the original metallic copper and also any copper reduced from cuprous

oxide. The solution is filtered and the copper determined separately in the filtrate and residue, preferably by the iodide method. The copper in the residue represents the sulphide and that in the solution the "oxide" or acid-soluble copper.—W. F. F.

*Sulphur analysis [in ores]; Method of —.* F. G. Hawley. Eng. and Min. J., 1918, 105, 385—386.

A METHOD is described for estimating sulphur gravimetrically in ores and furnace products by oxidation to sulphate and precipitation as barium sulphate, which avoids the difficulties usually experienced in oxidising mattes and calcines, or ores containing much copper or zinc sulphide. 0.5—1.0 gm. of the sample is treated with 10 c.c. of a 20% solution of sodium chlorate, and 7—15 drops of a mixture of equal parts of bromine and glacial acetic acid. 10 c.c. of nitric acid nearly saturated with potassium chlorate is then added and the mixture agitated. After 2 or 3 minutes the sulphur is oxidised, and the mixture is then evaporated to dryness. The residue is cooled, 5 c.c. of 60% hydrochloric acid added, and after heating till all soluble substances are dissolved, the solution is diluted to 150 c.c. and neutralised with ammonia. If lead is present, about 1 gm. of ammonium carbonate is also added, the mixture boiled for 1 minute, filtered, and the precipitate washed thoroughly. The filtrate is heated to expel ammonia, hydrochloric acid added in excess, and then about 15 c.c. of a 10% solution of barium chloride. The mixture is boiled for 10 minutes, allowed to settle, filtered, and the barium sulphate ignited and weighed. This method avoids the formation of globules of free sulphur during the oxidation by preventing heating of the solution. Potassium chlorate may be used instead of the sodium salt and is more stable in nitric acid, but much less soluble in water.—W. F. F.

*Zinc [smelting]; Fine-grinding and porous-briquetting of the charge [in] —.* W. M. Johnson. Amer. Inst. Min. Eng., Feb., 1918. Eng. and Min. J., 1918, 105, 370—371.

A PROCESS is described which reduces the consumption of coal in a zinc-smelting retort. The excess of about 400% above the amount theoretically necessary, which is used at present, is employed to secure a high reducing power, good heat-conductivity, and porosity of charge, and a residue free from slag, and the coal is used in large pieces. In experiments with zinc ore and coal dust ground to 80-mesh fineness, it was found possible to reduce the coal consumption, but the charge was so dense that it obstructed the passage of zinc vapour. Good results were obtained by the addition of broom straw to give porosity. The percentage of coal was reduced successively from 60% to below 30% of the ore, the lower amount effecting a complete reduction of the ore and avoiding the production of slag. The poor conductivity of the finely ground charge is avoided by briquetting the mixture into pieces of about 0.5 in. diameter. Assuming the present cost of zinc ore smelting to be \$17 per ton, it is estimated that a net saving of \$9.47 would be effected by this process.—W. F. F.

*Copper-nickel series of alloys; Potential measurements on the — and some observations on brasses.* N. T. Gordon and D. P. Smith. J. Phys. Chem., 1918, 22, 194—215.

COPPER-NICKEL alloys are shown to be very regular in electrochemical behaviour, and the potential values are reproducible and constant to within 3 millivolts, remaining so for many hours. Alloys containing 6.8, 10.9, 65.5, 74.3, 83.4, and 91.7% Cu were employed, and each



exhibited potential differences against mixed solutions of copper and nickel sulphates, *N/1* in combined strength, which, when plotted against the logarithms of the copper concentration, gave curves of a straight line character, and, with one exception, with a similar slope. Polished surfaces increase the tendency to give ions to the solution, whilst coarser grinding with emery produces small accidental variations from the steady values obtained with annealed surfaces.

Further experiments with Cu-Zn alloys (see *J. Phys. Chem.*, 1916, 20, 228) have shown these to be variable as regards electromotive force, and lend confirmation to suggestions made by other authors (Roberts-Austen, this *J.*, 1898, 214; Carpenter and Edwards, this *J.*, 1911, 133) of unexplained changes to which these alloys are subject. (See also *J. Chem. Soc.*, June, 1918.)—B. N.

*Manganese; Estimation of — in aluminium alloys and dust.* J. E. Clennell. *Eng. and Min. J.*, 1918, 105, 407–410.

THE dust, freed from grease, is dissolved in dilute hydrochloric acid, the solution filtered, and the hot filtrate treated with excess of hot caustic soda to redissolve the alumina. The precipitate containing iron, manganese, copper, and magnesium is filtered off and washed. Separation of the iron from the manganese by ammonia and ammonium chloride was found to be unsatisfactory even after successive redissolving and precipitating. The method of separation by ammonium carbonate described by Fresenius ("Quantitative Analysis," 7th Ed., Vol. I., p. 437) was satisfactory but tedious, and the zinc oxide method described by Low ("Technical Methods of Ore Analysis," 5th Ed., p. 162) was with some modifications found to give a perfect separation of the manganese. The manganese in the pure hydroxide may be estimated by oxidation of oxalic acid (Low, *loc. cit.*), or by Volhard's method depending on the titration of a manganous salt with permanganate (Low, *loc. cit.*, p. 167). The following much simpler method does not involve the separation of the manganese from all other metals. 1 grm. of the sample is heated with 50 c.c. of 50% nitric acid, and finally boiled to expel oxides of nitrogen. The solution is filtered, the residue washed, placed in a nickel crucible, dried, ignited, and the ash covered with sodium peroxide. The mixture is fused and the product dissolved in water and added to the previous nitric acid solution. The mixture is boiled and about 1 c.c. of standard silver nitrate solution added to remove chlorides. 0.1 grm. of lead peroxide is added for each estimated 0.1 grm. of manganese present, the mixture boiled, cooled, made up to 100 c.c., and filtered. 50 c.c. of the filtrate is titrated with standard sodium arsenite. The sodium arsenite solution is made by dissolving 10 grms. of arsenious oxide and 30 grms. of sodium carbonate in boiling water, cooling, and diluting to 1000 c.c.; 62.5 c.c. of this solution is diluted to 1000 c.c.: 1 c.c. of the final standard solution is equivalent to about 0.0025 grm. of manganese. The arsenite solution is standardised against the known manganese content of a standard potassium permanganate solution, or preferably against a standard manganous nitrate solution, obtained by dissolving pure manganous carbonate in nitric acid. Results of tests are given showing that the method is reliable and not affected by the presence of ferric iron or aluminium.—W. F. F.

*Bismuth; Determination of — in lead bullion.* D. W. Jessup. *Eng. and Min. J.*, 1918, 105, 603–604.

FIVE grms. of filings is dissolved in a flask in 60 c.c. of nitric acid (1 : 4); the solution is heated with 15 c.c. of sulphuric acid until fumes are

evolved and then directly over a blast lamp for a few minutes; after slight cooling, 40 c.c. of hot water is added and the solution rapidly decanted through a filter. To the residue are added 10 c.c. of sulphuric acid and 40 c.c. of water. The mixture is agitated and decanted, the residue again heated with 10 c.c. of sulphuric acid over a free flame until dense fumes are evolved, and treated as before. The combined filtrates are partly neutralised with ammonia, precipitated with hydrogen sulphide, the sulphides extracted with sodium sulphide and cyanide in succession, and the residue dissolved in 10 c.c. of nitric acid (1 : 1). The solution is evaporated just to dryness, the residue dissolved in a few drops of hydrochloric acid (1 : 1), and the solution diluted with 500 c.c. of warm water. After standing overnight, it is decanted through a tared filter, the latter washed, and the precipitate weighed as BiOCl. In the bismuth assay of lead ores and slags, the lead sulphate is treated as described above to extract all the bismuth.—W. R. S.

#### PATENTS.

*Iron; Manufacture of — [from black oxide scale].* F. Perry, and Metalloids, Ltd., Tipton. *Eng. Pat.* 114,237, May 18, 1917. (Appl. No. 7184 of 1917.)

SCALE or black oxide of iron is heated in an atmosphere of Mond gas or similar gas, preferably after the latter has been purified from sulphur and hydrocarbon gases. The resulting metal is purer than that from which the scale is originally produced.—T. H. B.

*Grey cast iron; Manufacture of —.* W. B. Hamilton, Sheffield. *Eng. Pat.* 114,328, Mar. 21, 1917. (Appl. No. 4112 of 1917.)

IRON or steel, either solid or molten, is introduced into an acid-lined electric furnace, and limestone, lime or other metallic oxide, and sand are added to form a slag, carbonaceous material being also added, either when charging, or during melting, or to the slag.—T. H. B.

*Steel [heating] furnaces [; Gas-fired —].* The Richmond Gas Stove and Meter Co., Ltd., Warrington, and J. Thompson, Sheffield. *Eng. Pat.* 114,359, May 14, 1917. (Appl. No. 6821 of 1917.)

A FURNACE for heating small articles, *e.g.*, steel cutters, is made of refractory brickwork and is circular in horizontal cross section. Heating burners are arranged tangentially at the upper and lower parts of the chamber. A sliding door is provided at one side, having a central opening to enable the heated articles to be manipulated, and the hot gases to escape. To facilitate exchange of articles to be heated, a central tubular support is provided in the floor of the chamber, into which supporting blocks of various sizes may be fitted.  
—W. F. F.

*Zinc powder; Method of converting — into liquid zinc.* Norsk Elektrisk Metalindustri-Aktieselskap, Sarpsborg, Norway. *Eng. Pat.* 105,558, Apr. 5, 1917. (Appl. No. 4954 of 1917.) Under Int. Conv., Apr. 8, 1916.

ZINC powder is subjected to a rubbing action without the use of fluxes, in a movable, internally heated furnace, *e.g.*, in a rotating electric furnace.  
—T. H. B.

*Copper; Process for tempering and hardening —.* J. A. Morterud, Duluth, Minn., U.S.A. *Eng. Pat.* 114,105, Aug. 27, 1917. (Appl. No. 12,265 of 1917.)

THE copper is heated until it becomes luminous and plunged into a bath containing either 60%



of nitric acid and 40% of vegetable alkali (or caustic potash) by weight, or 75% of nitric acid and 25% of alkali.—T. H. B.

*Copper; Treatment of — for various purposes.* W. T. Howard, Assignor to L. V. Howard, Baltimore, Md. U.S. Pat. 1,257,943, Feb. 26, 1918. Date of appl., June 25, 1915.

COPPER in a finely divided state, obtained by precipitation from a solution of a copper salt by means of fine-grained iron, is washed, and compressed sufficiently to give a mechanical interlocking union between the particles. The product has a lower specific gravity than cast or rolled copper, is soft and highly porous, has a high conductivity for heat and electricity, and marks paper.—T. H. B.

*Metals; Heat treatment of — and apparatus employed therein.* Woodward, Gatty, Saunt, and Co., Ltd., London, and O. Stott, Birmingham. Eng. Pat. 114,217, Apr. 10, 1917. (Appl. No. 4975 of 1917.)

A CHAMBER is divided into a series of superposed compartments to receive the metals to be heat treated, and between successive compartments are intermediate heating spaces. Air, gas, or vapour, or a mixture of these, is supplied to the heating spaces and is circulated through the chamber, passing into the compartments containing the metal through perforations in the roofs and floors. Gradual and uniform heating of the metal is thus effected. The atmosphere within the chamber may be maintained at a pressure slightly below that of the atmosphere.

—T. H. B.

*Refractory metals [tungsten and molybdenum]; Method of working —.* Westinghouse Metal Filament Lamp Co., Ltd., Westminster. From Westinghouse Lamp Co., Bloomfield, N.J., U.S.A. Eng. Pat. 114,282, Nov. 19, 1917. (Appl. No. 17,045 of 1917.)

WIRES of tungsten and molybdenum or other refractory metals are produced by alternate mechanical working and heat treatment of the metal. The mechanical working is carried out at approximately a red heat, and the heat treatment at a higher temperature, e.g., above 1000° C.

—T. H. B.

*Hot-blast stoves used in connection with blast-furnaces.* W. J. Mellersh-Jackson, London. From C. M. Stein et Cie., Paris. Eng. Pat. 114,333, Mar. 24, 1917. (Appl. No. 4292 of 1917.)

BEFORE combustion in a Cowper, Whitwell, or other hot-blast stove, the air or gas, or both, are passed through a continuous regenerating stove heated by waste gases. (Reference is directed in pursuance of Sect. 7, Sub-section 4, of the Patents and Designs Act, 1907, to Eng. Pats. 4536 of 1881 and 10,204 of 1887.)—T. H. B.

*Quicksilver [mercury] ores; Continuous retort for treating —.* W. H. Landers, New Almaden, Cal., Assignor to Pacific Foundry Co., San Francisco. U.S. Pat. 1,256,703, Feb. 19, 1918. Date of appl., Apr. 16, 1917.

A METAL retort is enclosed within a body of masonry provided with a series of fire-boxes, each fire-box being connected with a number of fire-brick flues, the walls of which surround the retort. The material is moved continuously through the retort by a conveyor.—T. H. B.

*Aluminium; Process of metal-plating —.* W. J. Travers, Buffalo, N.Y. U.S. Pat. 1,256,954, Feb. 19, 1918. Date of appl., June 8, 1915.

THE aluminium is cleaned electrolytically by using it as cathode in an alkaline solution, then plated in an electrolyte containing salts of a metal

of the iron group (e.g., a solution of nickel ammonium sulphate acidulated with boric acid), dried, and heated gradually to at least 240° C. to produce an adherence of the electro-plated metal with the aluminium.—T. H. B.

*Alloy; High-speed-cutting-tool —.* G. L. Kelley, Germantown, and A. H. Miller, Ambler, Pa., Assignors to The Midvale Steel Co., Philadelphia, Pa. U.S. Pat. 1,258,227, Mar. 5, 1918. Date of appl., June 26, 1917.

AN alloy containing a preponderating amount of nickel, with not less than 0.05%, preferably 0.5–2%, of zirconium and a quantity of silicon and aluminium, e.g., 2 to 16%, together equal to at least double the amount of the zirconium.—T. H. B.

*Magnesium; [Electrolytic] production of —.* G. O. Seward, Jersey City, N.J., Assignor to American Magnesium Corporation. U.S. Pat. 1,258,261, Mar. 5, 1918. Date of appl., July 3, 1915.

AN alloy of magnesium and tin, or magnesium and copper, is produced by electrolysis a suitable electrolyte containing magnesium, using molten tin or copper cathodes, with respect to which magnesium is more electropositive than it is with respect to aluminium. The magnesium is subsequently dissolved out of the alloy by using it as an anode with a suitable molten electrolyte, and depositing the magnesium at the cathode.—B. N.

*Alkali metals; Process and apparatus for producing — [by electrolysis].* J. Brode, Assignor to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,258,529, Mar. 5, 1918. Date of appl., July 3, 1915.

A FUSED alkali halide is electrolysed, and the alkali metal formed at the cathode is separated from the halogen evolved at the anode by a metallic partition in the form of a hood having a closed top above the cathodes, and a perforated base located opposite the anodes. During the electrolysis the partition is completely covered by the molten electrolyte. The alkali metal formed is drawn off from the cathode space and the halogen evolved in the anode space is drawn off under a reduced pressure.—B. N.

*Steel smelting furnaces; Gas valves for —.* Z. Davies, Llanelly. Eng. Pat. 114,589, Nov. 7, 1917. (Appl. No. 16,273 of 1917.)

*Casting metals and other substances in moulds; Process and apparatus for —.* E. Straus, Munich, Germany. Eng. Pat. 108,154, June 2, 1917. (Appl. No. 7951 of 1917.) Under Int. Conv., July 15, 1916.

*[Ore] concentrating or slimes tables.* M. H. Baker, Bendigo, Victoria, Australia. Eng. Pat. 114,594, Nov. 27, 1917. (Appl. No. 17,536 of 1917.)

*Speller; Condensers for redistilling —.* J. G. Granberg, Beckemeyer, Ill., U.S.A. Eng. Pat. 112,008, Sep. 18, 1917. (Appl. No. 13,405 of 1917.) Under Int. Conv., Dec. 8, 1916.

SEE U.S. Pat. 1,221,045 of 1917; this J., 1917, 556.

*Sulphide ores; Separation of mixed — [by flotation].* C. Faul, St. Kilda, and H. Lavers, Surrey Hills, Victoria, Australia, Assignors to Minerals Separation North American Corporation. U.S. Pat. 1,257,990, Mar. 5, 1918. Date of appl., June 26, 1916.

SEE Eng. Pat. 2974 of 1915; this J., 1916, 605.

*Aluminium; Production of —.* G. Giuliani, Lazzago, Italy. U.S. Pat. 1,257,995, Mar. 5, 1918. Date of appl., Apr. 29, 1913.

SEE Fr. Pat. 456,290 of 1913; this J., 1913, 948.

*Apparatus for testing the hardness of metals.* Eng. Pat. 114,593. See XXIII.

## XI.—ELECTRO-CHEMISTRY.

*Potential measurements on the copper-nickel series of alloys, and some observations on brasses.* Gordon and Smith. See X.

## PATENTS.

*Electrodes of electric furnaces; Holders or fittings for the —.* J. R. Hoyle and P. Fawcett. Sheffield. Eng. Pat. 114,395, Oct. 29, 1917. (Appl. No. 15,722 of 1917.)

A SUBSTANTIALLY air-tight protective casing, formed of a flexible, fire-proof or heat-resisting material, extends round the electrode of an electric furnace, being joined at its upper end to the electrode or its water-cooled holder, and having its lower end resting on, clamped to, or otherwise adapted to cover, the entrance of the electrode through the water-cooled opening in the roof. The flexible casing, made wholly or partly of woven asbestos cloth, may be in tubular sections connected by rings of a metal of low magnetic permeability, or may be pleated or otherwise formed so that it can be shortened or elongated after the manner of a bellows.—B. N.

*Battery. Primary battery.* E. Sokal, Chicago, Ill. U.S. Pats. (A) 1,258,265 and (B) 1,258,266, Mar. 5, 1918. Dates of appl., (A) Nov. 21, 1914, and (B) Oct. 22, 1915.

(A) THE battery comprises a receptacle with a number of porous electrodes arranged in units, with hollow separators interposed between each two units, the separators including porous elements engaging the surface of the electrodes, and non-porous elements provided with a flange in marginal engagement with the porous elements. Means are provided for forcing the electrolyte from the interior of some of the separators in several directions through the pores of the electrodes, and from the other separators into the electrolyte in the receptacle. (B) The battery comprises two electrodes, one of which is non-amalgamated zinc, in an electrolyte comprising a homogeneous solution of a ferric salt adapted to act as a solvent on the zinc, and as a depolariser.—B. N.

*Electric furnaces.* J. L. Dixon, Detroit, Mich., U.S.A. Eng. Pat. 111,104, Oct. 15, 1917. (Appl. No. 14,933 of 1917.) Under Int. Conv., Oct. 14, 1916.

SEE U.S. Pat. 1,214,764 of 1917; this J., 1917, 389.

*Electric furnace.* H. A. Greaves and H. Etchells, Sheffield. U.S. Pat. 1,257,997, Mar. 5, 1918. Date of Appl., Mar. 1, 1917.

SEE Eng. Pat. 106,626 of 1916; this J., 1917, 891.

*Electric storage battery plates and paste therefor; Manufacture of —.* H. Wade, London. From L. H. Flanders, Jenkintown, Pa., U.S.A. Eng. Pat. 114,475, Apr. 4, 1917. (Appl. No. 4886 of 1917.)

SEE U.S. Pats. 1,228,546 and 1,228,547 of 1917; this J., 1917, 891.

*Process and apparatus for producing alkali metals [by electrolysis].* U.S. Pat. 1,258,529. See X.

## XII.—FATS; OILS; WAXES.

*Castor oil; Determination of the purity of —.* Chercheffsky. Ann. Chim. Analyt., 1918, 23, 75—81.

THE method proposed depends on the critical temperature of solution of the oil in ethyl alcohol (sp. gr. 0.8181 at 15° C.). Ten drops of the oil and 40 drops of the alcohol are placed in a tube

10 cm. in length and 6 to 8 cm. in diameter, and the open end of the tube is then sealed hermetically. The tube is placed in an oil or glycerol bath and the temperature of the latter is raised gradually until the curved meniscus between the oil and alcohol layers becomes flat and horizontal. The contents of the tube are now mixed without removing the tube from the bath, and this is allowed to cool slowly. The temperature at which the mixture becomes permanently turbid is noted. In the case of pure castor oil this point lies between 66° and 67° C.; with other oils it is much higher, namely, rape oil, 202°, earlnut (arachis) oil, 197°, cottonseed oil, 172°, linseed oil, 163°, and fish oil, 167° C. The presence of 2% of any of these oils in castor oil raises the turbidity temperature of the latter to 69°—71° C. The author criticises the method described by Frabot (this J., 1918, 98 A), mainly on the difficulty of obtaining petroleum spirit of uniform composition for use in the test.—W. P. S.

*Analysis of cholesterol [compounds].* Lifschütz. See XX.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Pigments; The tinting strength of —.* T. R. Briggs. J. Phys. Chem., 1918, 22, 216—230.

THE power of one pigment to impart its colour to a mixture containing a second pigment is greatly increased if the first pigment coats over and hides the particles of the second one; the smaller the particles of the first are, relatively to the second, the less of the former is required to coat the particles, and consequently the greater becomes the tinting strength of the first pigment. Anything which favours the adhesion of the fine to the coarse particles promotes the coating-over effect, and increases the tinting strength of the first pigment, whilst with particles of the same size and with equal adhesive power to promote the coating tendency, the greater the hiding power the higher is the tinting strength. With proper control of the other factors it is stated that it ought to be possible to estimate hiding power from tinting strength, if the pigment to be tested is very fine compared with the one with which it is mixed, and if it actually coats over and hides it. Other physical properties, besides colour, of mixed powders are dependent very largely upon relative size of particles and the coating-over effect; thus the relative size has an important bearing on the colour of a mixture of two pigments one of which is inert. The coarser the inert pigment is, the larger is the amount which may be added without appreciably affecting the shade of the mixture. This accounts for the fact that ground barytes, silic, and asbestine are used so generally as inert fillers, as each substance consists of particles far larger than those of the majority of the pigments with which they are mixed.—B. N.

*Covering power of pigments with special reference to photographic prints.* Renwick. See XXI.

## PATENTS.

*Sulphide of zinc; Preparation of anhydrous —.* P. Comment, Dijon, France. Eng. Pat. 106,489, May 8, 1917. (Appl. No. 6551 of 1917.) Under Int. Conv., May 16, 1916.

DEHYDRATED, finely powdered zinc sulphate is intimately mixed with sodium sulphate (or potassium sulphate, or a mixture of both) and zinc persulphide in the proportions corresponding to the equation:— $2 \text{ZnSO}_4 + \text{ZnS}_2 + 2 \text{Na}_2\text{SO}_4 = 3 \text{ZnS} + 2 \text{Na}_2\text{SO}_4 + 4 \text{SO}_2$ . The mixture is



calcined at about 800° C., protected from the air, until decomposition of the zinc persulphide is complete, the zinc sulphide washed free of alkali sulphate and dried at 100° to 120° C. The quantity of alkali sulphate may be reduced in proportion to the degree of fineness and the intimacy of the mixture. Instead of anhydrous zinc sulphate, an anhydrous alkali-zinc sulphate may be used, or, during evaporation of a solution of zinc sulphate, sufficient alkali pentasulphide may be added for the formation of the necessary quantity of zinc persulphide. The zinc sulphide obtained is suitable for use in painting.—L. A. C.

*Resins: Method of manufacturing permanently fusible and soluble — from non-fusible phenol-aldehyde resins.* L. Berend, Amöneburg, Germany. U.S. Pat. 1,259,347, Mar. 12, 1918. Date of appl., June 28, 1915.

SEE Eng. Pat. 107,205 of 1916; this J., 1917, 969.

## XV.—LEATHER; BONE; HORN; GLUE.

*Glucose [dextrose]; New galloyl derivatives of — and comparison with chebulic acid.* E. Fischer and M. Bergmann. Ber., 1918, 51, 298—320.

PARTIALLY acylated glucoses may be obtained by the action of acyl chlorides on the acetone condensation products of dextrose in the presence of quinoline or pyridine, the isopropylidene group being removed subsequently by hydrolysis with acids. A trigalloylglucose has now been prepared, starting with glucosemonoacetone, and a galloylglucose from glucosediacetone. The former is a tannin-like mass, but the latter does not show the typical behaviour of tannins towards gelatin and alkaloids.

Chebulic acid, the eutannin of commerce, is a gallic acid derivative which yields dextrose on hydrolysis. Compared with trigalloylglucose, however, the rate of hydrolysis is slow, and for this and other reasons the conclusion is drawn that chebulic acid is not structurally similar to trigalloylglucose. (See further J. Chem. Soc., 1918, i., 224.)—J. C. W.

*Proteins [gelatin]; Ionisation of — and antagonistic salt action.* J. Loeb. J. Biol. Chem., 1918, 33, 531—549.

WHEN powdered gelatin is soaked in a sodium chloride solution of M/8 or M/4 strength, imbibition occurs to a certain extent. If now the gelatin is washed with water, or a weaker solution of a neutral salt of a univalent metal, a large additional swelling of the gelatin takes place. With a weak solution of a salt with a divalent cation (such as calcium) contraction instead of additional swelling occurs. The author explains the observed phenomena by the assumption that the additional swelling is due to ionisation and that a salt with a univalent metal forms an ionisable compound with gelatin, whilst a salt with a divalent metal forms a non-ionisable compound. Antagonistic salt action is due to the transformation of an ionisable into a non-ionisable protein compound. (See also J. Chem. Soc., 1918, i., 240.)—H. W. B.

*Gelatin: Effects of electrolytes on — and their biological significance. II. Effect of salts on the precipitation of acid- and alkali-gelatin by alcohol. Antagonism.* W. O. Fenn. J. Biol. Chem., 1918, 33, 439—451.

THE hindering action of acids and alkalis on the precipitation of gelatin by alcohol (see this J., 1918, 216 A) is reduced when univalent salts like sodium chloride are also present in the gelatin solution. Divalent cations (like calcium) are

antagonistic to alkalis, but not to acids, whilst divalent anions (like sulphate) have the reverse effect. (See also J. Chem. Soc., 1918, i., 240.)  
—H. W. B.

## PATENT.

*Leather: Process of manufacturing —.* A. Barclay, Prahran, Victoria, Australia. Eng. Pat. 114,358, May 4, 1917. (Appl. No. 6391 of 1917.)

DE-HAIRED and bated skins are immersed for 30 mins. to 8 hours, according to their size, in a mixture consisting of saturated magnesium sulphate solution, 1, and water, 2 parts, formalin being added in the proportion of 0.5 oz. per gall. of the mixture. The skins are then drained and tanned.—W. P. S.

## XVI.—SOILS; FERTILISERS.

*Boron; Its effects on crops and its distribution in plants and soils in various parts of the United States.* F. C. Cook and J. B. Wilson. Proc. Amer. Soc. Biol. Chem., Dec., 1917, vi.—vii. J. Biol. Chem., Mar., 1918.

THE authors describe the effects of manure to which a proportion of borax or colemanite (calcium borate) has been added, on various crops grown in different parts of the United States (see this J., 1915, 675; 1916, 319; 1917, 1186). The conclusion drawn is that the absorption by and the toxic effect of boron on plants varies with the variety of plants, the solubility of the boron compound, the amount of the boron compound added to the soil, the time elapsing after the compound is mixed with the soil before planting, the amount of rainfall, etc., and finally, with the character of the soil to which the boron compound is added. Thus, in some soils, 0.0044% boric acid added as borax and 0.0058% added as colemanite had no injurious action on lettuce, spinach, and kale, whilst at another spot 5 miles distant, similar percentages in the soil resulted in a distinct diminution in the crops of these vegetables. Wheat, oats, and rye absorbed only a little boron, whilst leguminous and succulent plants absorbed comparatively large amounts and were adversely affected by relatively small traces of the boron compounds in the soil.—H. W. B.

*Wheat; Effect of sodium nitrate applied at different stages of growth on the yield, composition, and quality of —.* J. Davidson and J. A. Le Clerc. J. Amer. Soc. Agron., 1917, 9, 145—154. Bull. Agric. Intell., 1917, 9, 180—181.

EXPERIMENTAL plots of wheat were treated at different stages with sodium nitrate either in solution or in the solid state, at the rate of 320 lb. per acre. Applied at the early stages of growth, when the plants were about 2 ins. high, the nitrate stimulated vegetative growth and increased the yield. Applied at the time of "heading," it improved the quality of the grain as regards colour and protein-content, without affecting the vegetative growth. Application at the "milk stage" had no effect on the yield, quality, or protein-content of the grain. The same results were obtained whether the nitrate was applied in the solid state or in solution, except in the case of early application, the solution then giving higher yields than the solid, no doubt owing to better distribution in the soil. The experiments confirmed the inverse relation between nitrogen-content and yellow colour of the grain; potassium chloride alone appears to increase this colour, but does not affect either the vegetative growth or the chemical composition of the grain.—J. H. L.

*Oats; Effect of fertilisers on the protein-content of —.* R. W. Thatcher and A. C. Army. J. Amer. Soc. Agron., 1917, 9, 344—348. Bull. Agric. Intell., 1918, 9, 153—156.

FIELD experiments at the Minnesota Agric. Exp. Station, with oats grown in a 3-year rotation of oats, clover, and maize, indicated that nitrogenous fertilisers, and especially sodium nitrate, increase the protein-content of the grain. Potassium chloride appears to have a slight effect in the opposite sense, whilst phosphate fertilisers do not materially affect the nitrogen-content. The average protein-contents of the oats (dry substance) corresponding to the different fertiliser treatments, ranged from 13.4% with potassium chloride, to 15.24% with a "complete" commercial fertiliser. —J. H. L.

*Nitrogen distribution of fibrin hydrolysed in the presence of ferric chloride.* Morrow and Fetzer. See XIXA.

*Nicotine-paraffin emulsion.* Lees. See XIXB.

#### PATENTS.

*Fertilisers; Production of —.* P. Becquevort, London. Eng. Pat. 114,277, Oct. 30, 1917. (Appl. No. 15,812 of 1917.)

THE calcium carbonate occurring in natural phosphates is converted into calcium chloro-ferrite,  $\text{CaCl}_2 \cdot \text{CaO} \cdot \text{Fe}_2\text{O}_3$ , which is not attacked by the acids present in soils. The phosphate is ground and for every 100 parts of calcium carbonate present there are added 111 parts of calcium chloride and 160 parts of ferric oxide; the mixture is formed into briquettes, then heated at  $1000^\circ \text{C}$ ., cooled, and ground.—W. P. S.

*Phosphatic and nitrogenous fertiliser; Process of making a mixed —.* R. F. Gardiner. Clarendon, Va. U.S. Pat. 1,258,106, Mar. 5, 1918. Date of appl., Aug. 6, 1917. Dedicated to the public.

APATITE is ground to pass any mesh between 60 and 200, then heated at about  $900^\circ \text{C}$ . and fused with ammonium sulphate and its dissociation products.—W. P. S.

### XVII.—SUGARS; STARCHES; GUMS.

*Cane sugar; The deterioration of raw —: A problem in food conservation.* C. A. Browne. J. Ind. Eng. Chem., 1918, 10, 178—190.

THE deterioration of raw and semi-refined cane sugars during storage, under the action of micro-organisms, has been recognised to depend on the relation between the moisture and the non-sucrose present in the sample. This relation has been formulated by the Colonial Sugar Refining Co. of Australia, according to the rule that in sugar intended for storage the percentage of moisture must not exceed half that of the non-sucrose. In other words, if  $W$  = the moisture and  $S$  the sucrose, the "factor of safety"  $\frac{W}{100-S}$  must not

exceed 0.333. The author confirms the general validity of this rule, with the modification that for sugars of the Cuban and Porto Rican type, the factor of safety must not exceed 0.30. In cold weather, if the temperature does not exceed  $20^\circ \text{C}$ ., no deterioration will take place, but it will start again when the warm season sets in. Sugar deteriorating in warehouse may lose moisture simultaneously with sucrose, and the action may be concealed for this reason. Cases in which the sugars deteriorate in spite of a low factor of safety are most frequently explainable by an uneven distribution of the moisture, either by the mixing of

different batches or by migration of moisture due to sweating. The danger of bagging sugar direct from the centrifugals has long been recognised. Certain corollaries follow from the "factor of safety" rule. For instance, slight fluctuations in moisture content will have a far greater influence on high-grade sugars than on low-grade products. This is confirmed by practical experience in the storage of high-grade raw sugars and moist refined sugars; it has even been found advantageous to wash the sugars in the centrifugals with molasses instead of with water. In the case of samples stored in closed bottles, the factor  $\frac{W}{100-S}$  will fall

as deterioration proceeds, until a limit is reached where the sugar is stable. In such cases it has been found that the limit factor is nearer 0.25 than 0.3, owing to a residual inverting power after multiplication of the organisms has ceased. Thus a sugar which has been in active deterioration soon after manufacture may arrive at its destination with a safety factor below 0.3, but still be liable to further deterioration. In over 90% of the cases investigated, deterioration follows the normal course, in which polarisation and sucrose diminish and invert sugar increases until the limit of deterioration is reached, the percentage of moisture remaining practically constant. Abnormal fermentations, however, are met with; in some cases there is an increase in polarisation, which is explained by the fermentation of levulose, this being shown by abnormal Clerget values for sucrose, lower than the direct polarisation. In such cases, also, an increase in moisture or other volatile matter is frequently observed. In deterioration in closed bottles an odour of alcohols and esters is usually perceptible; the principal product of the fermentation is carbon dioxide. Cases are on record of spontaneous combustion of sugar cargoes under certain imperfectly understood conditions. The author has made a bacteriological study of the micro-organisms found in raw cane sugars; some of these are harmless towards sucrose, others are destructive. The destruction of sucrose in deteriorated sugar is not due to any single organism or class of organisms; moulds and budding fungi are to be looked for, as well as bacteria. The organisms which cause the inversion of sucrose in raw sugars are unable to thrive in saturated solutions; the washing of raw sugars in the centrifugals, by diluting the films of saturated syrup to a point where inverting organisms can thrive, must be regarded as a leading cause of deterioration. One of the most fertile sources of infection is the water coming from the cooling tower, employed for cooling the warm condensation waters from the factory. This water frequently contains sugar and is highly favourable to the growth of micro-organisms. The use of this water in the centrifugals or the access of spray from the cooling tower to the sugar warehouse is certain to cause trouble. To prevent the deterioration of sugar, methodical care and cleanliness in manufacture and storage, systematic drying of the manufactured sugar, clean and dry warehouses with good ventilation in dry weather and exclusion of moisture in wet weather, are the principal points for attention.—J. F. B.

*Sugar reagents; Copper-phosphate mixtures as —. A qualitative test and a quantitative titration method for sugar [dextrose] in urine.* O. Folin and W. S. McEllroy. J. Biol. Chem., 1918, 33, 513—519.

THE quantitative reagents proposed for the estimation of dextrose in urine are a solution containing 60 grms. of crystallised copper sulphate and 4 c.c. of concentrated sulphuric acid per litre, and a dry mixture containing 100 grms. of



crystallised disodium hydrogen phosphate, 60 grms. of dry sodium carbonate, and 30 grms. of sodium or potassium thiocyanate. To carry out an estimation, 5 c.c. of the copper solution and 4 to 5 grms. of the powder are boiled in a test-tube and the urine run in, a few drops at a time, from a burette with a capillary tube attached, until the solution is colourless. 5 c.c. of the copper solution is equivalent to 25 mgrms. of dextrose (also to 45 mgrms. of anhydrous maltose or 40.4 mgrms. of anhydrous lactose). (See also p. 278 A, and J. Chem. Soc., May, 1918.)—H. W. B.

*Sugar; Note on Cammidge's method for the estimation of [reducing]—* R. W. Garrow. Pharm. J., 1918, 100, 148.

CAMMIDGE has described a modification of Scales' method (this J., 1915, 1264) for the volumetric determination of cupric-reducing sugars in urine, blood, and other physiological liquids. Instead of Fehling's solution, a modified Benedict solution is employed, containing 200 grms. of sodium citrate, 100 of dry sodium carbonate, 10 of sodium bicarbonate, and 21 of crystallised copper sulphate in 1000 c.c. After the reduction, hydrochloric acid is added to dissolve the cuprous oxide; carbon dioxide is thus liberated and protects the cuprous chloride against atmospheric oxidation. The cuprous chloride is added to a known excess of standard iodine solution and the excess of iodine is titrated back with thiosulphate. Each c.c. of N/10 iodine is equivalent to 0.0033 gm. of dextrose. Up to the point when the blue iodo-starch colour is discharged, the solution is perfectly transparent, but immediately after the first end-point is reached a slight opalescence begins to appear, increasing to a white precipitate of cuprous iodide, and the blue colour returns. The first discharge of the blue colour should therefore be taken as the end-point, and the titration should be done as rapidly as possible.

—J. F. B.

*Honey; The ash test for —* H. Stout. Pharm. J., 1918, 100, 147.

THE specification for honey in the British Pharmacopoeia of 1914 reads:—"Ash not more than 0.25%; solution of the ash in water is not alkaline to litmus." In Squires' "Companion to the B.P." (1916), it is pointed out that the requirement of a non-alkaline ash is evidently an error and is likely to exclude genuine honey while favouring an article prepared with starch or invert sugar. This view is confirmed by the author, who has examined several samples of honey of Cuban, Californian, Irish, and Scotch origin, showing ash contents of 0.1–0.14%, and the solutions of the ash in all cases were alkaline to litmus.—J. F. B.

## XVIII.—FERMENTATION INDUSTRIES.

*Beverages; Non-alcoholic carbonated —: sanitary condition and composition.* R. M. Allen, J. O. La Bach, W. R. Pinnell, and L. A. Brown. Kentucky Agric. Exp. Stat., Bull. No. 192, June, 1915. Pp. 65.

A REPORT on the inspection of about 70 factories producing non-alcoholic beverages, with the results of chemical and bacteriological examination of the raw materials and products. Bacterial counts of the waters used and of the bottled beverages, showed that carbonation greatly reduces the number of bacteria, but is not of itself sufficient protection against contaminated water or insanitary conditions of manufacture. The chief sources of infection were the waters used, and the

bottles, the latter having in many cases been very imperfectly cleansed before use. For the detection of bacteria of the *B. coli* group direct plating on Endo fuchsin medium (nutrient agar with lactose, fuchsin, and sodium sulphite added when ready for use) was found a very trustworthy presumptive test, the plates being incubated for 24 hours at 37° C.; the only disadvantage is that considerable care and experience is required in the preparation of the medium to obtain uniform results. Most of the beverages investigated consisted of carbonated water sweetened, flavoured, and coloured in imitation of a fruit. For the flavour of strawberry, raspberry, banana, and pineapple, mixtures of synthetic esters are invariably used, and as the true extracts of lemon, lime, and orange are only slightly soluble in water, terpeneless extracts and imitation flavours are largely used in these cases also. Owing to the slight solubility of oil of ginger, ginger ales are generally fortified with capsicum, which gives the desired pungency and is cheaper. A few non-alcoholic beverages contain actual grape juice, apple juice, or other fruit juice, but in most cases synthetic flavouring matters are used. Those having a vanilla flavour usually contain synthetic vanillin and coumarin. Caffeine is a constituent of another class of beverages, e.g., those of the cola type, made from certain vegetable extracts diluted with carbonated water; in 25 products of this class the proportion of caffeine ranged from 0.012 to 0.268 gm. per 100 c.c., which is considerably less than that present in tea or coffee. Beverages of this class have usually a naturally dark colour; ginger ales, root beers, etc., are coloured with caramel, whilst aniline dyes are generally employed for the beverages of brighter colours. Saccharin and benzoic acid were found in a number of the products and saponin in one of the concentrated foaming ingredients.—J. H. L.

*Wines; Methods for determining the adulteration of —* U. Pratolongo. Staz. Sper. Agrar. Ital., 1917, 1, 315–364. Bull. Agric. Intell., 1918, 9, 109–111.

VARIOUS empirical rules have been suggested, by Gauthier, Halphen, Blarez, Roos, and others, for discriminating between natural and adulterated wines, on the basis of the relation between alcohol-content and acidity. A study of the analytical data for 560 Italian wines showed that all these rules are subject to numerous exceptions. That of Blarez appears to be the most useful, and is recommended in a modified form for the detection of adulteration. For this purpose the author divides natural Italian wines into two categories, viz., those of "normal" and those of "abnormal" composition, and gives for each a table showing the minimum permissible acidity for various alcohol-contents. He proposes that wines of the second category (which constituted about 10% of the total number of wines studied) should be sold only with a declaration of origin which would justify their "abnormal" composition. All wines sold without such a declaration should be regarded as adulterated unless conforming with the table for those of "normal" composition.—J. H. L.

*Wines; Presence of copper in white —* P. Carles. Ann. Falsif., 1918, 11, 43–48.

THE presence of traces of copper (equivalent to 0.015 gm. of crystallised copper sulphate per litre) has been detected in certain white wines. These wines gradually become turbid when bottled, but the turbidity disappears if the wine is kept in an uncorked bottle for some time; it reappears when the bottle is again corked. If the turbid wine is centrifuged, a red sediment of cuprous oxide is obtained.—W. P. S.



*Cider*; Contribution to the study of the alcohols of —. E. Kayser. Bull. Soc. Agric. de France, 1917, 321—323. Bull. Agric. Intell., 1918, 9, 239—241. (See also this J., 1917, 608, 902.)

THREE portions of sterilised apple must were sprinkled with a strong yeast and an apiculated cider yeast and allowed to ferment for three and a half months at 4°—12° C. One portion was then decanted from the yeast deposit into a second sterile flask, the second was similarly decanted into a flask containing another yeast which had already fermented similar must, whilst the third was left undisturbed. After a further period of 2½ months (March—June) the ciders and the alcoholic distillates therefrom were analysed. The results showed that decantation before the secondary fermentation increased the production of aldehydes and higher alcohols, probably owing to aeration. The quantity of higher alcohols plus esters was greatest in the cider which had been decanted on to a new yeast, whilst the volatile acidity was highest in the sample which had been decanted into a clean flask. Although the particular kind of yeast employed is probably a very important factor, the results show that cider for drinking and cider for distilling require different treatment.—J. H. L.

*Horse-chestnuts*; Use of — for the production of alcohol. Kayser. Feuille d'Inform. du Minist. de l'Agric., (France), 1917, 22, 10. Bull. Agric. Intell., 1918, 9, 111—112.

FERMENTATION experiments indicated that 100 kilos. of dry horse-chestnuts (value 12s. 8d. in France) will yield 27—28 litres of alcohol, which, though lower than the yield obtainable from maize, would warrant their industrial use under present conditions (cp. this J., Goris, 1917, 1107; Baker and Hulton, 1917, 1285). They contain about 0.5% of potassium and 0.15% of phosphoric acid, both of which are indispensable yeast foods. —J. H. L.

*Acorns*; Utilisation of — by alcoholic fermentation. Kayser. Feuille d'Inform. du Minist. de l'Agric. (France), 1917, 22, 9—10. Bull. Agric. Intell., 1918, 9, 112.

ACORNS from three varieties of oak (pedunculate, sessile, and holm) found in the Mediterranean district, were freed from their cups, cut into small pieces and heated at 120°—122° C. for half an hour with 2—3 times their weight of water containing 2.5% of hydrochloric or 1% of sulphuric acid. The residual solid matter was separated from the liquid and completely exhausted by crushing and washing with hot water; and the extract, after partial neutralisation with potash, was seeded with grain yeast or cider yeast. Fermentation commenced in 24 hours, or sooner after addition of 1—2 parts per thousand of sweet rye extract. Dilution of the liquid also accelerated fermentation, owing to the fact that acorns contain considerable amounts of tannin which retards fermentation. The yield of alcohol varied, according to the variety of plant, the degree of ripeness, and, above all, the dilution of the extract, from 8.58 to 20.16 litres per 100 kilos. of whole acorns (dried), or from 28 to 31 litres per 100 kilos. of dried kernels. (See also Baker and Hulton, this J., 1917, 1285.)—J. H. L.

*Yeast manufacture*; The amino nitrogen in pressed —. K. Schweizer. Schweiz. Chem.-Zeit., 1918, 2, 26—28.

SOERENSEN'S formaldehyde titration method (this J., 1908, 135) may be usefully applied in the analysis of the mashers for the manufacture of pressed yeast by the aeration process. Ten c.c. of filtered mash or sweet wort is neutralised with N/10 sodium hydroxide to a standard pink in

presence of 6 drops of 1% phenolphthalein solution; 5 c.c. of commercial formaldehyde, similarly neutralised, is added, and the acidity of the amino-acids then determined by titration. Results of several determinations of amino-acids in pure grain mashers and part grain, part molasses mashers would appear to indicate that the concentration of amino nitrogen approximates to an equilibrium, corresponding to about 0.018%, irrespective of the quantity of total nitrogen present during the mashing process. Determinations of the amino-acids, total acidity, and density of the worts showed that a good proteolysis does not necessarily correspond with a good acidification and saccharification. Soerensen's titration method is also applicable to the so-called mineralised worts to which nitrogen is added in the form of ammonium salts. The titration of ammonium sulphate may be made with sufficient accuracy between the limits of 0.5—5.0% concentration.

—J. F. B.

*Yeast-nucleic acid*; Preparation of —. E. J. Baumann. Proc. Amer. Soc. Biol. Chem., Dec., 1917, xiv—xv. J. Biol. Chem., March, 1918.

FRESH brewer's yeast is diluted with water, and the nucleic acid is obtained from the nucleoprotein by treatment with a concentrated solution of sodium hydroxide (100 grms. of alkali per kilo. of yeast). The proteins are removed by precipitation with acetic acid, after partial neutralisation with hydrochloric acid, and the nucleic acid obtained from the filtrate as the insoluble magnesium salt by the addition of magnesium sulphate and hydrochloric acid. The yield is 4 to 7.5 grms. per kilo. of yeast. (See also J. Chem. Soc., May, 1918.) —H. W. B.

*Yeast-nucleic acid*; Structure of —. III. Ammonia hydrolysis. P. A. Levene. J. Biol. Chem., 1918, 33, 425—428.

THE occurrence of uridine-phosphoric acid among the products of hydrolysis of yeast-nucleic acid by dilute ammonia at 115° C. confirms the view according to which yeast-nucleic acid is constructed of four mono-nucleotides containing guanine, uracil, cytosine, and adenine respectively. —H. W. B.

*Optical method for determination of malic and tartaric acids in the same solution.* Willaman. See XXIII.

#### PATENTS.

*Evaporating apparatus [for alcohol]*. C. R. Harris, Los Angeles, Cal. U.S. Pats. (A) 1,258,562 and (B) 1,258,563, Mar. 5, 1918. Date of appl., May 4, 1917.

(A) AN evaporating apparatus for liquids, e.g., alcohol, comprises an evaporating vessel having a central horizontal perforated diaphragm, and an outlet at its upper end. The outlet conduit consists of a series of tubes oppositely inclined alternately, having their ends connected by short vertical tubes, thus forming a zigzag conduit. The inclined portions are filled with fibrous, porous, or granular material to retain entrained water, the vertical connecting pipes being empty. The water returns to the evaporator by gravity. (B) The evaporating vessel is provided with an inlet and outlet for liquid at its lower end, and in its upper part with radial arms supported centrally and carrying vertical evaporating surfaces. A slightly domed baffle-plate is arranged between the top of the evaporating surfaces and the cover of the vessel, and a zigzag discharge pipe is connected centrally in the cover. The baffle-plate is of greater diameter than the set of evaporating surfaces, so that any condensed liquid falling from the outlet pipe is directed beyond the evaporating surfaces.—W. F. F.



*Malt product.* U.S. Pat. 1,256,634. See XIXA.

*Method of manufacturing grape-juice.* U.S. Pat. 1,258,627. See XIXA.

### XIXA.—FOODS.

*Dried milks; Inquiry as to — with special reference to their use in infant feeding.* F. J. H. Coutts. Report to the Local Government Board. Food Reports, No. 24, 1918, 1—137.

THE report deals with the history, methods of preparation, and distribution of dried milk, its physical and chemical characters, its bacteriology, and more particularly its use in infant feeding. It is considered that dried milk is a valuable food and one which possesses certain special advantages; it is easy to transport, keeps for a reasonable period of time without developing an unpleasant flavour, and is economical. Dried milk is not germ-free, but the number of bacteria present is relatively very small as compared with ordinary milk, especially in those varieties of dried milk which have been prepared by passing milk over rollers heated above 100° C.; there is no tendency towards multiplication of the number of bacteria when the milk is kept. The pathogenic bacteria which may have been contained in the original milk are either destroyed or reduced in number and rendered less virulent by the drying process. Dried milk prepared from a mixture of separated milk and an emulsion of some vegetable oil such as cottonseed oil, forms a suitable food for some infants but not for all.—W. P. S.

*Dried milk; Some investigations bearing on the nutritive value of —.* G. Winfield. Report to the Local Government Board. Food Reports, No. 24, 1918, 139—156.

As a result of comparative feeding tests on infants, it is concluded that cows' milk during the process of desiccation loses none of the characters which are necessary for the support of normal growth in children. Experiments with rats proved that dried milk used as a sole food is capable of maintaining an animal in good health.—W. P. S.

*Milk powders; Examination of — at the Government Laboratory.* J. J. Dobbie. Report to the Local Government Board, Food Reports, No. 24, 1918, 157—184.

TWENTY-SIX samples of dried milk prepared from whole milk contained:—Moisture, 1·85 to 6·10; fat, 22·58 to 31·23; proteins, 22·27 to 26·50; hydrated lactose, 33·26 to 41·39; ash, 5·44 to 7·58%. Seven of these samples contained sucrose in quantity varying from a trace to 2·94%. Fifty samples prepared from skimmed milk contained from 0·67 to 3·40% of fat, whilst seven other samples examined had been made from partially skimmed or impoverished milk. A few of these samples were described as "half-cream milk powder," but they had been prepared from milk deprived of 56 to 84% of its fat. In some cases the quantity of ash present was higher than the normal amount due to the presence of very small quantities of such added substances as sodium bicarbonate, alkali phosphate, and calcium oxide combined with sugar. There was no evidence of the presence of foreign fats, but two of the powders contained boric acid and five salicylic acid. With the exception of one sample, which contained annatto, all the milk powders were free from artificial colouring matters. Three of the samples contained 0·08, 0·10, and 0·19%, respectively, of dirt, which consisted of sand, sawdust, vegetable fibres, charred organic substance, etc.; only two samples were quite free from insoluble foreign

matter. Minute traces of copper were present in eleven of the samples. Five samples other than dried milk were also examined; these consisted of milk powder sweetened with sucrose (39·7%); milk powder containing added lactose (6·5%); this sample was stated on the label to contain added sodium citrate, but the quantity of citric acid present did not exceed that found in normal milk); lactose (98·4%), and two protein preparations (76·17 and 75·05% of proteins, respectively).—W. P. S.

*Milk; Estimation of lactose in —.* O. Folin and W. Denis. J. Biol. Chem., 1918, 33, 521—524.

*Titration method:* By employing the reagent and apparatus described for the estimation of dextrose in urine (see page 275 A), the lactose in milk may be determined without previously removing either the protein or the fat from the milk. It is advisable to dilute the milk 1:4 (25 c.c. in a 100 c.c. flask) for cow's milk and 1:5 (5 c.c. in a 25 c.c. flask) for human milk. The titration is made as follows:—2·8 to 3·4 c.c. of the diluted milk (that is, nearly the full amount expected), 5 c.c. of the copper sulphate solution, 4 to 5 grms. of the dry salt mixture, and a pebble (to prevent bumping) are introduced into a large test-tube, shaken, and boiled gently for 4 minutes. Then a few drops more of the diluted milk are added, and the mixture boiled again, the addition of the diluted milk being continued, boiling for 1 minute after each addition, until the liquid is just colourless. The total boiling period should be 5 to 7 minutes. The volume of diluted milk required to bring about complete decolorisation contains 40·4 mgrms. of lactose.

*Colorimetric method:* This is a modification of Dehn and Hartman's method (this J., 1914, 271). A 100 c.c. volumetric flask is half filled with saturated picric acid solution and then 2 c.c. (accurately measured) of the milk is added. The volume is brought to 100 c.c. with saturated picric acid solution and the flask is then shaken and the contents filtered. 5 c.c. of the filtrate is transferred to another 100 c.c. flask, 15 c.c. of saturated picric acid solution and 3 c.c. of 20% sodium carbonate solution are added, and the mixture heated in a boiling water bath for 15 minutes. After cooling, the liquid is diluted to 100 c.c. The colour is then compared with a similar solution prepared at the same time from 20 c.c. of a standard 0·025% solution of lactose in saturated picric acid solution and 3 c.c. of sodium carbonate solution, subsequently diluted to 100 c.c. Accurate results are obtained only when the standard and unknown solutions are heated simultaneously and the colour values of the two solutions do not differ by more than 20%. For use with human milk, a second standard containing 0·035% of lactose in saturated picric acid solution is recommended.—H. W. B.

*Butyrometers; Graduation and calibration of Gerber new milk —.* F. E. Day and M. Grimes. Analyst, 1918, 43, 123—131.

THE volume of a 1% scale division on the tube should be 0·1240 c.c.; the capacity of the body of the tube, from the body end of the scale to the inner end of the neck, should be 20·5 to 21·5 c.c., and the milk pipette should deliver 11 grms. of water at 15° C., equivalent to 11·18 to 11·30 grms. of milk. The correct temperature for reading the volume of the fat in a determination is about 55° C.

—W. P. S.

*Condensed milk; Determination of the acidity of —.* M. Dugardin. Ann. Chim. Analyt., 1918, 23, 83—84.

TEN grms. of the sample is diluted with 25 c.c. of water free from carbon dioxide and the solution

is titrated with *N*/10 sodium hydroxide solution, using phenolphthalein as indicator. Wholesome condensed milk, whether sweetened, unsweetened, or skimmed, does not contain more than 0.5% of acidity, calculated as lactic acid.—W. P. S.

*Wheat flour; Non-protein nitrogen in* —. M. J. Blish. *J. Biol. Chem.*, 1918, 33, 551—559.

THE ordinary protein precipitants do not satisfactorily remove gliadin and the other vegetable proteins from an aqueous extract of wheat flour. For the estimation of non-protein nitrogen in flour the author recommends the preparation of an extract obtained by shaking 20 grms. of flour with 400 c.c. of water for 2 hours and then filtering through paper. The proteins are precipitated by treating 100 c.c. of the extract in a 200 c.c. flask with 25 c.c. of *N*/10 sodium hydroxide followed by 27 c.c. of *N*/10 copper sulphate solution, shaking vigorously until the precipitate settles rapidly, making up to the mark, filtering, and estimating the nitrogen in 100 c.c. of the filtrate by Kjeldahl's method. A portion of the filtrate may also be concentrated to 1/20th of its volume and the amino-nitrogen estimated by Van Slyke's method. Wheat flour contains about 0.030% of non-protein nitrogen, of which about 0.002% consists of  $\alpha$ -amino-nitrogen and 0.006% of amide-nitrogen. The nature of the remaining 0.022% of non-protein nitrogen has not been determined.—H. W. B.

*Flour; The soluble nitrogen as a factor in determining the value of* —. E. Rousseaux and M. Sirot. *Ann. Falsif.*, 1918, 11, 38—39.

THE authors have shown previously (this *J.*, 1918, 134A, 163A) that the ratio of total nitrogen to soluble nitrogen in flour affords an indication of the quality of flour for baking purposes; they now propose to use this ratio inversely, i.e., soluble nitrogen to total nitrogen. In the case of normal flour this value will be 0.175, or expressing the total nitrogen as 100, the ratio will be 17.5. For inferior flours the ratio will be more than 19, for maize, 18.1, rye, 22.7, beans, 12.1, barley, 12.1, and rice, 4.27.—W. P. S.

*Eggs; Photographic method for the examination of* —. G. A. Le Roy. *Ann. Falsif.*, 1918, 11, 10—18.

THE method consists in photographing the eggs directly, whilst illuminated by an arc or ordinary filament lamp. Before the method can be used practically it will be necessary to obtain more data regarding the increase in the volume of the air chamber in eggs on keeping and the effect of temperature, moisture, pressure, etc. on the same. The method appears to be useful for the examination of eggs preserved in cold-store, but would probably be difficult to apply to eggs pickled in sodium silicate, paraffin wax, lime-water, etc.—W. P. S.

*Potatoes; Alleged poisoning by* —. F. W. Harris and T. Cockburn. *Analyst*, 1918, 43, 133—136.

SIXTY-ONE cases (one fatal) of poisoning occurred recently in Glasgow; all the persons affected had partaken of potatoes obtained from one source. These potatoes had commenced to sprout and contained 0.041% of solanine, which was at least five or six times the quantity found in normal, unsprouted potatoes. This, in conjunction with the medical evidence, proves that the poisoning was caused by the excessive quantity of solanine present in the potatoes.—W. P. S.

*Cocoa; Alkalised* —. Rocques and Touplain. *Ann. Falsif.*, 1918, 11, 19—26.

ACCORDING to French law the quantity of added alkali in cocoa must not exceed 5.75%, calculated as potassium carbonate and expressed as a percentage of the dry, fat-free material; 2.75% of alkali is taken as being the natural alkalinity of cocoa, so the total alkalinity of an alkalised cocoa must not exceed 8.5%. Of 25 samples of cocoa examined by the authors, 7 contained more than 2.75% of alkali (as potassium carbonate), the largest quantity found being 3.23%, and the suggestion is made that the allowance for the alkali-content of natural cocoa should be increased to, say, 3.50%. French law also requires alkalised cocoa to have a slightly acid reaction, that is, the quantity of alkali added must not be sufficient entirely to neutralise the acidity of the cocoa.

—W. P. S.

*Sorghum kernels; Composition of grain* —. J. A. Le Clerc and L. H. Bailey. *J. Amer. Soc. Agron.*, 1917, 9, 1—16. *Bull. Agric. Intell.*, 1918, 9, 156—158.

COMPARISON of numerous data relating to six varieties of grain sorghum led to the conclusion that there is no well-defined relation between the thousand-corn weight and the protein-content of the grain. Low protein-content is usually accompanied by high bushel-weight and low contents of fibre, ash, and pentosans. Large corns have often less than the average proportion of fibre, and therefore ash, doubtless because their surface is relatively small in proportion to their volume.

—J. H. L.

*Feeding stuffs; Composition of some Indian* —. J. N. Sen. *Agric. Research Inst., Pusa, Bull.* No. 70, 1917, 1—60.

TABLES are given showing the composition of feeding stuffs available in India; in many cases, some dozens of analyses of different samples of a material are recorded. The fodders analysed include earthnuts, oats, colza, mustard, rape, pigeon pea, tea seed, safflower, chick-pea, coconut, sunn-hemp, cluster bean, zedoary, yams, horsegram, rocket, buckwheat, soya bean, cottonseed grass, hay, niger seed, sunflower, barley, vetches, lentil, linseed, lucerne, senji, rice, millet, guinea grass, poppy, Burma bean, green gram, field pea, sugar cane, sesamé, clover, wheat, cow pea, maize, ginger, etc.—W. P. S.

*Fibrin hydrolysed in the presence of ferric chloride; The nitrogen distribution of* —. C. A. Morrow and W. R. Fetzer. *Soil Sci.*, 1918, 5, 163—167.

THE results indicate that when a protein is hydrolysed in the presence of ferric chloride an accurate determination of the nitrogen distribution cannot be effected. Much of the recent work on the organic nitrogen distribution in soils by the Van Slyke method is thus quite unreliable. (See also *J. Chem. Soc.*, 1918, i., 218.)—W. G.

*Ionisation of proteins and antagonistic salt action.* Loeb. *See XV.*

*Effects of electrolytes on gelatin and their biological significance. Effect of salts on the precipitation of acid- and alkali-gelatin by alcohol.* Fenn. *See XV.*

*Effect of sodium nitrate applied at different stages of growth on the yield, composition, and quality of wheat.* Davidson and Le Clerc. *See XVI.*

*Effect of fertilisers on the protein-content of oats.* Thatcher and Arny. *See XVI.*

*Foam inhibitor in the Van Slyke amino nitrogen method.* Mitchell and Eckstein. *See XXIII.*



*Optical method for the determination of malic and tartaric acids in the same solution.* Willaman. See XXIII.

#### PATENTS.

*Fish, meat, fruit, and the like; Drying of* — by vacuum. A. S. Myrens Verksted, Christiania, Norway. Eng. Pat. 105,072, Mar. 20, 1917. (Appl. No. 4070 of 1917.) Under Int. Conv., Mar. 18, 1916.

THE material is placed in bags made of rubber or other waterproof substance which are immersed in water heated to a suitable temperature, and the interior of the bags is connected with a suction device.—W. P. S.

*Malt product [for bakers' use].* H. G. Akers and W. P. Kaufmann, Assignors to Malt Products Co. of Canada, Ltd., Toronto, Canada. U.S. Pat. 1,256,634, Feb. 19, 1918. Date of appl., Apr. 10, 1916.

THE product consists mainly of malt together with from 15 to 30% of the soluble constituents of malt combs, the latter being added as a denaturing ingredient to render the product unsuitable for the production of potable alcoholic liquors.—W. P. S.

*Coffee-like or tea-like beverages and products; Method of producing* —. A. Bjørcke, Christiania, Norway. U.S. Pat. 1,257,451, Feb. 26, 1918. Date of appl., May 29, 1916.

THE rinds of citrus fruits are heated at 180° C. to expel water and light oils, then at 215° C. to separate aromatic oils, fatty acids, and other organic substances, and the residue is ground.—W. P. S.

*Fodder; Process for preserving green* — in the form of sweet green fodder. J. R. Sonderegger, Nesslerau, A. Messmer, Rapperswil, and F. Erdreich, Zürich, Assignors to C. Reinhardt, Berne, Switzerland. U.S. Pat. 1,257,754, Feb. 26, 1918. Date of appl., Dec. 19, 1917.

AIR-DRIED fodder is stacked in successive layers, allowing each layer to become heated to 60°–65° C., by fermentation, before the next layer is added. When the top or last layer has been added and allowed to heat, pressure is applied, first to the central portions of the layers and then extended to the outside portions of the stack.—W. P. S.

*Corn [maize]; Process for treating* —. A. Woolner, jun., Peoria, Ill. U.S. Pat. 1,258,076, Mar. 5, 1918. Date of appl., Nov. 22, 1916.

MAIZE is steamed to swell the grains, the hulls and germs are loosened mechanically, the whole mass is dried, and reduced by gradual grinding, the hulls and germs being separated by sifting and aspiration from the starchy material.—W. P. S.

*Desiccating liquids [buttermilk]; Process of* —. J. C. MacLachlan, Chicago, Ill. U.S. Pat. 1,258,348, Mar. 5, 1918. Date of appl., July 11, 1914.

THE coagulable material in a liquid, such as buttermilk, is slightly coagulated, i.e., precipitated in a light flocculent form, and then removed and the remaining liquid is partially evaporated. The residual concentrated liquid is then mixed with the previously separated coagulated material and the mixture is sprayed into a chamber through which heated air is passed. The remaining liquid is evaporated and the resulting dried product is separated from the air.—W. H. C.

*Soya bean food.* M. Makino, San Francisco, Cal. U.S. Pat. 1,258,427, Mar. 5, 1918. Date of appl., Dec. 5, 1916.

SOYA bean flour is mixed with water and a small

quantity of an edible oil, the mixture is heated, filtered, and the proteins in the filtrate are precipitated and separated from the liquid.—W. P. S.

*Grape-juice; Method of manufacturing* —. P. R. Welch, Westfield, N.Y. U.S. Pat. 1,258,627, Mar. 5, 1918. Date of appl., Aug. 4, 1917.

THE pulp and juice of grapes, after removal of the seeds, is stored until the argol crystallises; the argol is then separated, and the juice in turn is separated from the remaining pulp.—W. P. S.

*Food products [from potatoes]; Method of preparing pre-cooked* —. B. Remmers, Philadelphia, Pa., Assignor to Armour Grain Co., Chicago, Ill. U.S. Pat. 1,258,047, Mar. 5, 1918. Date of appl., Apr. 21, 1917.

SEE Eng. Pat. 113,900 of 1917; this J., 1918, 256 A.

*Fruits, vegetables, grain, and other organic substances; Process and apparatus for preserving* —. S. A. Kapadia, London. U.S. Pat. 1,259,410, Mar. 12, 1918. Date of appl., Oct. 5, 1914.

SEE Eng. Pat. 9916 of 1914; this J., 1916, 903.

#### XIXB.—WATER PURIFICATION; SANITATION.

*Carbon dioxide; Gasometric determination of combined* —. W. Mestrezat. Ann. Chim. Analyt., 1918, 23, 45–47.

CARBONATES in a solution also containing hypochlorites (e.g. alkaline hypochlorite antiseptics) may be determined in an ordinary calcimeter if a tube containing a plug of cotton wool soaked in oil of turpentine is inserted between the reaction flask and the gas measuring burette. The chlorine liberated when the acid is added is absorbed by the turpentine whilst the carbon dioxide passes over into the burette.—W. P. S.

*Nicotine-paraffin emulsion.* A. H. Lees. J. Bd. Agric., 1918, 24, 1411–1417.

AN insecticide containing soft soap, 20, nicotine, 0.5 lb., paraffin, 2, and water, 100 gall., was found to be very effective against capsids, cabbage caterpillars, gooseberry sawfly (larvæ), raspberry and loganberry beetle (*Byturus tomentosus*), and aphides. The mixture may be given fungicidal properties by adding to it liver of sulphur, sodium sulphide, or ammonium sulphide (see Barker and Lees. J. Bd. Agric., Mar., 1916) but not lime-sulphur, Bordeaux, or Burgundy mixtures, since these latter combine with the soap. As nicotine sulphate is not volatile, it should not be used in place of nicotine in preparing the mixture.—W. P. S.

*Insect eggs; Toxicity of volatile organic compounds to* —. W. Moore and S. A. Graham. J. Agric. Res., 1918, 12, 579–587.

POTATO-BEETLE eggs were used for the experiments, clusters of 20–30 eggs being treated as a unit. The effects of a large number of compounds were studied by dipping the eggs therein for periods of 1–120 sec.; by spraying the eggs by means of an atomiser; and by fumigating the eggs for 15 hrs. with varying amounts of the compounds, and then in each case exposing them to the air till hatched or proved dead. It was found that slightly volatile compounds with high boiling points, such as trimethylene cyanide, nicotine, nitrobenzene, were more effective in dipping and spraying the eggs than compounds with high volatility and low boiling points, such as ether, alcohol, benzene. The latter compounds killed recently-laid eggs more easily than eggs in which the embryo was partially or fully developed, whereas the reverse was the case with the compounds of high boiling point. Kerosene was toxic to both

kinds of eggs, the higher boiling fractions being in general more effective than the lower. In the fumigation experiments it was found that toxicity was inversely proportional to the volatility; *e.g.*, ether, methyl alcohol, and acetone were very much less toxic than nicotine, nitrobenzene, and furfural. (See also this J., 1917, 939, 1146.)

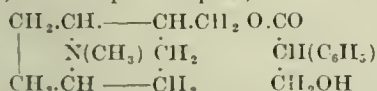
—J. H. J.

*New general method for determining iodine in inorganic and organic compounds.* Tarugi.  
See XXIII.

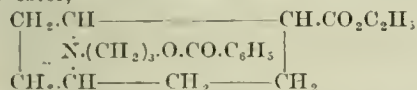
XX.—ORGANIC PRODUCTS; MEDICINAL  
SUBSTANCES; ESSENTIAL OILS.

*Cocaine: Conversion of — into new, physiologically-active substances.* J. von Braun and E. Müller. Ber., 1918, 51, 235—252.

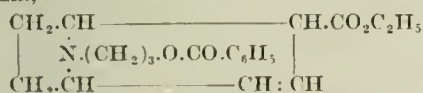
THE controlling factor in the physiological activity of the tropeines is the presence of an acyloxy-group in the  $\gamma$ -position with regard to the nitrogen atom, cocaine being modified further by an ester group in the  $\beta$ -position. The authors have studied the effect on the physiological activity of displacing the acyloxy-group to another place in the molecule, still in the  $\gamma$ -position with regard to the nitrogen atom. Cocaine has been converted into two compounds which fulfil these conditions, namely, homotropine tropate,



and N- $\gamma$ -benzoyloxypropyldihydronoregonidine ethyl ester,



These are both oils which form salts with the usual reagents for bases, and the former is as effective a mydriatic as atropine and the latter as powerful a local anæsthetic as cocaine. It follows, therefore, that it does not matter where the acyloxy-group is with regard to the tropane ring as long as it is in the  $\gamma$ -position with respect to the nitrogen atom. Another interesting product has the formula.



and is designated "ecaine." It is also an oil, and is superior to cocaine as a local anæsthetic, not only in actual power, but in being non-toxic, and sterilisable (see also Ger. Pats. 296,742 and 301,870, of the Chem. Werke Grenzach). (See further, J. Chem. Soc., 1918, i., 234.)—J. C. W.

*Cryptopine*; *Salts of*——. H. E. Watt. Pharm. J., 1918, 100, 147.

CRYPTOPINE gives a deep blue colour with sulphuric acid, whereas pure morphine should give no colour. The hydrochloride, nitrate, and sulphate of cryptopine possess the peculiar property of forming solid jellies in dilute aqueous solution. A 3% solution of the hydrochloride in warm water sets to a firm transparent jelly on cooling. A similar solution of the nitrate gives a translucent jelly, whilst that of the sulphate forms an opaque white jelly resembling curds. Stronger solutions, *e.g.*, a 10% solution of hydrochloride, do not gelatinise on cooling but solidify to a crystalline mass. (See also this J. 1916, 1177.)—J. F. B.

*Filicine and filicic acid*; *Determination of* — in  
extract of male fern. Perrin. Ann. Chim.  
Analvt., 1918, 23, 55—57.

Five grms. of the extract is dissolved in 40 c.c. of ether and the solution is shaken with 100 grms. of 3% barium hydroxide solution; after settling for 10 mins., the aqueous layer is drawn off, filtered, 86 grms. of the filtrate is acidified slightly with hydrochloric acid, and extracted four times with ether (40, 30, 20, and 15 c.c.). The ethereal extracts are filtered, evaporated, and the residue dried at 100° C. for 2 hours, and weighed. The weight obtained is multiplied by 25 to obtain the percentage quantity of crude filicine present. The extract should contain from 24 to 25% of crude filicine. To determine filicic acid, the dry crude filicine is treated with 2 c.c. of amyl alcohol; after 24 hours, 20 c.c. of pure methyl alcohol is added, at first drop by drop until a permanent precipitate is formed, and the remainder then poured in. The precipitate is collected after 24 hours, washed twice with 5 c.c. of methyl alcohol, dried at 100° C., and weighed. Extract of male fern should contain from 3.5 to 9% of filicic acid.

—W. P. S.

*Cholesterol* [compounds]; *Analysis of*——. J. Lifschütz. Z. physiol. Chem., 1918, 101, S9—98.

THE author describes a method for quantitatively separating the cholesterol or similar lipid from the digitonin-cholesteride complex resulting from the application of Windaus' digitonin method for the estimation of cholesterol. (See further J. Chem. Soc., 1918, ii., 179.)—H. W. B.

*Histidine; Detailed method for the preparation of*  
——. H. M. Jones. J. Biol. Chem., 1918, **33**,  
429—431.

FULL directions are given for the preparation of histidine from ox-blood corpuscles. (See also J. Chem. Soc., 1918, i., 232.)—H. W. B.

*Taurine; Preparation of* — in large quantities.  
C. L. A. Schmidt and T. Watson. J. Biol.  
Chem., 1918, **33**, 499—500.

A METHOD is described for preparing taurine from the muscle of the abalone, *Haliotis*, which occurs in abundance on the Pacific Coast of America. (See also J. Chem. Soc., June, 1918).—H. W. B.

*Catechol and quinol*; Occurrence of ——. E. O. von Lippmann. Ber., 1918, 51, 272.

DURING the long spell of dry weather at the end of the summer of 1917, the author found deposits of catechol on the inner surface of bark cast by some old plane-trees, and quinol sticking to the viscous exudations of some grafted pear-trees.

—J. C. W.

—J. C. W.

*Spearmint; Influence of time of harvest, drying, and freezing of—upon the yield and odorous constituents of the oil.* F. Rabak. J. Ind. Eng. Chem., 1918, 10, 275—279.

THE yield of oil obtainable from spearmint varies with the season and with the period of growth. It is highest during flowering, and the tops of the plant then contain the highest proportion of oil. The effect of drying is to reduce the yield of oil and to increase the proportion of alcohols and esters in the oil. A similar tendency to increased formation of alcohols and esters is observed as the plant matures, whilst freezing of the plant promotes the formation of the odoriferous constituents.—C. A. M.

*Alcohol and soda-lime.* B. H. Carroll. J. Phys. Chem., 1918, **22**, 128—149.

ALCOHOL vapour is decomposed when passed over soda-lime heated at  $250^{\circ}$  to  $450^{\circ}$  C. with the



formation of sodium acetate:  $C_2H_5OH + NaOH = CH_3CO_2Na + 2H_2$ . The acetate begins to decompose at the lower temperature mentioned, and the velocity of this secondary change increases so rapidly with the temperature that at  $450^\circ C$ . the acetate is decomposed almost as quickly as it is formed:  $CH_3CO_2Na + NaOH = Na_2CO_3 + CH_4$ . Aldehyde tars, ethylene, and water are formed as by-products, but the proportion of these is small. The same reaction occurs when the alcohol is passed over heated caustic soda, but lime has very little effect. It is supposed that the caustic soda acts as a catalyst, and that the first stage in the reaction is the formation of acetaldehyde, which then decomposes further. In support of this view, it has been found that sodium acetate is also formed when acetaldehyde vapour is passed over heated soda-lime. (See also J. Chem. Soc., 1918, i., 211.)—H. M. D.

*Acetone and lime.* M. E. Freudenheim. J. Phys. Chem., 1918, 22, 184—193.

WHEN acetone is passed over slaked lime heated to temperatures varying from  $350^\circ$  to  $630^\circ C$ ., methane, hydrogen, carbon monoxide, ethylene, and carbon dioxide are produced. The percentages of methane, carbon monoxide, and ethylene, calculated on the total gas evolved, rise from 10%, 0%, and 1% respectively at  $350^\circ C$ . to 54%, 23%, and 5% at  $630^\circ C$ ., whilst the hydrogen falls from 78% at  $350^\circ C$ . to 10% at  $630^\circ C$ . The rate of evolution of the gas increases from 5 c.c. at  $350^\circ C$ . to 170 c.c. per minute at  $630^\circ C$ .; very little carbon is deposited in the heated tube at lower temperatures, but the amount rapidly increases at higher temperatures. It is stated that acetone may be an intermediate product when calcium acetate and lime are heated together. The production of methane and hydrogen only at  $350^\circ C$ . has not been satisfactorily explained, but it is noted that sodium acetate and soda-lime when heated together yield hydrogen in addition to methane. The formation at higher temperatures of the gases enumerated is accounted for by the possible production of keten,  $CH_2CO$ , and the dissociation of acetone and keten separately into methane, hydrogen, carbon monoxide, and ethylene. (See also J. Chem. Soc., June, 1918.)—B. N.

*Antimony; The chemotherapeutics of —.* M. Tsuzuki. Schweiz. Chem.-Zeit., 1918, 2, 17—22, 36—40.

JUST as quinquivalent arsenic compounds, though far less poisonous than the trivalent derivatives, are without therapeutic value, so quinquivalent antimony compounds have been found to be similarly inert; in fact a biological method has been proposed for determining the valency of antimony in its compounds in this way, although a few exceptions to the rule exist. An outstanding difference between the trivalent compounds of antimony and arsenic lies in the observation that with antimony no immunity due to acclimatisation is acquired, either by the blood parasites or by the animal organism, whereas in the case of arsenic immunity towards increasing doses is a characteristic property. The toxicological properties of antimony are very similar to those of arsenic: as a rule, antimony compounds which are insoluble in water have little or no toxic properties. Ranken (Roy. Soc. Proc., 1913, B 86, 203) has studied the effect of intravenous injection of antimony on trypanosomes circulating in the blood. These are all killed within 20 minutes. At the same time a considerable percentage of the leucocytes is also destroyed, but a stimulus is set up which leads temporarily to an overproduction of leucocytes until equilibrium is again established. The therapeutic properties of corresponding derivatives of antimony and arsenic show no

parallelism either in character or degree. Many antimony compounds show a specific difference in their action on trypanosomes and spirochaetes, being decidedly more active towards the former. Such pronounced selective activity is not found among arsenic compounds. Observations made on arsenic compounds with regard to the influence of the constitution of the organic portion of the molecule on therapeutic or toxic properties are not valid for the corresponding antimony compounds. Arsacetin is very slightly active but the corresponding antimonoacetin (stibacetin) is strongly active; salvarsan is very strongly active but "antimony salvarsan" is quite inactive. These differences in the relation between constitution and activity of similar compounds may be due to the greater ease with which antimony is split off from its organic derivatives, so that the specific properties of the organic state of combination are more readily lost in the organism. For the same reason it is more difficult to suppress the poisonous properties of antimony by organic combination. The organic derivatives of bismuth are still less stable. On the other hand, the complex salts of metals of the arsenic group increase in stability with the atomic weight of the metal, and in this direction new valuable therapeutic agents may be looked for. Certain combinations of arsenic and antimony derivatives have shown useful results.—J. F. B.

*Preparation of yeast-nucleic acid.* Baumann. See XVIII.

*Structure of yeast-nucleic acid.* Ammonia hydrolysis. Levene. See XVIII.

*New general method for determining iodine in inorganic and organic compounds.* Tarugi. See XXIII.

*Optical method for the determination of malic and tartaric acids in the same solution.* Willmann. See XXIII.

#### PATENTS.

*Acetic anhydride and polymerised or non-polymerised acetic aldehyde; Process for the production of —.* Soc. Chim. des Usines du Rhône, Paris. Eng. Pat. 110,906, June 19, 1917. (Appl. No. 8810 of 1917.) Under Int. Conv., Oct. 30, 1916.

THE decomposition of ethylidene diacetate into acetaldehyde and acetic anhydride (see Eng. Pat. 23,190 of 1914; this J., 1915, 982) can be effected almost quantitatively, without formation of tar, by heating in a vacuum with the addition of an acid or acid salt as catalyst. The acetaldehyde which is produced distils in the form of paraldehyde. —D. F. T.

*Pharmaceutical products [bromodiethylacetylurethanes of N-acetylated-p-aminophenols]; Manufacture of new —.* P. A. Newton, London. From Farbenfabr. vorm. F. Bayer und Co., Leverkusen, Germany. Eng. Pat. 114,680, Apr. 23, 1917. (Appl. No. 5669 of 1917.)

p-ACETYLAMINOPHENOL, 453 parts, suspended in 1500 parts of acetone, is treated with 600 parts of bromodiethylacetylurethane until the mixture is dissolved. After standing several hours, the precipitate is filtered off, washed with ether, and recrystallised from alcohol. Alternatively, 213.5 parts of bromodiethylacetyl chloride is added slowly to a mixture of 194 parts of p-acetylaminophenolurethane and 121 parts of dimethylaniline, the mass being heated for a short time at  $70^\circ C$ . to complete the reaction; 500 parts of acetone is subsequently added and the product separated. The bromodiethylacetylurethane of

*p*-acetylaminophenol thus obtained melts at 144° C. Compounds of this group are useful in pharmacy as nervines and sedatives.—J. F. B.

*Mercury compounds of the acridine series and process of making same.* P. Hüsey and M. Hartmann. Assignors to Society of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,259,517, Mar. 19, 1918. Date of appl., Nov. 28, 1917.

YELLOWISH-BROWN powders of value as therapeutic agents and antiseptics are prepared by the action of a soluble mercury salt on an acridine dyestuff alkylated at the characteristic nitrogen atom; these products give yellowish-green solutions in water, and solutions in alcohol, acetic acid, and acetone which at great dilution show yellowish-green fluorescence, and solutions with intense green fluorescence in sulphuric acid. The mercury compound obtained from 3,6-diamino-2,7-dimethyl-acridine, which has been previously heated with methyl chloride under pressure, is particularly specified.—D. F. T.

*Glycols; Manufacture of.*—*Manufacture of glycols and glycol derivatives.* P. McElroy, Washington, D.C. Assignor to Chemical Development Co. U.S. Pats. (A) 1,259,757 and (B) 1,259,758, Mar. 19, 1918. Dates of appl., Jan. 25, 1917, and Mar. 29, 1913.

(A) A MIXTURE of ethylene-, propylene-, and butylene-glycols is obtained by chlorinating oil-gas which has been heated to about 700° C. and hydrolysing the resulting mixture of dichlorides by heating under pressure with water and calcium carbonate. (B) A mixture of alkylene halides prepared from oil-gas is heated under pressure with an aqueous solution of a suitable salt of a weak acid, e.g., calcium acetate.—D. F. T.

*Opium; Manufacture of an improved preparation of.* O. Inray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 114,190, Mar. 20, 1917. (Appl. No. 4058 of 1917.) SEE U.S. Pat. 1,243,729 of 1917; this J., 1918, 39 A.

*Bromodiethylacetylurethanes.* J. Callsen, Elberfeld, Germany. Assignor to Synthetic Patents Co., New York. U.S. Pat. 1,256,293, Jan. 31, 1917. Date of appl., Feb. 12, 1918.

SEE Eng. Pat. 114,680 of 1917; preceding.

*Stibinic acids; Manufacture of aromatic.*—C. Philipp and H. Schmidt. Assignors to Chem. Fabr. von Heyden A.-G., Radebeul, Germany. U.S. Pat. 1,260,707, Mar. 26, 1918. Date of appl., July 16, 1912.

SEE Eng. Pat. 16,350 of 1912; this J., 1913, 109.

*Esters [methyl acetate]; Manufacture of.*—A. Helbrouner and G. E. Criquebeuf, Paris. U.S. Pat. 1,260,977, Mar. 26, 1918. Date of appl., Jan. 15, 1914. Renewed Jan. 30, 1918.

SEE Fr. Pat. 464,646 of 1913; this J., 1914, 502.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Pigments; Covering power of—*with special reference to photographic prints. F. F. Renwick. Phot. J., 1918, 58, 140—153.

THE reflection density of a diffusing medium coated on a reflecting base is dependent on the reflecting power of the base, the surface reflection of the coating, and the optical properties of the diffusing particles suspended in the medium. Starting with the expressions deduced for the latter in a paper by himself and others on the

opacity of diffusing media (this J., 1918, 224 A), the author works out a mathematical expression for reflection density,  $D_r$ , in terms of  $\rho$ , the surface reflection,  $R_b$ , the reflecting power of the base,  $r_\infty$ , the maximum rejectance of the medium with increasing thickness,  $\lambda$ , a constant expressing the rate at which  $r_\infty$  is approached (this corresponds with the term "covering power"), and  $m$ , the quantity of the diffusing medium per unit area—thickness of coating or concentration of diffusing particles. Usually the reflecting power is determined in comparison with that of the base, or of the base plus medium without diffusing particles in the case of photographic prints, and the final expression for reflection density includes this comparison; it is of such a form that the curve connecting  $D_r$  and  $m$  for any value of  $R_b$  is part of the whole curve for the special case where  $R_b=1$ , the origin being moved by an amount representing the departure of the base from a pure white. When the factor  $r_\infty$  is very small, as in the case of photographic prints where the diffusing particles are very dark, a much simpler approximate expression is obtained and examples are given showing the close agreement between this approximation and actual observation; in this case  $m$  is taken as proportional to transmission density by diffused light with oil contact between the instrument opal and the density. The expression for reflection density previously suggested by Jones, Nutting, and Mees (this J., 1915, 250), which introduces only surface reflection, reflection of the base, and transmission density, is also discussed.—B. V. S.

*Photometer; A convenient accurate—*for the measurement of photographic densities. D. E. Benson, W. B. Ferguson, and F. F. Renwick. Phot. J., 1918, 58, 155—161.

THE photometer is an improved form of the Ferguson-Renwick photometer (Phot. J., 1911, 405, and 1914, 167), based on the law of inverse squares and using only one lamp for illuminating the density and for the variable comparison beam. An electric lamp with a small gridiron filament illuminates two pieces of opal set with their upper surfaces flush with the upper surface of a horizontal table. The front opal is directly over the lamp; the back opal is illuminated by means of a system of mirrors, two of which travel on a carriage sliding on horizontal bars, thus enabling adjustment of the effective distance between the lamp and the opal. The beams of light from the two opals are brought together in a horizontal tube by a mirror over the back opal and a Lummer-Brodhun cube over the front opal. A pointer on the mirror carriage passes along a scale logarithmically calibrated to give direct readings in densities. The length of the scale is about 22 inches and reads up to 1.8; by the use of a supplementary density in the comparison beam densities up to 2.8 may be measured, and it is suggested that the use of the much more powerful Pointolite lamp would enable the measurement of densities up to 4. The viewing tube is pivoted to swing out of the way, allowing the accurate placing of the negative on the table, which is large enough to permit the measurement of any part of a half-plate. The whole instrument may be tilted on an axis parallel to the slide bars to bring the viewing tube into convenient position. An auxiliary mirror attachment is provided in the table to give illumination of materials at an angle of 45° from above for the measurement of reflection densities.—B. V. S.

*Photographic method for the examination of eggs.* Le Roy. See XIX A.



## PATENT.

*Photographic films of varying porousness ; Method of producing* — J. H. Christensen, Sölleröd, Denmark. U.S. Pat. 1,256,981, Feb. 19, 1918. Date of appl., Mar. 9, 1916.

SEE Fr. Pat. 481,149 of 1916 ; this J., 1917, 164.

## XXII.—EXPLOSIVES ; MATCHES.

## PATENT.

*Nitro compounds and process of making same from tar-oils.* J. R. Mardick, New York. U.S. Pat. 1,258,587, Mar. 5, 1918. Date of appl., Jan. 17, 1917.

IMPURE coal tar, water-gas tar, or producer-gas tar distillate containing compounds removable by crystallisation and repeated washing with 2½ to 5% of sulphuric acid or 5% solution of soda-ash, is subjected to the action of a strong mineral nitrating acid between 10° and 60° C., the strength being gradually increased by the addition of strong acid. The polynitro compound is partially soluble in water, benzene, and toluene : it is of brownish colour, non-crystallisable, and semi-fluid below 60° C., above which it is fluid. It is partly soluble in nitroglycerin at 45° C., and completely miscible with it at ordinary temperatures.—L. A. C.

## XXIII.—ANALYSIS.

*Potash ; Determination of* — B. Blount. Analyst, 1918, 43, 117—120.

To determine potash in silicious rocks, clays, etc., from 0.5 to 2 grms. of the very finely ground sample is digested for some hours with 10 c.c., or more, of hydrofluoric acid and 2 c.c. of sulphuric acid, the mixture is then evaporated, heated until most of the sulphuric acid has been expelled, the residue of sulphates digested with hydrochloric acid and water, and the mixture filtered. The insoluble portion is again treated with hydrofluoric acid, etc. The filtrate is treated with bromine and ammonia to precipitate iron, aluminium, and manganese, and, after filtration, the calcium is precipitated as oxalate. The filtrate from the calcium oxalate is evaporated to a small bulk, nitric acid is then added and the mixture evaporated until sulphuric acid fumes appear. After cooling, the residue is dissolved in a few c.c. of dilute hydrochloric acid, a slight excess of barium hydroxide solution is added, the barium sulphate is separated by filtration, and the excess of barium salts in the filtrate removed by treatment with ammonium carbonate. The solution is then evaporated with hydrochloric acid, the residue heated to expel ammonium salts, treated with a few drops of ammonium carbonate solution, filtered, again evaporated, ignited gently, and weighed. The potassium in the mixed alkali chlorides thus obtained is determined by precipitation with platinum chloride or by means of the perchlorate method.—W. P. S.

*Copper, zinc, cadmium, nickel, and cobalt ; Determination of* — [as carbonates]. A. Carnot. Ann. Chim. Analyt., 1918, 23, 69—75.

WHEN copper, zinc, cadmium, nickel, and cobalt are precipitated as carbonates by means of sodium carbonate, the precipitation is incomplete and the precipitate always retains a quantity of alkali carbonate. If, however, a solution containing a salt of these metals is treated, in the cold, with a slight excess of sodium carbonate, and ammonia or ammonium carbonate then added in quantity just sufficient to dissolve the precipitate and the

mixture boiled, the metal is precipitated completely as carbonate or oxide. The boiling should be prolonged for at least 5 mins. and the volume of the solution should be about 200 c.c. for 0.5 grm. of metal. In the case of copper, nickel, and cobalt, ammonia or ammonium carbonate may be used ; for zinc, ammonium carbonate gives a precipitate of better physical character than does ammonia, whilst a mixture of ammonia and ammonium carbonate must be used for cadmium. —W. P. S.

*Bromine ions ; Detection and determination of* — by means of fuchsin-sulphuric acid reagent. G. Denigès and L. Chelle. Ann. Chim. Analyt., 1918, 23, 81—83.

A METHOD described previously (this J., 1913, 141) is modified as follows in order to prevent the interference of nitrites should these be present. Five c.c. of the bromide solution (containing not more than 1 grm. of bromine per litre) is mixed with 4 drops of hydrochloric acid, 4 drops of 10% potassium chromate solution, and 1 c.c. of sulphuric acid, and the mixture is immediately cooled to 15° C. ; after 5 mins., 1 c.c. of the fuchsin reagent and 1 c.c. of chloroform are added, the mixture is shaken, and then allowed to separate. The intensity of the red coloration exhibited by the chloroform layer is proportional to the quantity of bromine present. The presence of 1 grm. of sodium nitrite in a solution containing 1 mgrm. of bromine does not prevent the detection of the latter. (See also J. Chem. Soc., June, 1918.)

—W. P. S.

*Iodine in inorganic and organic compounds ; New general method for determining* — N. Tarugi. Gazz. Chim. Ital., 1918, 48, I, 1—16.

THE iodine and chlorine in solutions of iodine trichloride such as are used for the sterilisation of water may be estimated by means of the reaction :  $6\text{FeSO}_4 + 3\text{H}_2\text{SO}_4 + 2\text{ICl}_3 = 3\text{Fe}_2(\text{SO}_4)_3 + 2\text{I} + 6\text{HCl}$ . The ferrous sulphate solution employed is prepared by dissolving about 14 grms. of the pure, crystallised salt in 100 c.c. of water and adding to the filtered liquid 100 c.c. of 2N-sulphuric acid ; the titre of this solution is determined exactly by titration with standard permanganate solution. The procedure is as follows : A definite volume of the iodine trichloride solution and exactly 25 c.c. of the ferrous sulphate solution are shaken vigorously in a separating funnel, left for 1½ hours, and then repeatedly extracted with chloroform until the latter is no longer coloured. The chloroform solutions are washed with a little water, which is afterwards extracted with fresh quantities of chloroform as long as the latter becomes coloured. The total chloroform, which contains in solution the whole of the iodine, is mixed with an equal volume of water and titrated with N/10-sodium thiosulphate solution. The aqueous solution which was extracted with chloroform and which consists of the acid ferrous sulphate, is filtered and an aliquot part of it titrated with N/10-permanganate solution, the result being calculated for the total volume including the volume of the iodine trichloride solution used : 1 c.c. of N/10-permanganate corresponds with 0.00355 grm. Cl. The total active halogens in the iodine trichloride solution may be estimated by direct titration of the solution with N/10-thiosulphate solution. Tables are given showing the relative proportions of iodine monochloride and trichloride corresponding with different values of the ratio, iodine : chlorine, and the concentrations of iodine trichloride by weight and by volume for solutions of specific gravities between 1.010 and 1.365.

This method may be applied to the estimation of iodine in both inorganic and organic compounds.

With an inorganic compound, this is dissolved or suspended in water, the vessel containing the liquid being surrounded with ice, and chlorine passed through until it becomes straw yellow and excess of chlorine is evident in the vessel. The tube through which the chlorine is passed is washed into the liquid, the latter being made up to a definite volume and an aliquot part treated with the standard ferrous sulphate solution, extracted with chloroform, and so on. When the iodine compound to be tested contains oxygen, it is mixed with an equal weight of sodium bisulphite before being treated with chlorine.

A few organic compounds containing iodine give up the latter as trichloride when suspended in water and treated with chlorine, but in most cases it is necessary to heat the compound with sulphuric acid while the chlorine is passed. Use may be made of a tubulated retort, the end of the tube being turned downwards and joined to a glass tube dipping into about 10 c.c. of water in a beaker kept cool by ice. The substance is introduced into the retort together with 5—10 c.c. of concentrated sulphuric acid, and chlorine is passed in, the liquid being gradually heated to boiling meanwhile. The iodine trichloride solution thus obtained is then treated as described above. All the organic compounds examined yield their iodine as trichloride under this treatment.—T. H. P.

*Nitrogen; Kjeldahl's method for the determination of —.* E. Salm and S. Prager. *Chem.-Zeit.*, 1918, 42, 104—105.

WHEN a nitrogenous substance has been decomposed by digestion with sulphuric acid and mercury, or with sulphuric acid containing phosphoric anhydride and mercury, the subsequent distillation of the ammonia must be made with the addition of zinc dust if potassium sulphide is not used together with the requisite quantity of sodium hydroxide. If, however, potassium sulphide is used, zinc turnings or shavings may be employed in place of zinc dust. In the absence of zinc, or if this is replaced by aluminium, the results obtained are too low.—W. P. S.

*Foam inhibitor in the Van Slyke amino-nitrogen method.* H. H. Mitchell and H. C. Eckstein. *J. Biol. Chem.*, 1918, 33, 373—375.

DIPHENYL ether is recommended as an excellent substitute for isocaprolic alcohol for the prevention of foaming during the evolution of nitrogen in the Van Slyke method (this J., 1911, 771, 1135) of determining amino-nitrogen in animal and plant extracts.—H. W. B.

*Malic and tartaric acids; Optical method for the determination of — in the same solution.* J. J. Willaman. *J. Amer. Chem. Soc.*, 1918, 40, 693—701.

THE method depends on the facts that uranyl acetate enhances the rotation of *l*-malic and *d*-tartaric acids (see this J., 1911, 1177, 1407), whilst ammonium heptamolybdate reverses the sign of rotation of malic acid, giving strongly dextro-rotatory solutions with each acid. It is designed for solutions containing from 0.1—0.6 or 0.8 gm. of each acid, the necessary volume of liquid, e.g., apple juice, being ascertained by titration. The acids are precipitated as their barium salts and liberated again, and then aliquot parts are mixed with the two activating agents and examined in the polarimeter. The two readings are referred to a chart which is prepared by plotting the rotations of different mixtures activated by uranyl acetate against those of the same mixtures plus ammonium heptamolybdate. The curves cross, and the point of intersection in a given case corresponds with the weight of malic

acid along one axis and the tartaric acid along the other. (See further, *J. Chem. Soc.*, June, 1918.)—J. C. W.

*Analysis of glass.* Ronnet. See VIII.

*Volumetric determination of manganese [in steel] by means of sodium arsenite.* Ibbotson. See X.

*Determination of cobalt and nickel in cobalt steel.* Schoeller and Powell. See X.

*Determination of copper minerals in partly oxidised ores.* Van Barneveld and Leaver. See X.

*Sulphur [copper sulphide] and copper oxide determination.* Maier. See X.

*Method of sulphur analysis [in ores].* Hawley. See X.

*Estimation of manganese in aluminium alloys and dust.* Clemmell. See X.

*Determination of bismuth in lead bullion.* Jessup. See X.

*Determination of the purity of castor oil.* Chercheffsky. See XII.

*Copper-phosphate mixtures as sugar reagents. Qualitative test and quantitative titration method for sugar [dextrose] in urine.* Polin and McElroy. See XVII.

*Note on Cammidge's method for the estimation of [reducing] sugar.* Garrow. See XVII.

*The ash test for honey.* Stout. See XVII.

*Methods for determining the adulteration of wines.* Pratolongo. See XVIII.

*Non-protein nitrogen in wheat flour.* Blish. See XIXA.

*Estimation of lactose in milk.* Folin and Denis. See XIXA.

*Graduation and calibration of Gerber new milk butyrometers.* Day and Grimes. See XIXA.

*Determination of the acidity of condensed milk.* Dugardin. See XIXA.

*Photographic method for the examination of eggs.* Le Roy. See XIXA.

*The nitrogen distribution of fibrin hydrolysed in the presence of ferric chloride.* Morrow and Fetzer. See XIXA.

*Analysis of cholesterol [compounds].* Lifschütz. See XX.

*A convenient accurate photometer for the measurement of photographic densities.* Benson and others. See XXI.

#### PATENTS.

*Metals; Apparatus for testing the hardness of —.* Reid Bros. (Engineers), Ltd., London, and G. Brown, South Norwood, Surrey. Eng. Pat. 114,593, May 22, 1917. (Appl. No. 17,231 of 1917.)

THE specimen to be tested is placed on an anvil,



and an indenting member with a hardened point slides in a vertical guide and rests freely on the specimen. Means are provided for dropping a ball or other known weight from a predetermined height on to the indenting member. The amount of indentation is measured by means of the deflection of a beam of light by the angular movements of a small mirror, which is mounted on a flat spring so arranged that it is deflected by a collar on the indenting member to an amount depending on the depth of penetration of the specimen. This indentation is compared with that produced in a standard specimen.—W. F. F.

*Melting-points; Method and apparatus for determining*—L. W. Chubb, Edgewood Park, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,259,363, Mar. 12, 1918. Date of appl., May 6, 1915.

THE sample to be tested is supported on a member of refractory conducting material which is heated by the passage of an alternating current, and which forms with another connected member a thermoelectric couple. The terminals of the supporting member are connected by a non-inductive resistance, and a direct current measuring instrument has its terminals connected to the second member of the thermoelectric couple, and to the neutral point of the resistance respectively.—W. F. F.

*Flue-gases; Apparatus for determining the quantity of carbon dioxide in*—Z. Olsson, Assignor to A. Rust-Oppenheim, New York. U.S. Pat. 1,260,020, Mar. 19, 1918. Date of appl., Mar. 29, 1917.

THE gas is passed through a closed receptacle containing a buoyant expansible body adopted to rise and fall with changes in the density of the gas. The movements of the body are indicated by connecting it to a pointer moving over a graduated scale.—W. F. F.

## Patent List

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Aubert. Furnaces. 7473. May 3. (Fr., Mar. 10, 1917.)  
Beeby. Apparatus for filtering and straining liquids. 7583. May 6.  
Cooper and Griffiths. Apparatus for evolving gases from liquids. 7515. May 4.  
Cunningham. Separation of liquid mixtures. 7530. May 4.  
Fox. Dehydrating and desiccating apparatus. 7382. May 2.  
Harvey, and Harvey Gas Furnace Co. Regenerative or recuperative furnace installations. 7529. May 4.  
Hodson. Kiln for incinerating earthy matters. 7623. May 7.  
Linden. 7464. See XIX.  
Linden. 7430. See XII.  
Mann. Drying, concentrating, or evaporating liquids or semi-liquids. 7518. May 4.

Petrie. Machines for drying granular or fibrous materials. 7612. May 7.

Reynard. Effecting rapid crystallisation of substances in solution. 7959. May 11.

Rudge, and United Alkali Co. Apparatus for subjecting matter to the action of gases or vapours, for making bleaching-powder, etc. 7875. May 10.

Wilson. Automatic stills. 7541. May 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

43 (1917). Marks (Isko, Inc.). Refrigerating apparatus. (115,051.) May 8.

884 (1917). Aylsworth and others. See XX.

5829 (1917). Testrup, and Techno-Chemical Laboratories, Ltd. Conducting high-temperature reactions. (115,087.) May 8.

7867 (1917). Enock & Co., and Enock. Jacketed vats for heating and cooling liquids. (115,126.) May 8.

7940 (1917). Roy, and Morson and Son. Means for purifying and absorbing gaseous products of chemical reactions. (115,311.) May 15.

9015 (1917). Jeffcock and Yardley. Separating water and other matter from compressed air and other gases. (115,325.) May 15.

11,539 (1917). Bloxam (Zellstofffabrik Waldhof). Disposal of waste gases from chemical operations. (115,350.) May 15.

16,135 (1917). Schlatter and Frorath. Drying-installations. (115,193.) May 8.

18,516 (1917). Marks (Isko, Inc.). Refrigerating apparatus. (115,208.) May 8.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Arkell. Artificial fuels, and processes for manufacturing same. 7803. May 9.

Ballingall and others. 7557. See VII.

Boissier. Solid fuel. 7862. May 10. (Fr., May 10, 1917.)

Corthésy. Distillation of liquid hydrocarbons. 7315. May 1.

Corthésy. Generation of gas from coal dust, etc. 7710. May 8.

Glover, West, and Wild. Retorts for continuous carbonisation of coal, etc. 7586. May 6.

Harger. 7772. See VII.

Lamberty. Manufacture of briquettes. 7528. May 4.

Leadbeater. Means for drying and carbonising peat, etc. 7415. May 3.

Maseyk. Automatic coal-gas producer. 7765. May 9.

Parker. Generation, from incandescent carbonaceous material, of fuel gas for internal-combustion engines. 7435. May 3.

Soddy. Stripping illuminants from and purifying coal gas by means of charcoal. 7698. May 8.

Stevenson. Fuel. 7263. May 1.

Stobart. Washers for coal, etc. 7246. Apr. 30.

Thuman. Apparatus for feeding fuel, etc., to gas-producers. 7532. May 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,575 (1916). Riedel. Operation of gas generators. (101,220.) May 8.

16,469 (1917). Soc. Franco-Belge de Fours à Coke. Horizontal coking ovens. (113,779.) May 8.

5095 (1918). Gamet and others. See X.

### III.—TAR AND TAR PRODUCTS.

#### COMPLETE SPECIFICATIONS ACCEPTED.

5567 (1917). Thuman (Evans). Manufacture of toluol. (115,066.) May 8.

6035 (1917). Inray (Soc. Chem. Industry in Basle). Manufacture of  $\beta$  aminoanthraquinone, (115,259.) May 15.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATIONS.

Clark (Chem. Fabr. Worms). Colours soluble in oils and fats, and process of preparing same. 7719. May 8.

Dydynski. Manufacture of sulphur colour for printing textile fabrics, etc. 7475. May 3.

Imray (Soc. Chem. Industry in Basle). Manufacture of mordant-dyeing azo dyestuffs and metal compounds thereof, and their application in dyeing. 7816. May 9.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Brégeat. Recovery of acetate of cellulose from industrial waste material. 7809. May 9. (Fr., Dec. 1, 1917.)

Carper and Tulloch. Extraction of fats, etc., from wool. 7255. Apr. 30.

Dreyfus. Manufacture of compositions or articles having a basis of cellulose acetate. 7192 and 7193. Apr. 29.

Groves and Ward. 7177. See XIII.

Harvey. 7740. See XIII.

Johnston and Sutherland. Degumming or cleansing stalk fibres. 7486. May 4.

Moore. Production and treatment of fibrous materials. 7312. May 1.

Robbins. 7907. See XIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Barnes, and Spence & Sons. Mordanting vegetable fibres. 7829. May 10.

Dydynski. 7475. See IV.

Moore. Dyeing with direct (substantive) cotton colours. 7656. May 7.

Imray (Soc. Chem. Ind. in Basle). 7816. See IV.

Robertson. Printing woven fabrics. 7547 and 7826. May 6 and 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

5785 and 9702 (1917). Farrar and Whitehead. Dyeing machines, washing-azof machines, etc. (115,082.) May 8.

6339 (1917). Albrecht. Production of printed cloths. (115,100.) May 8.

6414 (1917). Calico Printers' Assoc., and Fourneaux. Production of black on vegetable fibres. (115,278.) May 15.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Ballingall, and R. and J. Dempster. Heat-controlling means for hydrogen-gas plants. 7557. May 6.

Becket. Manufacture of calcium carbide. 7239 and 7240. Apr. 30.

Blumenberg. Treatment of borax ores. 7848. May 10.

Dutt and Dutt. Manufacture of aluminium chloride, aluminium-sodium chloride, and carbonates of soda and potash. 7841. May 10.

Gres et Bouchardy. Manufacture of nitric anhydride. 7392. May 2. (Switzerland, May 16, 1917.)

Gres et Bouchardy. Manufacture of nitric acid. 7393 and 7394. May 2. (Switzerland, June 2 and 16, 1917.)

Harger. Manufacture of hydrogen. 7772. May 9.

Heberlein and Mond. Production of preparations containing colloidal copper compound. 7576. May 6.

Hirst. Recovery of rare earths. 7216. Apr. 30.

Hulin. Preparation of anhydrous metallic chlorides. 7661. May 7.

Jones and Parsons. Process of oxidising ammonia, and catalytic bodies. 7221. Apr. 30.

Mackintosh. Extraction of radioactive substances from silicious and aluminous materials. 7819. May 9.

Rideal and Taylor. Manufacture of hydrogen. 7232. Apr. 30.

Rudge, and United Alkali Co. 7875. See I.

## COMPLETE SPECIFICATIONS ACCEPTED.

6317 (1917). Westling and Andersen. Separation of molybdenum compounds. (115,274.) May 15.

8126 (1917). Richards. See VIII.

10,286 (1917). Grossmann. Method of obtaining nitre-cake in a disintegrated or ground form. (115,340.) May 15.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Marlow. Ovens or kilns for making tiles, pottery, etc. 7487. May 4.

Parsons. Glass furnaces. 7638. May 7.

Steele. Sifting apparatus for potters' slip, glaze, etc. 7488. May 4.

## COMPLETE SPECIFICATIONS ACCEPTED.

1339 (1917). Chambers. Manufacture of glass. (103,672.) May 15.

8126 (1917). Richards. Furnaces or kilns for calcining limestone, fireclay, or other refractory materials. (115,316.) May 15.

8536 (1917). Soc. Nouv. des Etabl. E. Muller. Baking graphite crucibles, bricks, and other refractory objects having graphite for a base. (109,247.) May 15.

## IX.—BUILDING MATERIALS.

## APPLICATION.

Hadfield and Hadfield. Manufacture of concrete. 7919. May 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

6121 (1917). Middlesboro' Slag Co., and Major. Manufacture of slag bricks. (115,100.) May 8.

11,419 (1917). Ito. Heat-insulating compounds. (115,163.) May 8.

14,247 (1917). Abraham. Roofing materials. (113,778.) May 8.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Aitken. Crucible furnaces for melting metals. 7474. May 3.

Ashcroft. Manufacture of magnesium by electrolysis. 7245. Apr. 30.

Ashcroft. Manufacture of magnesium or its alloys by electrolysis. 7705. May 8.

Ashcroft. Treatment of complex or refractory sulphide ores or products. 7706. May 8.

Bowes. Aluminium soldering process. 7289. May 1.

Burnett and Jones. Case-hardening materials. 7556. May 6.

Burr. Admixture of metals or substances for alloying. 7311. May 1.

Clevenger. Electrolytic production of zinc. 7665 and 7667. May 7. (U.S., May 7, 1917.)

Crocker and Crocker. Manufacture of zinc. 7505. May 4.

Edser, Tucker, and Minerals Separation, Ltd. Concentration of ores. 7742. May 8.

Elmore. Extraction of lead and zinc from their ores. 7732. May 8.

Gröndal. Mechanical roasting-furnace. 7650. May 7. (Sweden, Mar. 24, 1917.)



Mayrow. Apparatus for chemical and electro cleaning and depositing metals. 7188. Apr. 29.

Mordey. Heat-treatment of iron and steel and their alloys. 7752. May 8.

Stabilimenti Biak. Process for melting metals. 7648. May 7. (Ital., May 7, 1917.)

Wright and Eagle Range, Ltd., and Yates. Tilting crucible furnaces. 7457. May 3.

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,310 (1916). Reichenstein. Protecting iron surfaces from chemical action. (103,814.) May 15.  
1674 (1917). Faulkner. Electric furnaces for melting or refining non-ferrous alloys. (115,248.) May 15.

5835 (1917). Ross and Crump. Extraction of copper or other metals from ores, etc. (115,088.) May 8.

6240 (1917). Temple. Composition for use in welding steel or wrought iron. (115,102.) May 8.

8653 (1917). Rollason. Combined tilting and reverberatory open-hearth melting-furnace and converter. (115,323.) May 15.

10,385 (1917). New Delaville Spelter Co., and Shortman. Extraction of zinc. (115,409.) May 15.

16,129 (1917). Stabilimenti Biak. Metal alloys. (111,288.) May 15.

17,325 (1917). Kimbell. Separation or concentration of ores. (115,199.) May 8.

5095 (1918). Gaunt, Brookfield, and Tylor and Sons. Means for preheating the charge in crucible and like furnaces. (115,222.) May 8.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Armelin. Dry batteries. 7727. May 8.

Ashcroft. 7245 and 7705. *See* X.

Bowen and Marino. Secondary batteries. 7175. Apr. 29.

Campbell and Gow. Control of power absorbed in electric furnaces. 7957. May 11.

Clevenger. 7665 and 7667. *See* X.

Hawkins. Accumulator cell. 7387. May 2. (U.S., May 2, 1917.)

Mayrow. 7188. *See* X.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,338 (1916). Ajax Metal Co. Induction furnaces. (103,645.) May 15.

1588 (1917). British Electric Transformer Co., and Berry. Electric furnaces and transformers for use therewith. (115,247.) May 15.

1674 (1917). Faulkner. *See* X.

15,815 (1917). Dean, and Chloride Electrical Storage Co. Electric accumulators. (115,374.) May 15.

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Carper and Tulloch. 7255. *See* V.

Linden. Separating, recovering, and washing oil, grease, fat, etc., from water, etc., or separating two liquids of different densities. 7430. May 3.

Janaka. Manufacture of soap containing sulphur. 7249. Apr. 30. (Japan, Aug. 1, 1917.)

#### COMPLETE SPECIFICATION ACCEPTED.

6761 (1917). Huntington and Barnes. Soap. (115,110.) May 8.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Clark (Chem. Fabr. Worms). 7719. *See* IV.

Groves and Ward. Dopes or varnishes for aeroplanes. 7177. Apr. 29.

Harvey. Dopes for fabrics. 7740. May 8.

Robbins. Preparation of dope for treating fabrics for aircraft. 7907. May 10.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Clark (Chem. Fabr. Worms). Tanning animal hides. 7720. May 8.

Haack and Testas. Tanning fishing-nets. 7526. May 4.

Kay and Platt. Automatic tanning plant. 7694. May 8.

Weiss. Manufacture of horny masses from casein, etc. 7479. May 3. (Germany, Apr. 28, 1917.)

### XVI.—SOILS; FERTILISERS.

#### APPLICATIONS.

Brown. Grub-resisting fertilising material. 7684. May 8.

Furse. Manufacture of manure from refuse. 7843. May 10.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATION.

Lang. Manufacture of syrup from beetroot. 7762. May 9.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Adams. Vegetable meal and/or flour. 7551. May 6.

Johnston and Kearney. Preservation of food-stuffs. 7784. May 9.

Linden. 7430. *See* XII.

Linden. Means for drying sludge, etc. 7464. May 3.

#### COMPLETE SPECIFICATIONS ACCEPTED.

6086 (1917). Bendixen. Method of making soluble the nucleo-albumins in preserved eggs. (115,261.) May 15.

17,607 (1917). Reiser Ges. Process of softening water. (112,127.) May 8.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Newton (Bayer and Co.). Manufacture of a pharmaceutical product. 7867. May 10.

Newton (Bayer and Co.). Manufacture of dialkylbromoacetylurea. 7868. May 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

884 (1917). Aylsworth, and Savings Investment and Trust Co. Apparatus for performing organic chemical reactions. (103,665.) May 8.

5755 (1917). Callimachi. Manufacture of salicylic acid. (115,080.) May 8.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATION.

Keerotype, Ltd., and Middleton. Photographic printing-paper and transfer processes. 7641. May 1.

### XXIII.—ANALYSIS.

#### APPLICATION.

Richardson. Means for testing hardness of materials. 7825. May 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,102 (1917). Guillemin, and Comp. des Forges d'Audincourt et Dépendances. Calorimetric meters. (115,354.) May 15.

14,096 (1917). Siemens und Halske A.-G. Photometry and apparatus therefor. (112,614.) May 8.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Gas reactions; Method of obtaining general velocity curves for complete homogeneous — at constant pressure.* G. W. Todd. Phil. Mag., 1918, 35, 435—444.

REACTION velocity curves for bimolecular and termolecular reactions at constant pressure have been plotted, and their application to technical gas reactions is referred to. The graphs have reference to termolecular reactions of the type  $2A + B$  with either A or B in excess, and afford directly the percentage of the original substances which has undergone transformation after different time intervals. The technically important cases, in which the reaction products occupy two-thirds and one-third respectively of the volume of the reacting substances, are both dealt with in the paper. (See also J. Chem. Soc., 1918, ii., 190.)—H. M. D.

**PATENTS.**

*Condensers or coolers for gases or liquids.* E. C. Stables, and J. and E. Hall, Ltd., Dartford, Kent. Eng. Pat. 114,651, Apr. 5, 1917. (Appl. No. 4893 of 1917.)

A SHALLOW casing is provided with two partitions arranged in spiral form or of any modified shape, and coiled in opposite directions so as to form a tortuous passage leading from the circumference to the centre and back to the circumference in the opposite direction. The cooling medium is passed through the passage. A number of tubes for the fluid to be cooled are arranged in the passage and are connected in parallel at each end to a single inlet or outlet box inside the casing, from which an inlet or outlet pipe passes through the casing. The cooling fluid may pass through the passage in the same or in the opposite direction to that of the fluid to be cooled. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 936 of 1874.)—W. F. F.

*Condensers for acids.* W. Hayhurst, Accrington. Eng. Pat. 114,706, May 30, 1917. (Appl. No. 7737 of 1917.)

A CONDENSER for acids, such as nitric and hydrochloric acids, comprises a vessel of prismatic form having a cross-section of triangular or other form with large cooling surface area. In one form the cross-section may be of inverted T-shape. A series of such vessels is arranged in a sloping tank, the vessels being connected at the bottom.—W. F. F.

*Drying medium; Method of and apparatus for producing a —.* L. C. Sadezky, London. Eng. Pat. 114,810, Feb. 19, 1918. (Appl. No. 2888 of 1918.)

A DRYING medium suitable for drying clothing, textile fabrics, powder, liquids, or semi-liquids such as milk, eggs, and the like, is produced by separating the moisture from air by cooling with liquid

ice is deposited, and thence to the conduit, E, liquid air being sprayed in at F. The mixture passes into a second depositing chamber, G, and the dry air then passes through a conduit, H, provided with steam heating pipes, J. The dry hot air is withdrawn for use by a fan, K.—W. F. F.

*Dehydrating-machine for desiccating liquids.* T. J. Coster and D. G. Putnam, Chicago, Ill. U.S. Pat. 1,258,988, Mar. 12, 1918. Date of appl., Feb. 2, 1917.

A STATIONARY cylindrical casing having means to supply heated gas to its interior is mounted on a rotary shaft. A drum within the casing is secured at one end to the shaft and at the other end is provided with an annular ring which is journaled on a stationary disc. A feed pipe passes through the disc and supplies the fluid to be dried to the interior surface of the drum. The dried material is removed from the drum by means of a scraper carried by the disc, and a conveyor carries it out of the machine.—J. H. P.

*Drying machine.* F. G. Sargent, Westford, Assignor to C. G. Sargent's Sons Corporation, Graniteville, Mass. U.S. Pat. 1,259,675, Mar. 19, 1918. Date of appl., July 11, 1917.

THE machine consists of a casing, inside which is a movable screen to support the material to be dried. Heated air is conducted to the space above the screen and passes through the screen to a number of passages, through which part of the air is returned to the heating chamber and used again, whilst the remainder passes to the exhaust.

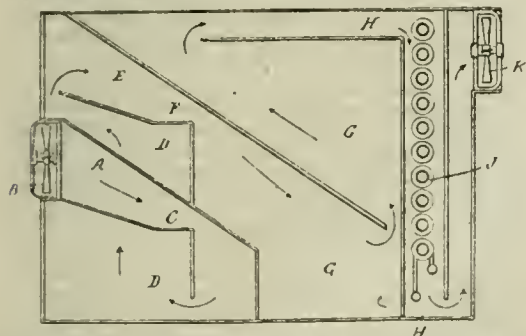
—J. H. P.

*Heat interchanging apparatus; Tubular —.* E. L. Pease, Hurworth Moor, A. Bell, and The British Still Tube Co., Ltd., Darlington. Eng. Pat. 114,863, Mar. 6, 1917. (Appl. No. 3327 of 1917.)

IN an apparatus for heating air by means of steam, a number of heating units are arranged in two tiers. Each unit consists of a pair of vertical rectangular headers connected by horizontal tubes provided with vertical metal radiating ribs, e.g., by expanding the tubes. Openings are provided in the headers for the insertion of an expanding tool and for cleaning purposes. The headers of several units are adapted to fit together so as to form the side walls of the conduit through which air to be heated is passed by pressure or suction. The front headers are divided horizontally into two compartments so that the steam passes through the upper pipes and returns through the lower pipes, and the units are connected to a common steam supply pipe above the apparatus and to a common return pipe for condensed water below the apparatus, valves being provided so that any or all of the units may be put into action. The upper and lower compartments of the upper tier of front headers are connected by intermediate pipes to the upper and lower compartments of that front header of the lower tier which is below the adjacent upper unit, so that all the connecting pipes are inclined. The first unit in the lower tier and the last unit in the upper tier have a direct valved connection with the steam supply pipe, and the last unit in the upper tier has a direct valved connection with the return pipe for condensed water.—W. F. F.

*Evaporators, steam generators, steam separators, or the like apparatus; Anti-priming devices or baffle plates for —.* H. Fay, Liverpool. Eng. Pat. 114,888, Apr. 18, 1917. (Appl. No. 5432 of 1917.)

AN anti-priming device for evaporators comprises two horizontal sets of annular or spiral baffles which intermesh with one another and divide the evaporator horizontally into two parts. The baffles are each carried by a horizontal plate, the upper set



air. A fan, B, forces air into a conduit, A, and liquid air is sprayed in at the contracted passage, C. The mixture passes into the chamber, D, where



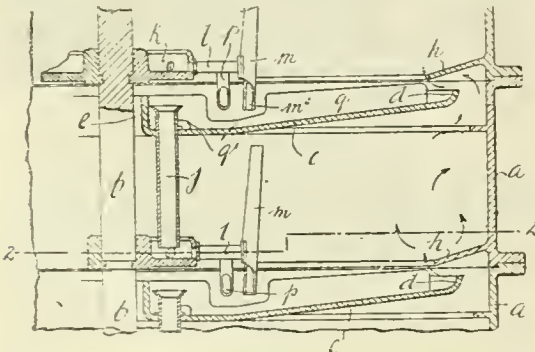
being suspended from a rod which screws through the top of the casing so that the extent of intermeshing of the baffles may be regulated. An indicating pointer may be attached to the screwed rod so as to indicate the position of the baffles on a scale. The steam passes upwards through a central opening in the lower plate and then radially outwards in a zigzag path over the baffles, while the separated water escapes through perforations in the lower plate into a conduit from which it flows back into the water space of the evaporator. The edges of the baffles may be beaded, flanged, or curved inwards against the direction of steam flow.—W. F. F.

*Separating liquids of different specific gravity, insoluble or difficultly soluble in each other, such for instance as an emulsion or mixture of grease and water.* J. Carroll, Wigan. Eng. Pat. 114,921, May 15, 1917. (Appl. No. 6913 of 1917.)

The warm or hot emulsion of grease and water is placed in a chamber provided with an internal contracted neck at the top, the space surrounding the neck being filled with cold water. A short tube fits into the neck and the grease solidifies in the tube, which may then be withdrawn and the grease removed by pulling a scraping disc through the tube. In a modification, the vessel into which the emulsion is passed at the bottom, may be provided with a horizontal partition, having openings over which inverted funnels are placed to collect the solidified grease. The top of each funnel is flared outwards, and all may be united by shallow channels. One may be provided with a lip for the overflow of water. The apparatus is specially applicable for recovering fats from water used for washing sheep. Soluble fats are previously rendered insoluble by the addition of sulphuric acid.—W. F. F.

*Liquids and gases, vapours, fumes, and the like; Apparatus for bringing — into intimate contact.* W. B. Davidson, and W. C. Holmes and Co., Ltd., Huddersfield. Eng. Pat. 115,006, Jan. 18, 1918. (Appl. No. 1025 of 1918.)

In an apparatus of the type described in Eng. Pat. 9302 of 1914 (this J., 1914, 1082), a number of shallow trays, *e*, with upturned edges, *d*, *e*, are superposed in a casing, *a*, and liquid overflows from one to the other by pipes, *f*, which dip into



the spraying vessels, *k*. These vessels are mounted on a rotating shaft, *b*, and carry short radial projecting pipes, *l*, to which spraying vanes, *m*, having scoop portions, *m'*, are secured. The upper portions of the vanes are serrated and an additional scoop, *p*, may be provided. The gas to be treated passes upwards through the apparatus as shown by the arrows, and its contact with the spray is facilitated by the inclined deflecting plate, *h*. Rotation of the liquid is prevented by radial plates, *q*, and additional radial ribs may be provided to act

as gas scrubbers. In a modification, the arms carrying the sprayers are formed as tangential tapering extensions from the vessels, *k*, so that the liquid is directed towards the vanes. In another modification a horizontal annular plate may extend inwards from the plate, *h*, and may be provided with radial slots to distribute the gas.

—W. F. F.

*Refrigerating apparatus.* M. F. Ewen and A. L. S. McCurdy, Chicago, Ill., Assignors to Larsen Ice Machine Co. (Inc.), Wilmington, Del. U.S. Pat. 1,259,006, Mar. 12, 1918. Date of appl., Nov. 4, 1915.

BETWEEN the absorber and the still and connected with them by valved pipes is a measuring device, and the valves are operated alternately, so that at definite intervals predetermined amounts of saturated absorbent are transferred from the absorber to the still by gravity. Between the expansion chamber and a combined condenser and liquid receiver is a positively operated valve which is actuated in fixed time relation with the valves controlling the inlet and outlet of the measuring device.

—J. H. P.

*Dust filter and the like.* G. H. Fraser, Brooklyn, N.Y. U.S. Pat. 1,259,008, Mar. 12, 1918. Date of appl., Apr. 20, 1915.

A NUMBER of chambers connected with a common exhaust and a common inlet main, are each provided with a filter chamber through which the dust-laden gas is caused to flow. Any one of the chambers may be cut out of the circuit by means of valves, and other valves are provided by means of which a reverse flow may be induced through the chamber to clean the filter.—J. H. P.

*Filter.* C. W. Merrill, Berkeley, Cal. U.S. Pat. 1,259,032, Mar. 12, 1918. Date of appl., Dec. 8, 1915.

A NUMBER of flat circular filter elements are arranged parallel to one another and the liquid is directed against their surfaces by perforated radial pipes mounted on a central rotating supply conduit passing concentrically through all the filter elements. Each filter element is provided with an aperture extending from the centre to the side wall, and the solid material is removed mechanically through the aperture.—W. F. F.

*Filter; Rotary suction —.* O. J. Salisbury, Salt Lake City, Utah. U.S. Pat. 1,259,139, Mar. 12, 1918. Date of appl., May 24, 1917.

THE filter is formed of a series of similar units arranged side by side in spaced relation, each comprising a solution tank and a filter agent mounted to revolve therein. The filter agents are mounted on a longitudinal, hollow shaft above the tanks.

—J. H. P.

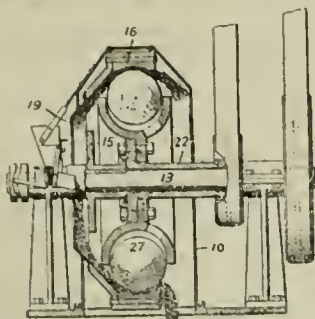
*Classifier.* A. L. Blomfield, Denver, Colo., Assignor to The Dorr Co. U.S. Pat. 1,260,135, Mar. 19, 1918. Date of appl., Apr. 21, 1916.

A HOLLOW container has an overflow for liquid and suspended matter and a flat bottom surface provided with an outlet for material which settles. Sweeps move the latter across the bottom towards the outlet, and a current of liquid introduced at the bottom lifts the finer solids to the overflow. The material is fed into the container below the level of the overflow.—J. H. P.

*Pulveriser.* J. C. Clark, Atlanta, Ga. U.S. Pat. 1,260,330, Mar. 26, 1918. Date of appl., Jan. 13, 1916.

A STATIONARY casing, 10, is provided with a discharge opening at the bottom, and an opening in one side in which a dish-shaped plate, 19, fits. This plate is carried by the cup-shaped member, 15, 16,

which is mounted on the horizontal shaft, 13, and provided with perforations in its base through which material is guided by the plate, 19. A sleeve, 22, is mounted to rotate on the shaft, 13, and carries a spider provided with peripheral pockets,



27, carrying grinding rolls which bear against the ring portion, 16, of the cup-shaped member. The shaft and sleeve carry pulleys and are independently rotated.—W. F. F.

*Cooling towers of reinforced concrete; Construction of* —. F. K. T. van Isteron, Heerlen, and G. Kuypers, Rotterdam, Netherlands. Eng. Pat. 108,863, Aug. 8, 1917. (Appl. No. 11,459 of 1917.) Under Int. Conv., Aug. 16, 1916.

*Pulverising apparatus.* R. A. Lachmann, Chicago, A. W. Raymond, Evanston, and W. B. Senseman, Assignors to Raymond Bros. Impact Pulverizer Co., Chicago, Ill. U.S. Pats. 1,260,406 and 1,266,407, Mar. 26, 1918. Dates of appl., Oct. 23, 1915, and Mar. 31, 1917.

See Eng. Pats. 101,962 and 105,763 of 1916; this J., 1917, 1262, 995.

*Separating particles from a fluid by combined centrifugal and filtering action; Method and means for* —. K. and A. Ward, Stockholm. U.S. Pat. 1,262,146, Apr. 9, 1918. Date of appl., May 22, 1914. Renewed Oct. 23, 1917.

See Fr. Pat. 472,701 of 1914; this J., 1915, 479.

*Annealing furnace.* U.S. Pat. 1,258,885. See X.

*Apparatus for drying and deodorising night soil, slaughter house refuse, and analogous materials.* Eng. Pat. 114,700. See XIXa.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coke-ovens; Chemical problems at [ammonia-recovery plants in connection with]* —. G. A. Phillipson. Coke Oven Managers' Assoc., Apr. 27, 1918. Chem. Trade J., 1918, 62, 355—356.

The view is advanced that the formation of "rock salt" in the saturators is due in most cases to irregular working. In direct and semi-direct plants uneven distribution of the gas in the saturator favours the formation of "rock salt" at the quietest parts. In semi-direct plants the variation of temperature is a contributory cause, and the feed of liquor and steam to the still should be maintained as constant as possible. The scum of arsenious sulphide which forms when arsenical acid is used favours the development of "rock salt," but the author has been able to avoid this trouble, although for two years he has used an acid mixture 75% of which was arsenical. The acidity of the bath is allowed to fall during a shift from 20% at the start to 6%. Half an hour before the end the ammonia liquor feed to the still is stopped and steam allowed to blow through to the saturator, while the acidity of the bath is brought

up to 20% again. The scum of arsenious sulphide coagulates and is precipitated. When the feed of liquor is started again at the beginning of the next shift the sulphide is ejected with the first crop of sulphate crystals. This method of working also prevents accumulations of pitch. The use of arsenical acid has led to rapid destruction of the saturator curtain. In semi-direct plants it is desirable to introduce the feed of fresh acid and drainings at the same level as the serrations of the distributor. In general, the use of hot water is a great aid to the prevention of "rock salt."

—H. J. H.

*Fuel oil from vertical retorts by carbonisation of cannel and coal.* J. West. Gas J., 1918, 142, 133—134.

EXPERIMENTS have been made to see if the vertical retorts of gasworks could be adapted to fuel oil production by the carbonisation of cannel and coal. Trials at low temperatures (700° C. outside the retort) had given poor results in regard to yields of gas, oil, and ammonia, whereas the by-products are important when the coke is of low value. Satisfactory yields were obtained when the retorting temperature was raised to 1150°—1200° C., the temperature at the top of the retort being reduced and steam admitted at the bottom. Carbonisation of a low-grade cannel (ash 9.57%, volatile matter 30.35%, and moisture 2.90%) in Glover-West retorts at 1130° C. (outside) gave the following results: Throughput, 4.78 tons per 24 hours; gas make per ton, 11,180 cu. ft., of calorific value 541 B.Th.U.; tar oil (dehydrated) per ton, 29.9 gallons; ammonium sulphate per ton, 41 lb.; fuel oils per ton, 17 gallons. Similar results were obtained when coal was carbonised.—H. J. H.

*Lubricating oils; Relation between viscosity and the chemical constitution of [mineral]* —. A. E. Dunstan and F. B. Thole. Inst. Petroleum Tech., Apr. 16, 1918. [Advance proof.]

VISCOSITY is a function both of mechanical friction of molecule against molecule, and of resistance to deformation brought about by the mutual attraction of the molecules for one another. It must be therefore largely a constitutive property, the important variables being unsaturation, conjugation, and symmetry. Unsaturation has in general the effect of raising viscosity; the alternation of double and single linkings of carbon atoms has the same effect in a higher degree; a symmetrically-distributed molecule possesses a lower viscosity than one markedly unsymmetrical. A lubricant should have as low a viscosity as possible whilst possessing the necessary "body" to persist as a film between shaft and bearing. All the evidence goes to show that "body" and viscosity run closely parallel in the case of lubricating oils, but this does not apply to all liquids. It is proposed therefore to combine with viscosity the determination of surface tension by the measurement of the weight of a falling drop. If, as appears probable, heavy oils are iso-colloids, the disperse phase being a molecular aggregate suspended in a dispersion medium of simpler structure, then "body" may be merely the effect of the degree of dispersion exhibited by a particular oil. In no case has the chemical constitution of a component of a lubricating oil been established, but the chemical behaviour of these oils indicates that among the components are unsaturated hydrocarbons (possibly open-chain, but more probably naphthenic and polynuclear, or perhaps both types), saturated hydrocarbons (naphthenic and probably to some extent polynuclear, but not to any appreciable extent paraffinoid), and aromatic hydrocarbons (to an unknown and possibly a limited extent), together with small amounts of sulphur- and oxygen-containing compounds, the latter of



which can be isolated, and are probably the principal cause of gumming. It is the polynuclear naphthenes, above all the unsaturated components of an oil, which confer viscosity and "body." When crude petroleum is distilled, the tendency is for the lower fractions to be saturated, yielding good petrol and burning oil, whilst the higher fractions become more and more unsaturated. The wax is a survivor of the paraffin series, and is accompanied by large amounts of naphthenic saturated and unsaturated hydrocarbons. The unsaturated compounds constitute between 20 and 40% of most lubricating oils, and can be removed from the oil by agitation with concentrated sulphuric acid. The residual saturated hydrocarbon is invariably of a higher molecular weight, but lower in density and lower in viscosity, the acid having removed the chief lubricating components. A lubricating oil should therefore contain as large a proportion of unsaturated hydrocarbons as is compatible with not too great a susceptibility to oxidation, polymerisation, and gumming. Mineral lubricating oils have, however, very low iodine values, indicating low reactivity, a fact which is in keeping with the view that the unsaturated hydrocarbons are probably polynuclear and naphthenic in type. The tendency for these bodies to oxidise to gummy products is therefore very small, and probably it is the small amount (up to 3%) of oxygen-containing resinous matter which gives rise to gumming.—T. St.

*Rectification of benzol.* Hamer. See III.

*Importance of coke hardness.* Cochrane. See X.

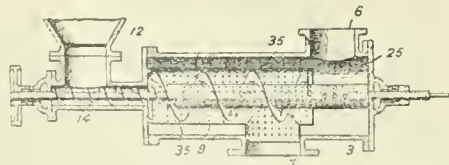
#### PATENTS.

*Pulverised fuel; Apparatus for burning* — T. B. Cram, Assignor to The Railway Materials Co., Chicago, Ill. U.S. Pat. 1,258,654, Mar. 12, 1918. Date of appl., July 19, 1915.

A VERTICAL cylindrical combustion chamber of refractory material is provided with a superposed concentric conduit of smaller cross-section, arranged so that an abrupt shoulder is produced at the junction. The pulverised fuel is injected by a concentric nozzle at the upper end of the conduit, and air is supplied through the annular passage between the nozzle and the conduit. A limited combustion takes place in the conduit, and the shoulder causes the formation of eddies surrounding the fuel jet, thus mixing part of the fuel with the surrounding air and preventing backward propagation of the flame. The lower end of the combustion chamber terminates at one end of a horizontal chamber into which a further supply of air is delivered by a jet at the end.—W. F. F.

*Finely-divided material [fuel] and air; Apparatus for mixing* — A. G. Kinyon and A. I. Jacobs, Assignors to Powdered Coal Engineering and Equipment Co., Chicago, Ill. U.S. Pat. 1,258,912, Mar. 12, 1918. Date of appl., June 21, 1917.

POWDERED fuel is supplied from the hopper, 12, to the rotary helical conveyor, 14, which delivers it to the perforated mixing chamber, 8, surrounded by a casing, 3. The conveyor shaft carries also a perforated tube, 25, opening into the mixing chamber



and also into the casing, and a helical rib, 35, is mounted on the tube. Air under pressure is supplied to the casing at the inlet, 6, passes through the perforated tube to the chamber 8, where it is

mixed with the powdered material, and both are then delivered at the outlet, 7.—W. F. F.

*Artificial-fuel composition and process.* R. F. Dalton, New York. U.S. Pat. 1,260,286, Mar. 19, 1918. Date of appl., Jan. 12, 1918.

Loose anthracite coal dust or screenings, 1 ton, and a liquid fuel of low viscosity and high boiling point, 10 galls., are mixed together so that the surfaces of the coal particles are merely wetted and the interstices are not filled with the liquid. A substantial proportion of the liquid fuel remains in the mixture up to the ignition point of the coal. The mixture is delivered to the fire in loose condition.—W. F. F.

*Coke ovens of the horizontal or approximately horizontal flue type.* H. B. Porteous and P. T. Allen, Redcar, Yorks. Eng. Pat. 114,936, June 9, 1917. (Appl. No. 8269 of 1917.)

In a battery of coke ovens separated by walls containing heating flues, the air for combustion passes through a horizontal passage in the upper part of each dividing wall to the back of the oven, and thence down a vertical passage in the wall to a pair of return flues in the wall, below the oven floor level. The heated air then rises through a central passage in each wall communicating at intervals in its length with horizontal combustion chambers on each side of it within the wall. The fuel gas is supplied to the combustion chambers, and the burnt gases pass back again through horizontal flues in the wall similar to the combustion chambers and alternating with them. The burnt gases then pass downwards through a vertical flue at the front of the wall to a pair of horizontal flues, one under each of two adjacent ovens. The air flues mentioned above are between these horizontal gas flues, and the air is thereby preheated as it passes through them in the opposite direction to that of the gas. The hot gases finally pass to discharge flues in the base of the setting. The gas and air supplied to each flue may be regulated at the front of the ovens.—W. F. F.

*Coke-oven foundation.* A. Roberts, Evanston, Ill. U.S. Pat. 1,259,133, Mar. 12, 1918. Date of appl., Feb. 23, 1915. Renewed June 22, 1917.

A BENCH of coke-ovens is provided with a line of "sole blocks" beneath the ovens, supporting the carbonising chambers and the heating walls. Each block has a longitudinal opening and a transverse opening communicating with each other in the form of a Maltese cross. The spent gases pass from a passage at each side of the line of blocks through the transverse openings to the passage formed by the longitudinal openings in the blocks. Transverse tunnels are formed below each of the last-mentioned passages and communicate with them by vertical connections. The spent gases finally pass by downward passages from each end of the transverse tunnels to discharge tunnels below and at each side of the bench of ovens.—W. F. F.

*Retorts or offtake pipes; Method of and means for equalising or maintaining an equilibrium or any predetermined condition of pressure in — in the manufacture of coal gas or in the volatilisation of other suitable substances.* A. A. Macintosh, St. Kilda, Victoria, and W. H. Hunt, Melbourne, Australia. Eng. Pat. 105,563, Apr. 10, 1917. (Appl. No. 5037 of 1917.) Under Int. Conv., Apr. 3, 1916.

In an apparatus for maintaining the pressure in retorts for coal-gas manufacture, coke ovens, or other apparatus from which gas is liberated by volatilisation, the gas offtake passes downwards into a closed chamber, its outlet being below the surface of water maintained at a desired level. A dividing partition extends downwards from the top of the chamber into the water, and an outlet pipe for gas is provided in the top of the same compart-

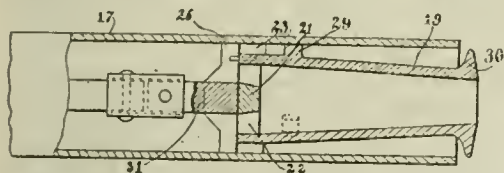
ment which contains the inlet pipe. A flow of gas from the retort is induced by an exhauster connected to the outlet, the gas passing through the water. Water is continually supplied to the chamber and overflows from the second compartment by a vertical pipe extending upwards into it, the level of the top of the pipe being adjusted as desired by a screwed sleeve. The second compartment is open to the air. Heavy tarry material is withdrawn from the chamber by a pipe at the bottom.—W. F. F.

[Gas] retort settings; Regenerative —. Drakes Ltd., and W. A. Drake, Halifax. Eng. Pat. 114,859, July 30, 1917. (Appl. No. 1534 of 1917.)

Is regenerative furnaces for heating retorts for the production of coal gas and the like, of the type in which the producer and regenerative flues are situated within the arch which supports a set of retorts, the hot gases from the producer at the front of the setting pass through a horizontal conduit to the back of the setting and then back through other horizontal conduits to a common chamber just behind the producer. The gases then pass through the regenerative passages to the back of the setting, and return to a transverse discharge tunnel behind the producer. The secondary air passes through transverse passages in the regenerator to longitudinal passages below and surrounding the hot gas conduit leading from the producer. These passages are connected in parallel in groups to common chambers at the back of the setting, the exit from each of these chambers being controlled by a damper. Other flues may be provided so that part of the secondary air may be conducted to the space below the grate in the producer.—W. F. F.

Gas producing and consuming plants. H. A. Dreifein and M. S. Eilan, Chicago. Eng. Pat. 114,474, Apr. 4, 1917. (Appl. No. 4873 of 1917.)

In an apparatus in which a flow of gas from a producer is induced and burnt by a jet of air, the air nozzle shown in the diagram is placed in the conduit leading to the burner nozzle. The inner tapered tube, 19, is closed at its rear end by a disc, 21, provided with equally spaced sector shaped openings, 22. A collar, 23, fitting the annular space between the tube, 19, and the outer sleeve, 17, is also provided with openings which are angularly between the openings in the disc, 21, and of equal area. A valve member, 26, is mounted on a spindle, 31, which can be operated externally by hand, and has projecting radial sectors which are adapted to



close either the outer or inner set of openings. The maximum flow of gas is induced when the openings, 22, are wide open, and complete closing of the openings, 23, is prevented by a stop so that a small flow of air takes place. This is obstructed by the lugs, 29, and by the collar, 30, thus producing eddies, so that back flow of gas and deposition of carbon are prevented. When the openings, 22, are closed, and 23 are open, the greater diffusion of the air jet induces a smaller flow of gas. In a modification, the nozzle is provided with peripheral openings only and fits closely into the surrounding tube, and a varying induction of gas is produced by moving the nozzle to and from the outlet of the surrounding tube.—W. F. F.

Sulphate of ammonia; Process and apparatus for the recovery of — from producer gases. F. Mellor, Glasgow. Eng. Pat. 114,658, Apr. 11, 1917. (Appl. No. 5096 of 1917.)

PRODUCER gas is passed through a mechanical washer throwing a minimum of back pressure, to remove dust, and then passed at a temperature of 95°–105° C. through a second mechanical washer through which liquor containing 3% of sulphuric acid is circulated. The gas is scrubbed by suitable rotating paddles, the liquor becomes saturated, and ammonium sulphate crystals are deposited in wells in the bottom of the washing tank. The crystals are discharged periodically and dried in centrifugal dryers, the discharge outlet from each well to the dryers being controlled by a valve. A tar separator may also be connected to the washer by a trapped pipe, and the liquor circulated through it.—W. F. F.

Explosive gaseous mixtures; Apparatus for burning —. C. E. Lucke, Assignor to Gas and Oil Combustion Co., New York. U.S. Pat. 1,259,029, Mar. 12, 1918. Date of appl., May 20, 1913. Renewed July 28, 1917.

The explosive gaseous mixture passes from a supply chamber through a large number of passages formed by partitions arranged so that the passages are sufficiently narrow to prevent backward propagation of the flame. The passages and supply chamber are cooled by a cooling device intersecting the partitions, and the mixture, which leaves the passages with a velocity greater than that of flame propagation, is reduced in velocity and burnt in a porous refractory medium.—W. F. F.

Petroleum; Process for refining —. M. J. Trumble, Los Angeles, Cal., Assignor to Simplex Refining Co. U.S. Pat. 1,259,171, Mar. 12, 1918. Date of appl., Sept. 27, 1909.

PETROLEUM is introduced successively into a series of bodies of water, and a heating medium is also introduced so as to heat the successive bodies of water to progressively higher temperatures, and to agitate the mixture of oil and water to produce an emulsion. The vapours produced at each temperature are condensed separately, and the residuum is removed from the last body of water.

—L. A. C.

Hydrocarbon oils; Apparatus for the treatment of —. R. Seeger, St. Louis, Mo., Assignor to New Process Oil Co. U.S. Pat. 1,259,786, Mar. 19, 1918. Date of appl., Sept. 23, 1916.

The apparatus consists of two chambers, the first containing a cracking retort, and the second, which is heated by gas issuing from the first chamber, having means for generating steam which passes to the cracking retort. Spaced outlets enable the heat in the second chamber to be controlled so that the temperature of the steam does not exceed 250° F. (about 120° C.). A chimney is connected with both the top and the bottom of the second chamber.—L. A. C.

Petroleum and the like; Method of and apparatus for distilling —. L. O. Sherman, East Chicago, Ind., Assignor to The Cleveland Trust Co., Cleveland, Ohio. U.S. Pat. 1,260,584, Mar. 26, 1918. Date of appl., Apr. 24, 1916.

CRUDE petroleum is heated under pressure in a still to 650°–850° F. (about 340°–450° C.). The vapours are condensed under pressure in two portions, and the portion of higher boiling point is led through a pipe immersed in a false bottom of lead in the still. The vapours thus produced pass into the vapour space of the still.—L. A. C.



[Hydrocarbon] oils; Process of distilling and cracking — J. A. Swaton, Los Angeles, Cal. U.S. Pat. 1,260,731, Mar. 25, 1918. Date of appl., Aug. 20, 1917.

HYDROCARBON oils are distilled at a progressively rising temperature and the vapours led off to a cracking zone maintained at a temperature 100°–400° C. higher than the distilling zone. The heavier hydrocarbons are separated from the lighter.

—L. A. C.

Gasoline; Process for making — F. C. Devericks, Assignor to L. S. Hornor, Clarksburg, W. Va. U.S. Pat. 1,260,970, March 26, 1918. Date of appl., Aug. 3, 1915.

From the product obtained by condensing casing-head natural gas, a hydrocarbon oil of approximately 85° B. (sp. gr. 0.651) is recovered. A portion of the uncondensed gas is absorbed in low-grade oil, thereby raising the gravity of the oil. The resultant oil is mixed with the hydrocarbons of 85° B., to produce a homogeneous liquid of approximately 70° B. (sp. gr. 0.700).—L. A. C.

Cementitious bituminous emulsion. W. M. Fraser, Woodbridge, N.J., Assignor to The Barbur Asphalt Paving Co., Philadelphia, Pa. U.S. Pat. 1,259,223, Mar. 12, 1918. Date of appl., Oct. 25, 1917.

ABOUT 75 galls. of a semi-liquid asphaltic residuum and about 43 galls. of an aqueous solution of rosin or rosin oil soap are mixed together until thoroughly emulsified and miscible in all proportions with water. The emulsion will not separate into its components within thirty days, and when tested with a drop of water is stained a light brown colour at the point of contact.—L. A. C.

Apparatus for bringing liquids and gases, vapours, fumes, and the like into intimate contact. Eng. Pat. 115,006. See I.

Processes and apparatus for obtaining lower from higher carburets of hydrogen [hydrocarbons]. Eng. Pat. 108,454. See III.

Process of making cyanide. U.S. Pat. 1,259,702. See VII.

Process of manufacturing lampblack [and hydrochloric acid, from natural gas]. U.S. Pat. 1,259,121. See XIII.

Process of preserving bagasse, etc., and making fuel therefrom. Process for economic steam production in cane-sugar factories. U.S. Pats. 1,258,888 and 1,258,889. See XVII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

Destructive distillation of birch; Effect of incomplete distillation on the yield of products in the — R. C. Palmer. J. Ind. Eng. Chem., 1918, 10, 260–262.

WHEN insufficiently seasoned or excessively wet wood is distilled, it is difficult to complete the distillation within 24 hours, and the proportion of incompletely charred pieces ("brands") is then large (8 to 10%). These brands are usually redistilled. In experiments in which yellow birch cord wood was distilled at about 340° C. in a retort taking a charge of 50 lb., one set of distillations was carried out to completion, whilst others were interrupted at definite points, and the brands made to absorb 33% of moisture and then redistilled. By stopping the distillation at the point when 10% of brands was present, the loss of wood alcohol was about 5% and that of acetate of lime 25%. The results also showed that formic acid is formed more rapidly than acetic acid, the total yield of the

former being obtained when about half of the wood remains as brands. Tar soluble in the pyroligneous acid is the next volatile product to be formed, and this is followed by the wood alcohol, and then by the oily tar which separates from the pyroligneous acid. In order to obtain the maximum yield of wood alcohol it is necessary to control the temperature of distillation just before the formation of the tar. The total yield of formic acid was about 30% higher, that of the acetic acid about 2.4% lower, the alcohol about 2.8% higher, the tar 20–25% lower, and the charcoal about 4% higher in the double distillation.—C. A. M.

Destructive distillation of hard wood; Influence of moisture on the yield of products in the —

R. C. Palmer and H. Clonkey. J. Ind. Eng. Chem., 1918, 10, 262–264.

CORD wood from beech, yellow birch, and hard maple, which had been seasoned for about 18 months, and similar wood seasoned for 4 to 6 months, were destructively distilled in a laboratory retort taking a charge of about 50 lb. In the uncontrolled distillations the maximum heat was maintained until the distillates showed a considerable proportion of tar, and the distillation then finished largely by means of the exothermic reaction; whilst in the controlled distillations the heat was checked on the first indications of tar, and then regulated so that the rate of rise of temperature was lower than in the uncontrolled distillations. It was found that moisture has a favourable influence on the yield of acetic acid when the distillation is thus controlled. In the case of beech and maple the best yields of acetic acid under these conditions are obtained from wood which has only been moderately seasoned. About 15% more was obtained from wood seasoned only for 6 months than from that seasoned for 18 months. In the case of birch, however, there is little difference in the results given by wood seasoned for the two periods. For the production of formic acid the best yields are obtained by the rapid (uncontrolled) distillation of wet wood, especially in the case of beech. An excess of moisture also tends to increase the yield of wood alcohol from beech, and to a less extent from maple, in uncontrolled distillation, but in the case of birch and of maple in controlled distillations the dry wood gives the highest yields of alcohol. As a rule the yields of tar and charcoal are increased by the presence of moisture, except in the case of beech for tar and of birch for charcoal.—C. A. M.

Destructive distillation of hard woods; Effect of catalysts [phosphoric acid] on the yield of products in the — R. C. Palmer. J. Ind. Eng. Chem., 1918, 10, 264–270.

WOOD chips were impregnated with aqueous solutions of phosphoric acid, and destructively distilled. The yield of acetic acid was thus greatly increased, e.g. from 5.13 to 13.85% in one experiment in which 2.45% of catalyst was added. The presence of the phosphoric acid also increased the yield of wood alcohol, in some experiments by 40 to 90%. Distillation of mixtures of wood and tar under pressure showed that the methoxy groups can be readily split off, with the formation of methyl alcohol. In one distillation under a pressure of 90 lb., about 20% of the theoretical yield of methyl alcohol was obtained. The metaphosphoric acid in the residue of charcoal could probably be quantitatively recovered by leaching. The use of a mixture of phosphoric acid and creosote as catalyst did not increase the yields of methyl alcohol, whilst sulphuric acid, as claimed in Ger. Pat. 185,934, reduced the yields of formic and acetic acids and prevented the formation of any methyl alcohol.

—C. A. M.

*Vegetable decolorising carbons [for sugar refining].*  
Schneller. See XVII.

#### PATENTS.

*Hot carbonising apparatus and process [for peat].*  
W. H. Boorne, London. Eng. Pat. 114,603,  
Dec. 22, 1917. (Appl. No. 19,019 of 1917.)

The apparatus comprises two horizontal tubes held together by a coupling, part of which forms a bracket inside the tube. Around part of the first tube is an annular space into which the peat is introduced, and from which it travels by an external pipe to the far end of the second tube. Within this tube is a steel casing capable of rotation, having its innermost end supported by the bracket. There is an annular space between the casing and the tube, the outer end of the space being closed by a bush. This arranged spirally on the outside of the casing impart a rotary motion to the peat as it passes through the annular space, which is heated both internally and externally by steam, there being a steam jacket around the tube. Leaving the annular chamber through openings in the bracket, the peat passes into the first tube, where it pre-heats the peat fed into the annular space around this tube, and leaves the apparatus through a spring-controlled valve.—L. A. C.

*Carbonaceous materials; Destructive distillation of*  
— F. M. Perkin, London, and J. West, Southport. Eng. Pat. 114,937, June 11, 1917. (Appl. No. 8337 of 1917.)

Is the continuous distillation of carbonaceous material, e.g., coal, in vertical retorts at high temperatures, a higher yield of coal tar and of aromatic hydrocarbons of lower boiling point, and a smaller yield of aliphatic hydrocarbons, is obtained by reducing the period of low-temperature distillation to a minimum. An inert gas such as stripped coal gas or producer gas is passed into the lower end of the retort at such a rate that it becomes highly heated in passing through the red-hot coke in the lower part of the retort. The hot gas carries off volatile matter from the partly coked zone, and finally passes through the cooler zone at the top where heat is given up, but not sufficiently to cause condensation of the volatile matter on the coal. The gas and distillates are then drawn off through a discharge conduit at the top of the retort.—W. F. F.

*Coal; System of carbonising* — J. A. Yeadon and T. Whitaker, Leeds. Eng. Pat. 114,971, Sept. 24, 1917. (Appl. No. 13,694 of 1917.)

Is a process for the continuous carbonisation of coal (including lignite, cannel, shale, peat, and analogous materials) the coal is very finely ground and is fed continuously from a hopper by a horizontal screw conveyor into the top of a retort. The screw does not extend to the edge of the retort, so that the opening is always sealed by a mass of fine coal filling the conduit. The retort is heated uniformly, and the coal falls freely through it so that it is continuously and instantaneously carbonised. The fine coke is discharged at the bottom of the retort by a similar screw conveyor, the discharge conduit being similarly sealed by a mass of powdered material.—W. F. F.

*Wood; Method of destructively distilling* — R. C. Palmer, Pensacola, Fla., Assignor to Govt. and people of U.S.A. U.S. Pat. 1,259,277, Mar. 12, 1918. Date of appl., March 23, 1917. (Dedicated to the public.)

Wood is intimately mixed with a solution of orthophosphoric acid, and any excess water removed by evaporation. The mixture is then heated in a retort to a temperature yielding the maximum amount of acetic acid and methyl alcohol, the vapours being

confined until they attain a pressure not exceeding 300 lb. per sq. in., and then allowed to escape under pressure to a condenser.—L. A. C.

*Electrode for luminous-arc electric lamps and process of making the same.* F. Buchanan, Syracuse, N.Y. U.S. Pat. 1,258,984, Mar. 12, 1918. Date of appl., June 19, 1911.

ABOUT 12 lb. of dry pulverised rutile is mixed intimately with the unevaporative residue of 1 quart of water-glass to form a plastic mass, the water of the moist mass is evaporated, the dry product pulverised, and then filled into an electric conducting shell of metal, such as iron, about  $\frac{1}{16}$ " in thickness, the shell being of sufficient substance to carry the current to supply a conductive metal at the arc during the consumption of the electrode, and to render the fused end of the conductor conductive when cold.—B. N.

*Carbon and method of manufacturing same.* R. D. Pike, Richmond, Cal. U.S. Pat. 1,262,251, Apr. 9, 1918. Date of appl., Dec. 21, 1915.

See Eng. Pat. 102,645 of 1916; this J., 1917, 127.

### III.—TAR AND TAR PRODUCTS.

*Benzol; Rectification of* — J. D. Hamer, Coke Oven Managers' Assoc., Apr. 27, 1918. Chem. Trade J., 1918, 62, 359.

The steam-heated still as commonly used, in which the temperature of the wash oil falls in its passage through the system, is considered wasteful and irrational compared with the newer types in which the oil is heated by steam coils on the trays. In normal plants, the temperature of the oil after the pre-heater should be within 10° C. of that of the steam. The quality of the crude benzol is regulated by the quantity of live steam used and the dephlegmating devices, which should be capable of regulation independently of the heat exchangers, and should not return their condensed products to the wash oil in the still. Wash oils should not dissolve any appreciable quantity of water. Economy of acid may be effected by separating the "once run" benzol into three fractions prior to washing if the carbon disulphide and unsaturated compounds have been largely removed by a slow and careful distillation of the fore-runnings. Chlorine reacts selectively with the thiophenes. If chlorine sufficient to combine with the thiophene and thiotolene be used these are preferentially chlorinated and the chloro-derivatives are so much less volatile than the benzene and toluene that a separation by distillation can be readily effected. (See also this J., 1918, 173 r.)

—H. J. H.

*Sulphite turpentine.* Schorger. See V.

#### PATENTS.

*Carburets of hydrogen [hydrocarbons]; Processes and apparatus for obtaining lower from higher*

— J. Oltmans, Hilversum, Holland. Eng. Pat. 108,451, Jan. 2, 1917. (Appl. No. 54 of 1917.) Under Int. Conv., Aug. 1, 1916.

HIGHER hydrocarbons are converted into lower hydrocarbons, e.g., coal tar or coal tar oil to benzol, petroleum to benzine, etc., by vaporising the oil and mixing the vapour with steam at 5–6 atmos., thorough admixture being ensured by passing the mixed vapours through a zig-zag length of piping. The vapours then pass through a mass of coal or coke previously brought to incandescence by ignition in a current of air. The products of reaction pass through a scrubber, and are then mixed with a further quantity of steam and pass into a condenser. The aqueous portion of the condensed liquid is separated and the remainder purified by distillation.

—L. A. C.



*Anthraquinonesulphonic acids; Process of making salts of* —. E. G. Griffin, Cliffside, N.J., Assignor to The Barrett Co. U.S. Pat. 1,260,535, Mar. 26, 1918. Date of appl., Mar. 22, 1917.

Sodium sulphate is added to a solution containing anthraquinonemonosulphonic and disulphonic acids and the comparatively insoluble sodium anthraquinonemonosulphonate filtered off. The anthraquinonedisulphonic acids are recovered from the filtrate.—L. A. C.

*Benzenesulphonic acid and alkali benzenesulphonate; Production of* —. J. W. Aylsworth, deceased, by A. M. Aylsworth and Savings Investment and Trust Co., East Orange, N.J. U.S. Pat. 1,260,852, Mar. 26, 1918. Date of appl., Jan. 23, 1917.

Excess of benzene is heated in a closed apparatus with sulphuric acid, the temperature at first being kept at 70° C., and then raised to approximately 120° C. after a number of hours. The mass is cooled below 70° C., and sufficient sulphuric anhydride added, with stirring, to convert the water of reaction into sulphuric acid. The mixture is heated as before, and the process repeated if necessary.—L. A. C.

#### IV.—COLOURING MATTERS AND DYES.

*Hamatoxylin africanum*. A. G. Perkin. J. Soc. Dyers and Col., 1918, 34, 99.

A SOUTH AFRICAN species of *Hamatoxylin* was discovered in 1909 in Great Namaqualand, and consists of a shrub 1—1.5 m. in height, with yellow flowers and smaller leaves than *H. campechianum*. Stems 0.5—1.25 in. in diameter consist mainly of a reddish-brown core surrounded by a nearly colourless layer of wood; this core, as in the case of logwood also, darkens on exposure to air. The colour reactions of the extract resemble those of Brazilwood rather than those of logwood; it gives a brown with ferric chloride, not a black, and a pale pink precipitate with lead acetate, not a blue. Dyeing trials with *H. africanum* on mordanted wool show colours similar to those obtained with Brazilwood, but in all cases duller in shade and poorer in the red constituent. On this account it is of no technical importance. The slight difference in dyeing properties of *H. africanum* and Brazilwood may be due to impurities present in the former, but, on the other hand, it might be expected that a methyl ether of hamatoxylin would dye shades almost identical in character with those of brazilin itself.

—J. F. B.

#### PATENT.

*Chlorinated products of xylene and new compounds and [triphenylmethane] dyestuffs therefrom; Manufacture of* —. R. B. Ransford, London. From L. Cassella and Co., Frankfurt, Germany. Eng. Pat. 114,645, Mar. 12, 1917. (Appl. No. 3609 of 1917).

SEE U.S. Pat. 1,219,166 of 1917; this J., 1917, 500.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Yellow pine chips; Experiments on the pulping of extracted — by the sulphate process*. O. Kress and C. K. Textor. J. Ind. Eng. Chem., 1918, 10, 268—270.

LONGLEAF pine chips from which the rosin and turpentine have been extracted are at the present time a waste material used for fuel. In experiments to determine the suitability of the extracted chips for the manufacture of "Kraft" paper, the material was coarsely screened and treated in an upright steel digester similar to that used in the ordinary

pulping process. The yield of crude pulp ranged from 35.3 to 43.8%, but it would be possible to increase this considerably by efficient screening of the chips. The oil condensed from the gases leaving the digester amounted on the average to 2.3 galls. of oil per ton of dry chips. The strength of the paper made from the pulp was lower than that of paper prepared from longleaf pine round wood, this being attributed partly to the small size of the chips, and partly to overheating in the preliminary treatment to remove the rosin and turpentine.

—C. A. M.

*Sulphite turpentine*. A. W. Schorger. J. Ind. Eng. Chem., 1918, 10, 258—260.

THE oil which collects on the surface of the liquid in the separator used in recovering sulphur dioxide in the manufacture of wood pulp is known as sulphite turpentine. It varies in colour from yellow to black, and is often impregnated with sulphur dioxide. From 0.36 to 1 gall. of this turpentine is obtained per ton of pulp derived from spruce or balsam, but hemlock does not yield this by-product. The present annual production of sulphite pulp in U.S.A. and Canada exceeds 1,500,000 tons, and assuming that only 0.5 gall. per ton was recovered, the annual yield of turpentine from this source should be about 750,000 galls. As was pointed out by Herty and Graham (this J., 1914, 1073), sulphite turpentine is mainly composed of cymene. The following results were obtained in the distillation of the oil from three different mills:—

Composition of wood.	Up to 175° C.	175°–176° C.	176°–177° C.	177°–178° C.	178°–182° C.	Residue.
%	%	%	%	%	%	%
Spruce, 100 ...	1.11	7.76	84.03	—	—	6.65
Spruce, 90 } Balsam, 10 }	7.66	15.18	39.00	33.66	—	3.74
Spruce, 65 } Balsam, 35 }	0.43	2.57	22.00	19.00	35.00	8.81

These results indicate that the highest yield of cymene is obtained when spruce is used as the raw material. Cymene from this source can be used in the production of toluene (see Schorger, J. Amer. Chem. Soc., 1917, 39, 2671; and Rittmann, Fr. Pat. 479,786 of 1915; this J., 1916, 1103). By fusing the sodium salt of the 2-sulphonic acid of cymene with alkali at about 300° C., dissolving the mass in water, and acidifying the solution and distilling it with steam, carvacrol was obtained, but the yields under laboratory conditions were very small, probably owing to the difficulty of controlling the temperature. (See also J. Chem. Soc., June, 1918.)

—C. A. M.

#### PATENTS.

*Textile materials or the like; Waterproofing compositions for* —. J. D. Williams, Rhyl. Eng. Pat. 114,494, Apr. 17, 1917. (Appl. No. 5406 of 1917.)

THE fabrics are treated with a composition consisting essentially of gum tragacanth associated with lead acetate and aluminium sulphate together or separately, to which may be added gold size, terebene, soap, and other ingredients.—J. F. B.

*Cellulose [wood pulp] and like fibrous materials; Apparatus for washing and airing* —. A. D. Berglund, Gulslogen, Norway. Eng. Pat. 114,459, Mar. 23, 1917. (Appl. No. 4248 of 1917.)

FOR washing and airing cellulose to free the mass from chemicals and gas after bleaching, the pulp is introduced into a tub the bottom of which slopes

towards the middle from both sides, and from one end towards a gutter, which communicates through a valve with a centrifugal pump. The tub is provided with a stirring device mounted longitudinally, and water is sprayed into the mass from sprinklers arranged above it and others which pass nearly to the bottom of the tub. The centrifugal pump delivers the pulp up a vertical pipe from which it is discharged over a spreading device and collects in a vessel which serves as an aerating apparatus. As it falls from the spreading device it comes in contact with air from a blower delivering the air through perforations below the pulp discharge orifice. From the aerating vessel it flows down a pipe to a distributing box, whence it is returned to the washing tub. The walls of the latter are lined with perforated filter tiles through which water escapes from the pulp.—J. F. B.

*Viscose; Process for the production of articles from* —. B. Borzykowski, Cleveland, Ohio. U.S. Pat. 1,260,508, Mar. 26, 1918. Date of appl., May 29, 1917.

FRESH viscose solution is passed through an extended path in a precipitating bath having a density above 18° B. (sp. gr. 1.134) and containing less than 10% of sulphuric acid or the equivalent amount of other acids.—J. F. B.

*Paper; Coated* —. H. R. Rafsky, Portland, Me. U.S. Pat. 1,260,448, Mar. 26, 1918. Date of appl., Dec. 11, 1915. Renewed Aug. 13, 1917.

The surface of a body paper is coated with "lime mud," ground to a uniformly fine state of division and containing a small amount of an alkali and an adhesive, such as casein, soluble in such alkali.

—J. F. B.

*Paper; Method and apparatus for drying coated* —. C. F. Pease, Assignor to The C. F. Pease Co., Chicago, Ill. U.S. Pat. 1,260,570, Mar. 26, 1918. Date of appl., Mar. 6, 1917.

The coated paper is first passed through a chamber containing combustion gases and hot air, and then through a chamber in which hot air is circulated. The apparatus consists of an entrance chamber containing a bank of heating tubes and communicating with the combustion gases chamber, an air heating chamber with several heaters, a conduit for combustion gases surrounding the air heating chamber and communicating with the entrance chamber, a transverse chamber communicating with the hot air chamber and the air heating chamber, and provided with means to increase the flow of combustion gases through the conduit.—J. F. B.

*Process of producing artificial leather.* U.S. Pats. 1,257,665 and 1,257,756. See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Cotton fabrics (tissues, yarns, and embroideries); Process for imparting new properties [transparent effects] to* —. Heberlein & Co. A.-G., Wattwil, Switzerland. Eng. Pat. 108,671, July 4, 1917. (Appl. No. 9666 of 1917.) Under Int. Conv., Aug. 11, 1916.

The material is treated with a solution of caustic alkali of a density above 20° B. (sp. gr. 1.15) at temperatures below 0° C. (e.g., -10° C.) until it acquires a transparent appearance which is not lost after washing and drying. Increased transparency is obtained by subjecting the treated fabric to the action of sulphuric acid of a density about 50.5° B. (sp. gr. 1.50), which may be cooled below 0° C. in

order to obtain a slower action. The treatments with alkali and acid may be repeated alternately until a sufficient effect is obtained. If desired, pattern effects may be produced by local treatments or by the use of a reserve.—J. F. B.

*Fabric pieces; Process and plant for the treatment of* —. [Weighting of silk goods.] F. J. B. Kniblichler and A. Fankhauser, Emmetbaden, Assignors to Wegmann & Co., Baden, Switzerland. U.S. Pat. 1,259,526, Mar. 19, 1918. Date of appl., Nov. 25, 1916.

SILK piece goods are prepared for the weighting treatment by forcing a liquid through the fabric rolled upon a metal drum, the drum is transferred to a drying machine, and the prepared fabric dried by centrifugal action. The fabric is then wound off the metal drum and rolled up on an acid-proof drum while it is simultaneously treated with stannic chloride; the acid-proof drum, together with the fabric, is placed in a bath of stannic chloride, then transferred to the drying machine, where the fabric is again dried by centrifugal action. The fabric is then unwound from the drum in a washing machine and again wound up on a metal drum, passing through a washing treatment on the way, after which it is dried by centrifugal action as before. The material is repeatedly treated with phosphate and stannic chloride until the desired charge of loading is fixed upon the silk.—J. F. B.

*Dyeing machines.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,259,839 and (B) 1,259,840, Mar. 19, 1918. Dates of appl., (A) Mar. 29 and (B) Mar. 31, 1917.

(A) A hollow reel, comprising non-perforated flanges extending outwards and connected by a perforated body, revolves within a receptacle. A foraminous member, which may be constructed of a series of "upwardly and downwardly extended pyramids," with passages for liquid through them, surrounds the perforated body and is spaced therefrom. Fabric to be dyed is wrapped around this foraminous member and enclosed by a foraminous belt of similar construction, capable of being tightly adjusted around the fabric. A series of tubes connects the inner face of the perforated body with a source of liquid supply from opposite ends of the reel, and means are provided within the tubes for breaking up and distributing the flow of liquid equally over the inner face of the body, the tubes and distributing means being integral with the reel and revolving therewith. Liquid or air is forced through the reel and through the fabric while the reel is in motion and is returned from the dyeing chamber into circulation. (B) The machine consists of a receptacle having non-perforated sides, a fixed foraminous plate extending across the receptacle and integral with the sides, and a similar movable plate above the first, these plates being composed of a series of pyramidal bars with openings between them for the passage of a liquid. A second receptacle connects the first with a source of liquid supply, and serves to secure an even pressure and distribution of the liquid on the lower face of the fixed foraminous plate, and an overflow pipe from the first receptacle above the movable foraminous plate conducts the liquid back to the second. In the liquid-chamber below the fixed plate is a member having its ends converging upwards and downwards, and this liquid-chamber is connected by a pipe with the liquid-chamber of a smaller vessel of similar construction, which serves as a testing vessel. Means are provided for circulating the liquid through both vessels simultaneously, for heating the liquid as it passes through the larger vessel, and for interrupting the flow through the smaller vessel for testing purposes, while continuing the flow through the larger one.

—J. F. B.



## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Nitric acid; Production of — from nitrogen oxides.* G. B. Taylor, J. H. Capps, and A. S. Coolidge. *J. Ind. Eng. Chem.*, 1918, **10**, 270—275.

LABORATORY experiments showed that by absorbing nitrogen peroxide in sulphuric acid containing chromic acid, nitric acid was produced directly, and when the absorbing liquid was maintained at 150° C., the mist emitted consisted of 95 to 100%  $\text{HNO}_3$ . The drawbacks to this process are the necessity for a hot absorbent and for the electrolytic recovery of chromic and sulphuric acids from the chromium sulphate. Attempts to prepare a strong mixed acid by electrolysis of solutions of nitrogen peroxide in strong sulphuric acid were unsuccessful owing to the efficiency of the process falling to a very low value as the concentration of the nitric acid in the solution increased. In experiments to determine the most suitable conditions for absorption of nitrogen oxides by water, the apparatus consisted of an ammonia saturator for producing a mixture of air and ammonia, an oxidiser for producing nitric oxide, a cooler composed of sets of U-tubes immersed in running water, and five stoneware towers, the first of which—the "oxidation tower"—was empty, whilst the others were packed with coke, leaving a reaction space of 1.9 cub. ft. at the top of each tower. The coke in each tower was flooded at intervals of about 30 mins. with 40% nitric acid. The results indicated that the absorption should be carried out at as low a temperature as possible. Artificial cooling of the air space in the oxidation tower would be advantageous. So long as the proportion of oxygen is kept near the theoretical requirement there is no pronounced increase in the loss of acid, but it is advisable not to allow the oxygen in the exit gas to fall below 5%. The oxidation tower and first two absorption towers absorb about 85% of the acid and the last two about 10%, and it is improbable that even doubling the number of towers would enable the last 5% to be recovered. Calculated on the basis of 90% efficiency and 10% of ammonia in the mixture supplied to the oxidiser, it would be necessary to have 25% additional air after oxidation to secure 5% of oxygen in the exit gas, and it would be preferable to introduce this before the mixture enters the reaction space before the first absorption tower. The experimental plant had the following capacity calculated on the basis of 0.285 grm.  $\text{HNO}_3$  per litre of nitrogen:—

Cub. ft. of gas per min. . . . .	1.25	2.5	4.0
Efficiency of absorption, % . . .	98	95	91
$\text{HNO}_3$ recovered in 24 hours, lb. . .	23	44.5	68

Part of the loss of acid is attributable to the formation of a non-absorbable acid mist. By placing a Cottrell precipitator between the third and fourth towers, the mist could be reduced to about 0.5%. The precipitator consisted of a  $\frac{3}{4}$  in. glass tube having in the middle a platinum wire connected with the anode of a "kenotron" high-tension current rectifier, whilst a coil of copper wire was wrapped round the tube and connected with the inner wall by means of platinum wire. The acid recovered from the precipitator was 15 to 25% nitric acid with very little nitrous acid. The formation of the mist was found to be promoted by increasing the velocity of the gas, reducing the excess of oxygen, and by increase of the strength of acid in the third tower. The controlling reaction in the process is—



and in order to allow sufficient time for this reaction as much space as possible should be given. Removal of packing material from the towers and the substitution of liquid sprays would increase the

oxidation space, whilst the use of the Cottrell precipitator would prevent loss of acid through the increase of mist thus produced. (See also *J. Chem. Soc.*, 1918, ii., 196)—C. A. M.

*Formic acid and hydrosulphites: New reaction of —.* E. Comanducci. *Boll. Chim. Farm.*, 1918, **57**, 101—102.

THE reddish-yellow coloration developed when formic acid or a liquid containing it is heated gently with concentrated sodium bisulphite solution (this *J.*, 1904, 1116) cannot be detected when the original solution is coloured. In this case the heating with the bisulphite is continued until gas-bubbles begin to escape, the liquid being then cooled and treated with a fresh, dilute sodium nitroprusside solution. A more or less intense green or blue coloration is produced according to the amount of formic acid present, hydrogen cyanide being evolved at the same time; the reaction is more sensitive if the nitroprusside solution is poured gently on to the other so as to form two layers. The composition of the blue precipitate corresponds with the formula  $\text{Na}_4\text{Fe}_2(\text{CN})_{10}$ . In this reaction, the formic acid reacts with the sodium bisulphite yielding sodium hydrosulphite, this giving the blue coloration with the nitroprusside.—T. H. P.

*Potash in the Pintados Salar, Tarapacá, Chile.* H. S. Gale. *Eng. and Min. J.*, 1918, **105**, 674—677.

THE Pintados Salar is one of the larger saline deposits in northern Chile. There is about 20 sq. miles of a hard rugged crust in the central or lowest part of the Salar surface which gradually changes outwards to a soft, crumbling deposit. The hard, rugged, weathered crust is the richer, potash-bearing portion of the Salar, the percentage of potassium present varying from nearly 3% up to about 7%, which exists in the form of glaserite; it consists largely of sodium chloride. Underlying the hard deposit is a loose granular deposit made up largely of glauberite, or in some places of gypsum.—W. G.

*Potassium salts: Extraction of — from the Pintados Salar.* R. C. Welts. *Eng. and Min. J.*, 1918, **105**, 678—679.

A LABORATORY method for the extraction of the potash salts from the Pintados Salar (see preceding abstract) is described as follows. The crushed saline material (100 grms.) is heated with 37 c.c. of water at 106° C. for 15 mins., with constant stirring, and then drained in a centrifuge and washed twice with 5.5 c.c. of hot water. The extract may be evaporated to dryness and the residue used as crude potassium salt (composition see column IV.) or the extract may be cooled to room temperature and the crystalline deposit separated (composition see column V.). The salts left in the mother liquor have the composition shown in column VI. and would be utilised again in a continuous process.

I.	II.	III.	IV.	V.	VI.
K	5.0	63.5	18.3	23.4	16.1
Na	32.2	13.1	24.3	19.4	26.4
Cl	41.3	18.9	45.2	35.0	49.5
$\text{SO}_4$	17.3	12.3	12.2	22.2	8.0

Column II. shows the composition of the Salar salts used, and column III. the percentage of each constituent extracted. There is a suggestion that advantage might be taken of the interaction of sodium nitrate and potassium chloride to form potassium nitrate, the Salar salts and caliche being treated together by the usual nitrate process so as to produce a mother liquor rich enough in potassium nitrate to yield that salt by simple crystallisation.—W. G.

*Line: Deterioration of — on keeping.* S. A. Woodhead. Analyst, 1918, 43, 161—163.

POWERED lime kept in a dry shed and turned frequently was converted to the extent of 43% into carbonate in 64 days; the same lime heaped outside and exposed to the air for 4 months was converted to the extent of 12% only. Lump lime stored in bags and protected from rain lost 38% of its calcium oxide in 4 months, whilst the loss in the same time amounted to 52% when the lump lime was heaped and exposed to the atmosphere.

—W. P. S.

*Carbon dioxide: Detection of — in the analysis of carbonates or oxalates.* O. F. Stafford. J. Amer. Chem. Soc., 1918, 40, 622.

To detect the presence of carbon dioxide in the gaseous products of the action of acids on minerals, a glass tube is inserted into the mouth of the test-tube; this tube is drawn out to a capillary fine enough to cause a drop of the reagent (baryta water) to remain in it, and not too fine to prevent bubbles of the gas from being drawn upward through the liquid. To make the test the gas from the test-tube is drawn through the baryta water by aspirating at the top of the inserted tube. The method may be made roughly quantitative for traces of carbonates or oxalates. Two such capillaries are used; the material to be examined is placed in one, and the second tube containing a drop of baryta water inserted into the mouth of the first and sealed to it by means of "Khotinsky" cement. Then by aspiration a drop of 30% sulphuric acid is drawn on to the material and the gas generated drawn through the baryta water. In the case of oxalates, 30% sulphuric acid mixed with potassium permanganate is used. The turbidity produced in the baryta water is compared with turbidities produced similarly from known amounts of carbonates or oxalates.—J. F. S.

*Cream of tartar; [Determination of] lead in commercial —.* A. J. Jones. Chem. and Drug., 1918, 90, 47.

The following methods yield accurate results for the determination of lead in cream of tartar which contains iron, aluminium, calcium phosphate, etc. *Direct method.*—Ten grms. of the sample is dissolved in 10 c.c. of hydrochloric acid (B.P., sp. gr. 1.052) and 20 c.c. of water, the solution is boiled, filtered, and the insoluble portion washed with 40 c.c. of hot water. The filtrate is treated with a small crystal of potassium iodide, boiled, cooled, and the liberated iodine is removed by the addition of N/10 thiosulphate solution, 3 drops being added in excess. After the addition of 4 c.c. of 2% hydrocyanic acid solution and 16 c.c. of concentrated ammonia, the mixture is boiled until it becomes practically colourless, then cooled, and diluted to 100 c.c. Seventy-five c.c. of this solution is now diluted to 100 c.c., sodium sulphide is added, and the coloration obtained compared with that of a standard prepared with the remaining 25 c.c. of the solution and containing a known amount of lead together with proportionate quantities of the reagents used in the test solution. *Ignition method.*—Five grms. of the sample is incinerated gently, the charred mass is digested with 50 c.c. of water, filtered, the insoluble portion washed, and ignited to a grey ash. This is treated with 4 c.c. of hydrochloric acid, the solution diluted with 8 c.c. of water, boiled, filtered while hot, and the filter washed with 30 c.c. of boiling water. To the filtrate is added a crystal of potassium iodide, and, after boiling, the iodine is removed by the addition of N/10 thiosulphate solution. The solution is then boiled with the addition of 2 c.c. of 2% hydrocyanic acid solution and a slight excess of

ammonia, cooled, acidified with hydrochloric acid, 5 c.c. of 20% tartaric acid solution is added, the mixture is rendered ammoniacal, diluted to 100 c.c., treated with sodium sulphide, and the coloration compared with a standard.—W. P. S.

*Sodium cyanide.* W. J. Sharwood. J. Ind. Eng. Chem., 1918, 10, 292—295.

For the past year cyanides have for the first time been sold on the basis of their cyanogen content, the purest technical sodium compound containing "sodium cyanide 96 to 98%" or "cyanogen 51 to 52%," whilst the old so-called "98% KCN" contained about 39% of cyanogen. Commercial fused sodium cyanide contains only a trace of potassium and not more than 2 to 4% of total impurities. It is largely prepared synthetically by heating sodium amide (produced by the interaction of ammonia and metallic sodium) with carbon. The most recent method of preparing it for the market is to cast it mechanically into egg-shaped cakes weighing 1 oz. For technical purposes the most convenient method of determination is to titrate the solution with N/20 silver nitrate solution (1 c.c.=5 mgrms. of commercial NaCN), with potassium iodide as indicator. The burette readings then also give directly "lb. per ton of solution." For the determination of sodium and potassium in mixed cyanides, in which chlorides and carbonates are the usual impurities, it is often practicable to evaporate the salt with hydrochloric acid, to ignite the residue gently, to weigh the mixed chlorides, and to titrate the chlorine in part of the residue. From these data the proportions of sodium and potassium may be calculated. Alkali sulphides may be detected by shaking the cyanide with fine lead carbonate suspended in water; or by dissolving it in a solution of silver nitrate containing less than 1 mol. AgNO<sub>3</sub> for 2 equiv. CN; or by dissolving it in mercuric chloride solution. In each case a black precipitate or dark coloration indicates the presence of sulphide. Sodium cyanide is not deliquescent, and is much more soluble than potassium cyanide. The concentration of dilute solutions may be approximately ascertained by means of the formula—

$$(\text{Sp. gr.} - 1) \times 200 = \text{Grms. NaCN per 100 c.c.}$$

Strong solutions of sodium cyanide differ but little in stability from those of potassium cyanide; if anything, they are somewhat more stable.

—C. A. M.

*Alkali cyanide; Manufacture of —.* S. Urano. Kogyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1309-1340.

CALCIUM cyanamide containing 19% of nitrogen, when fused with sodium chloride and charcoal in a small iron crucible, gives 70% of the theoretical yield of cyanide. The following conditions were found to be most suitable:—A charge consisting of 100 parts of calcium cyanamide, 100 of sodium chloride, 50 of charcoal, and 30 of calcium carbide is heated uniformly, without contact with air, at 940°—959° C. for 5 mins., the maximum temperature being attained gradually and the charge cooled quickly. The product contains about 20% of sodium cyanide, which is extracted with water or with 59% alcohol. When water was used as the solvent the yield of cyanide was 40% of the theoretical and when alcohol was employed the yield was 70%.

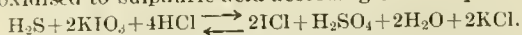
—J. F. B.

*Sulphides; Oxidation of — with potassium iodate.* R. S. Dean. J. Amer. Chem. Soc., 1918, 40, 619—620.

In continuation of previous work (see this J., 1915, 608), it is shown that with a large excess of iodate, and hydrochloric acid of any strength sufficiently high to prevent the hydrolysis of the iodine chloride formed, the whole of the sulphur is quantitatively



oxidised to sulphuric acid according to the equation



The estimation is carried out as follows: A solution of hydrogen sulphide is treated with the requisite excess of potassium iodate solution and hydrochloric acid (3–6 N) and the excess of iodate estimated by means of a standard iodine solution. The method has also been applied to the estimation of lead sulphide: the freshly precipitated sulphide is introduced into the iodate solution, hydrochloric acid added, and the titration completed as before. The results in both cases agree to within 0.1% of the theoretical value.—J. F. S.

*Magnesia-alumina-silica; The ternary system* — G. A. Rankin and H. E. Merwin. *Amer. J. Sci.*, 1918, 45, 301–325.

The ternary system, magnesia-alumina-silica, has been investigated thermally to determine the melting temperatures of the various phases, and microscopically to identify the crystalline substances produced. The boundary curves of the various phases have been determined and the quintuple points fixed. It is shown that the following crystalline substances are stable in contact with the liquid phase: periclase ( $\text{MgO}$ ), corundum ( $\text{Al}_2\text{O}_3$ ), tridymite ( $\text{SiO}_2$ ), forsterite ( $2\text{MgO} \cdot \text{SiO}_2$ ), clinoenstatite ( $\text{MgO} \cdot \text{SiO}_2$ ), spinel ( $\text{MgO} \cdot \text{Al}_2\text{O}_3$ ), sillimanite ( $\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$ ), and a ternary silicate,  $2\text{MgO} \cdot 2\text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2$ , which may be regarded as an end member in the cordierite series. This last compound is unstable at its melting point, and is considerably affected by solid solution. It has been observed in two forms: the  $\mu$ -form (unstable) crystallises from glass at temperatures below  $950^\circ\text{C}$ . and is transformed at a somewhat higher temperature into the stable  $\alpha$ -form. The properties of both forms, but more especially those of the  $\alpha$ -form, are very like those of the mineral cordierite. Seven quintuple points have been established which lie respectively at  $1345^\circ$ ,  $1425^\circ$ ,  $1460^\circ$ ,  $1370^\circ$ ,  $1360^\circ$ ,  $1375^\circ$ , and  $1700^\circ\text{C}$ . (Compare *J. Chem. Soc.*, 1918, ii., 199.)—J. F. S.

*Lead; The sub-bromide and sub-chloride of* — H. G. Denham. *Chem. Soc. Trans.*, 1918, 113, 249–256.

LEAD sub-chloride and lead sub-bromide have been prepared by a similar process to that employed in the case of the sub-iodide (this J., 1917, 336) by passing ethyl chloride or ethyl bromide vapour over lead sub-oxide at  $311^\circ$  and  $261^\circ\text{C}$ . respectively. Both compounds are grey, sparingly soluble, fairly stable in air, and readily oxidised by bromine water. The sub-chloride has a solubility of 2.2 milliequivalents per litre and the sub-bromide 0.4 milliequivalent per litre. The sub-salts are readily decomposed by acids into the normal salt and metal. In the preparation of the sub-chloride the reaction is very slow, and it is not necessary to carry it out in the presence of silica.—J. F. B.

*Holmium and dysprosium. Rare earths. V.* H. C. Kremers and C. W. Balke. *J. Amer. Chem. Soc.*, 1918, 40, 593–598.

The fractionation of the bromates of yttrium earths effects a rapid and efficient concentration of dysprosium and holmium. Starting with 12 kilos. of rare earth oxalates from gadolinite which were converted into bromates, three parallel series of 50 recrystallisations were carried out. The series showed a concentration of samarium, neodymium, and praseodymium towards the insoluble end, dysprosium and holmium in the central portion, and yttrium, thulium, and erbium towards the soluble end. At this point the dysprosium-holmium material was removed from the three series, combined, and made into three new series, A, B, and C. Series A consisted of the more soluble fractions, and contained dysprosium, holmium, erbium, and

yttrium. Series B contained the fractions richest in holmium and dysprosium, with small quantities of neodymium, praseodymium, erbium, and yttrium. Series C was composed of the less soluble bromates, and contained holmium, dysprosium, terbium, neodymium, praseodymium, and traces of erbium and yttrium. The three series were fractionated in parallel. The concentration of the earths in the different series was controlled mainly by the colour of the solutions. As the holmium and dysprosium became concentrated towards the less soluble end of series A these fractions were added to series B, placing them where the fractions showed a similar colour. From series C the more soluble dysprosium and holmium fractions were removed and added to series B in the same way. In the same manner yttrium and erbium, which collected in the soluble end of series B, were added to series A, and the neodymium and praseodymium from the less soluble end of B were placed in C. The three series were recrystallised 30 times, which caused most of the dysprosium and holmium to collect in series B. This series was eventually given a further 60 recrystallisations, which caused the holmium and dysprosium to concentrate very rapidly. A difficulty was experienced due to the repeated separation of basic cerium bromate as a brown sludge. It is therefore recommended that cerium be removed by the sodium sulphate method before the fractionation commences. Fusion of a mixture of the nitrates of samarium, yttrium, and holmium gave a rapid concentration of the holmium. A mixture which had an equivalent of 98.6 at the start gave after 31 fusions four end fractions with equivalents 154.3–155.2. Attempts to separate holmium and yttrium by this method failed, and it was only after the addition of samarium that the separation became possible.—J. F. S.

*Dysprosium; Purification and atomic weight of* — *Rare earths. VI.* H. C. Kremers, B. S. Hopkins, and E. W. Engle. *J. Amer. Chem. Soc.*, 1918, 40, 598–611.

Dysprosium material of a high state of purity has been further fractionated as ethyl sulphates and as bromates. Fractionation of the ethyl sulphates is more efficient than that of the bromates for the separation of dysprosium from neodymium, praseodymium, and terbium, but neither of the methods is efficient for the separation of holmium and dysprosium. For the atomic weight of dysprosium, determined from the ratio  $\text{DyCl}_3 : 3\text{Ag}$ , the value 162.52 was obtained. (See also *J. Chem. Soc.*, 1918, ii., 202.)—J. F. S.

*Diamond; Formation of* — C. A. Parsons. *Inst. Metals*, May 2, 1918.

Up to the present diamonds have only been produced by the more or less rapid cooling of intensely heated silver (black diamonds, Mansard, 1880–1881) or iron (transparent diamonds, Moissan). The author's experiments were designed to test Moissan's theory that pressure is the determining factor in causing the crystallisation of carbon. Experiments under varied conditions to obtain diamond from carbon or carbon compounds by the combined action of very high temperatures and pressures (see this J., 1918, 174 B) proved unsuccessful, and it is concluded that pressure alone will not account for the production of diamond through the cooling of an ingot of carbon-containing iron. The effective agent is occluded carbon monoxide, the escape of which is prevented by the solidification of the outer layers of the ingot. Experiments show that if the ingot is heated in a vacuum and freed from gas, no diamond is produced. In a low vacuum of 1 in. of mercury, the gas present being carbon monoxide, diamonds up to 0.7 mm. in length were formed. It is significant that the greatest quantity of diamond

found in an iron ingot is estimated to be  $\frac{1}{26000}$ th the weight of the iron and  $\frac{1}{1500}$ th part of the carbon present, this amount of carbon being almost exactly equal to that estimated to be originally present in the iron as carbon monoxide. The diamond is probably formed not far from the recalcination point at 600° C., for it is known that diamond is rapidly corroded by carburised iron just before setting. Little can yet be said about the actual mechanism of diamond formation. There is strong evidence to support the view that the action may take place solely between iron, carborundum, sulphur, and carbon monoxide, but it is quite possible that other metals or their carbonyls may be involved. The possible mode of formation of diamond in nature is discussed, and the suggestion of Bonney, that eclogite is the parent rock of South African diamonds, is found to be in agreement with the author's conclusions.—E. H. R.

*Iodine; Recovery of — from dilute residues.*  
H. F. Stevenson. Analyst, 1918, 43, 165–166.

The neutral or faintly acid liquid (e.g., waste liquids from Nessler and oxygen absorption tests) is treated with mercuric chloride to precipitate the iodine as mercuric iodide. The latter is treated, in a moist condition at about 45° C., with a mixture of nitric acid, 1, and hydrochloric acid, 5 vols., added in small quantities at a time. The reaction is complete when all the red mercuric iodide has disappeared. The mixture is then cooled, the iodine collected on a filter, and washed until free from mercury; mercuric chloride may be recovered from the filtrate. To convert the iodine into iodide, iron filings, 1, and water, 10 parts, are placed in a large flask and the moist iodine, 4 parts, is added gradually with shaking; the mixture is heated subsequently, treated with a slight excess of potassium hydroxide, the ferric hydroxide is separated, and the potassium iodide crystallised from the solution.—W. P. S.

#### PATENTS.

*Sulphurous anhydride; Manufacture of —.* M. Kaltenbach, Paris. Eng. Pat. 107,589, June 26, 1917. (Appl. No. 9171 of 1917.) Under Int. Conv., May 23, 1916.

Gases derived from the combustion of sulphur are passed through a column into which an alcohol, preferably ethyl alcohol, is sprayed, to absorb the sulphur dioxide, which is subsequently recovered by heating the solution and condensing the liberated gas. The alcohol for absorbing the gas is cooled previously by a portion of the liquefied gas. The solution which has been heated to expel the gas is used to heat a further quantity of solution, and the liberated gas is partly cooled by some of the cold solution prior to heating the latter to extract sulphur dioxide.—W. F. F.

*Sulphur dioxide or the like from gases containing the same; Recovering —.* Apparatus for segregating and recovering gases. U. Wedge, Ardmore, Pa., and P. A. Eustis, Milton, Mass. U.S. Pats. (A) 1,260,492 and (B) 1,260,493, March 26, 1918. Dates of appl., May 29 and July 10, 1915.

(A) In a continuous process for recovering sulphur dioxide from furnace gases, a solution of sulphur dioxide is obtained by treating a portion of the gas with a liquid. Part of this solution is then brought into contact with a fresh portion of the hot gas, or is heated by the same while spread over a large area under reduced pressure. This portion of gas, now cooled and enriched in sulphur dioxide, is treated with the remainder of the solution, and the sulphur dioxide is finally recovered from the latter by known means. (B) The apparatus consists essentially of a primary and a secondary

absorption tower, and a vacuum pan. Means are provided for conveying to the primary, a portion of the liquid discharged from the secondary tower, and for conveying another portion of the same liquid to the vacuum pan. The latter has a packing of porous material through which the liquid is caused to pass before entering the discharge conduit; and the outlet from the vacuum pump communicates with a compression pump.—W. E. F. P.

*Titanic acid; Production of —.* Det Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank, Christiania, Norway. Eng. Pat. 108,850, May 18, 1917. (Appl. No. 7213 of 1917.) Under Int. Conv., Aug. 9, 1916.

To produce titanic acid practically free from iron, a solution of quadrivalent titanium (and other metals, including iron) is boiled with a compound of trivalent titanium, the latter being added as such or formed by reduction within the original solution. For example, a solution of titanium and iron sulphates, obtained by treating ilmenite with sulphuric acid, is electrolysed with diaphragm and lead cathodes until all the ferric sulphate is converted into ferrous sulphate and about 5% of the titanium is reduced to the trivalent condition. The electrolysis is then discontinued and the liquid heated to boiling when, owing to the reducing action of the trivalent titanium, re-oxidation of the ferrous sulphate is prevented and a white precipitate of metatitanic acid produced, all the reduced titanium remaining in solution.—W. E. F. P.

*Ammonium perchlorate; Manufacture of —.* Aktiebolaget Carlit, Stockholm. Eng. Pat. 110,544, Sept. 24, 1917. (Appl. No. 13,721 of 1917.) Under Int. Conv., Sept. 25, 1916.

In the manufacture of ammonium perchlorate by double decomposition of ammonium sulphate and sodium perchlorate, the liquor is evaporated under reduced pressure at temperatures between 45° C. and 100° C., preferably commencing at 60° C. and rising to 80°–85° C. The sodium sulphate is thus deposited in an anhydrous state without admixture of perchlorate, and a strong solution of the latter is obtained.—W. F. F.

*Sulphate of soda from chrome residues; Manufacture of —.* E. P. Potter and Co., and E. P. Potter, Little Lever, Lanes. Eng. Pat. 114,501, Apr. 28, 1917. (Appl. No. 6009 of 1917.)

To recover sodium sulphate and chromium oxide from the residues obtained in the manufacture of sodium bichromate by the action of sulphuric acid on sodium chromate, the material is heated to incipient fusion with an excess of reducing agent (metallic iron, pyrites, sulphur, coal, etc.) and the mass lixiviated, the chromium being recovered as dissolved in water and the solution is treated with sulphur dioxide, hydrogen sulphide, or an organic compound. In either case chromium oxide or hydroxide is separated by filtration, and the colourless liquors are evaporated for the recovery of sodium sulphate by crystallisation. Alternatively, a mixture of the chrome residues with an excess of a lead compound, e.g. lead sulphate ("chamber bottoms"), is heated to incipient fusion and the mass lixiviated, the chromium being recovered as lead chromate by filtration, and the sodium sulphate by crystallisation.—W. E. F. P.

*Caustic potash; Removal of soda from feldspars or natural or artificial silicates or aluminates and manufacture of commercially pure —.* E. A. Ashcroft, London. Eng. Pat. 114,743, July 29, 1917. (Appl. No. 10,435 of 1917.)

A FELDSPAR, silicate, or aluminate, is pulverised, mixed intimately with an excess (say an equal



weight) of potassium chloride, sulphate, or nitrate, and heated for 2 hours to 800°–1200° C. out of contact with air and furnace gases. The soda in the felspar is thereby replaced by potash and the resulting sodium chloride is removed by solution in water. The residue is then mixed with 1–2 parts by weight of lime or magnesia or both, and the mixture digested with water or steam at 10–20 atmospheres and 200° C. for 2–6 hours. About 90% of the potash is thus obtained as commercially pure caustic potash, and is extracted by solution in water. If digestion is effected at 90 atmospheres pressure and 300° C., the extraction of the potash is practically complete. The residue may be converted into hydraulic cement clinker by drying and burning it.—W. E. F. P.

*Alkalies from silicate rock and other silicates: Method of obtaining* — F. A. Rody, Newark, N.J., Assignor to Metallurgical Co. of America, New York. U.S. Pat. 1,260,944, Mar. 26, 1918. Date of appl., May 18, 1915.

To obtain alkalies in soluble form from leucite, felspar, or similar silicate, the latter (100 parts) is fused to a glass-like, stringy mass which is then reduced to powder (100-mesh) and digested for about an hour with water and lime (6 parts) at a pressure of 100 lb. per sq. in.—W. E. F. P.

*Caustic alkalies; Powders for generating* — J. Armstrong, London, Eng. Pat. 114,896, Apr. 20, 1917. (Appl. No. 5559 of 1917.)

ALKALI carbonate and slaked lime or baryta, both in desiccated condition, are intimately mixed in suitable proportions.—W. E. F. P.

*Nitre cake; Method of utilising* — E. Hart, Easton, Pa. U.S. Pat. 1,258,895, Mar. 12, 1918. Date of appl., May 24, 1917.

An aqueous solution of nitre cake, of sp. gr. about 1.30, is cooled to about -40° C. by blowing cooled air through it, whereby sodium sulphate crystallises out as a granular mass.—W. E. F. P.

*Kelp-incinerator*. H. W. Judson, Long Beach, and H. A. Lowman, Wilmington, Cal., Assignors to Sea Products Co., Long Beach, Cal. U.S. Pat. 1,259,457, Mar. 12, 1918. Date of appl., Dec. 19, 1916.

The apparatus consists of a vertical, cylindrical casing having a flat bottom and an inclined top, the latter being provided with a flue and with a covered charging opening large enough to receive kelp from a pitchfork. A gas burner is mounted centrally within the casing at some distance above the bottom; while from the vertical sides horizontal kelp-receiving bars extend towards the centre, these being of different lengths and arranged out of alignment with each other from the level of the burner to the top of the casing. A door for removal of the ashes is provided immediately above the floor of the apparatus.—W. E. F. P.

*Potassium salts [from kelp]; Process of recovering* — F. K. Cameron, Salt Lake City, Utah, Assignor to Alaska Products Co., New York. U.S. Pat. 1,259,486, Mar. 19, 1918. Date of appl., May 9, 1917.

KELP is minced and strained to separate the coarser portions and produce thorough aeration, and an acid flocculating agent is then added to produce a clear liquor and a floating coagulum; the former is evaporated to crystallisation, and the latter returned for treatment with fresh kelp.—W. E. F. P.

*Cyanide; Process of making* — W. H. Wright, Indianapolis, Ind. U.S. Pat. 1,259,702, Mar. 19, 1918. Date of appl., Feb. 17, 1917.

In a process for recovering cyanogen from illuminating gas by absorption in a mixture of ferrous and alkali hydroxides, the mixture is prepared by treating an aqueous solution of ferrous sulphate with an alkali hydroxide, filtering, and suspending the precipitate in excess of aqueous alkali hydroxide. All the operations are conducted out of contact with air.—W. E. F. P.

*Borax ores; Process of treating* — H. Blumenberg, Assignor to C. T. Hinman, Los Angeles, Cal. U.S. Pats. (A) 1,259,717, (B) 1,259,718, and (C) 1,259,719, Mar. 19, 1918. Dates of appl., (A) Apr. 26, (B) and (C) Nov. 16, 1916.

(A) NATIVE calcium borate is digested with nitric acid and the mixture is filtered; or, if silicates are present, the mixture is evaporated to dryness and the residue extracted with boiling water. After separation from the calcium nitrate by crystallisation, the boric acid is heated with sodium nitrate, under reduced pressure and in presence of oxygen and aqueous vapour, to produce borax and nitric acid. (B) Calcium nitrate obtained as in (A) is heated in a closed chamber, and in presence of oxygen and aqueous vapour, to produce nitric acid and calcium oxide. (C) The native calcareous borate is heated with sodium nitrate to produce a borax glass which is subsequently digested with water for the recovery of borax and calcium nitrate.

—W. E. F. P.

*Nitrogen and mixtures of nitrogen and hydrogen; Manufacture of* — E. B. Maxted and T. A. Smith, Walsall, Eng. Pat. 114,663, Apr. 12, 1917. (Appl. No. 5125 of 1917.)

Air is passed over heated iron or copper until about 5% of oxygen remains, and is then mixed with hydrogen and passed through a highly heated retort filled with porous briquettes to remove the last quantities of oxygen. The water produced in the reaction is condensed and removed. The hydrogen may be replaced by a mixture of or a gas containing nitrogen and hydrogen, in which case a mixture of nitrogen and hydrogen is finally obtained.—W. F. F.

*Sulphur; Process of mining* — A. F. Lucas and G. M. S. Tait, Washington, D.C. U.S. Pats. (A) 1,259,536 and (B) 1,259,537, Mar. 19, 1918. Dates of appl., Dec. 27, 1916, and Mar. 9, 1917.

(A) THE sulphur in the deposit is ignited and partly burnt in a mixture of air and carbon dioxide in such proportions that sufficient heat is generated to melt a further quantity of sulphur. The molten sulphur is forced to the surface by the gas pressure. (B) Two concentric vertical passages are provided in the deposit, and hot air diluted with combustion products is passed down through the outer passage and back through the central passage so as to heat the central passage to about 450° C. A limited combustion of the sulphur is permitted and a further quantity of sulphur is volatilised, the sulphur vapour and combustion products being withdrawn through the central passage and the sulphur then condensed and removed.—W. F. F.

*Sodium and potassium bitartrates; Manufacture of mixtures of* — T. Gladysz, La Madragne de Montredon, France. Eng. Pat. 107,020, June 8, 1917. (Appl. No. 8224 of 1917.) Under Int. Conv., Mar. 12, 1915.

SEE Fr. Pat. 480,620 of 1915; this J., 1917, 137.

*Sulphurous anhydride; Manufacture of* — M. Kaltenbach, Paris. U.S. Pat. 1,260,681, Mar. 26, 1918. Date of appl., July 21, 1917.

SEE Eng. Pat. 107,589 of 1917; preceding.

*Ammonia; Purifying* —. C. Bosch, Assignor to Badische Anilin u. Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,261,900, Apr. 9, 1918. Date of appl., June 30, 1913.

SEE Eng. Pat. 25,259 of 1912; this J., 1913, 909.

*Condensers for acids*. Eng. Pat. 114,706. See I.

*Process and apparatus for the recovery of sulphate of ammonia from producer gases*. Eng. Pat. 114,658. See IIA.

*Furnace for roasting ores*. Eng. Pat. 114,953. See X.

*Roasting pyrites, ores, or the like in shelf-burners*. Eng. Pat. 114,954. See X.

*Process for the preparation of anhydrous sulphide of zinc*. Eng. Pat. 114,407. See XIII.

*Process of manufacturing lampblack [and hydrochloric acid, from natural gas]*. U.S. Pat. 1,259,121. See XIII.

*Process of making calcium cyanamide*. U.S. Pat. 1,258,747. See XVI.

## VIII.—GLASS; CERAMICS.

*Glass; Crystals of barium disilicate in optical* —. N. L. Bowen. J. Wash. Acad. Sci., 1918, 8, 265—268.

IN "barium crown" and other optical glasses rich in barium there is a tendency for colourless six-sided plates upwards of 3 mm. by 2 mm. by 0.03 mm. to form on devitrification. These plates have white outlines due to the sprouting of minute plates of the same material around the edges of the larger ones. The optical properties of these plates enable them to be identified as synthetic barium disilicate ( $\text{BaSi}_2\text{O}_6$ ) with a small amount of alkali disilicate in solid solution. The crystals have a melting point of  $1426^\circ\text{C}$ . (See also J. Chem. Soc., 1918, ii., 198.)—A. B. S.

*The ternary system: magnesia - alumina - silica*. Rankin and Merwin. See VII.

### PATENTS.

*Silicious firebrick and method of making the same*. H. A. Kennedy, Clearfield, Pa. U.S. Pat. 1,260,398, Mar. 26, 1918. Date of appl., Sep. 28, 1917.

PARTICLES of silica of substantial size and free from finely-powdered silica or sand are mixed with a binder such as fireclay and made into bricks.—A. B. S.

*Silica, plastic, gannister, common, fire and like bricks; Manufacture of briquettes*. —. T. Longstaff and A. D. Geairns, Crook, Durham. Eng. Pat. 114,676, Apr. 18, 1917. (Appl. No. 5441 of 1917.)

## IX.—BUILDING MATERIALS.

*The ternary system: magnesia - alumina - silica*. Rankin and Merwin. See VII.

### PATENTS.

*Impregnation of wood and the like*. P. Jolnerkevitch, Petrograd. Eng. Pat. 114,886, Apr. 18, 1917. (Appl. No. 5420 of 1917.)

ABOUT 0.1—1% of a sticky substance or viscous colloid, such as resin or other soap, starch, gum arabic, or glue is dissolved in water or in a solution of common salt, zinc chloride, or other salt, and 10—20% of an oily antiseptic such as creosote or resin is added. The mixture is heated to  $50^\circ$ — $90^\circ\text{C}$ ., and is emulsified under a pressure of 100—300

atmos. The emulsion is diluted with water or salt solution until it contains 2—10% of the antiseptic. The particles of the preservative are so finely divided that they readily and uniformly penetrate the pores of wood impregnated with the emulsion under vacuum or otherwise, and a much smaller proportion of preservative is required than with other methods. Diseased portions of wood which cannot otherwise be impregnated are efficiently treated with this emulsion.—A. B. S.

*Blast-furnace slag; Making articles of* —. J. B. Shaw, Alfred, N.Y., Assignor to I. H. Shaw and W. G. Tlee, Trenton, N.J. U.S. Pat. 1,259,304, Mar. 12, 1918. Date of appl., May 23, 1917.

ARTICLES of a dense structure are made by collecting the molten slag discharged from a blast furnace or cupola, allowing it to settle, and adding during the settling process an agent which will accelerate the liberation of gas from the slag. The slag is poured into moulds, a thin crust is permitted to form on the surface, and the article is removed from the mould whilst the bulk of the interior is still fluid. The solid crust is then heated to about the fusion point, whilst the article is surrounded by an envelope of heated solid material which excludes air and furnace gases, and the article is finally cooled slowly.—A. B. S.

*Cement; Method of making mixtures with water-reacting* — and cement product. C. M. Chapman, Douglaston, N.Y., and N. C. Johnson, Englewood, N.J. U.S. Pat. 1,259,462, Mar. 12, 1918. Date of appl., Feb. 23, 1917.

A WATER-REACTING cement is wetted and subjected to a heavy grinding action until the cement forms a somewhat viscous smooth liquid mass of highly hydrated colloid with an initial set of 1 to 4 hrs. The production of concrete in which this colloid material is used is also claimed.—A. B. S.

*Cement for pickling-vats*. C. E. Manby, Carnegie, Pa. U.S. Pat. 1,259,651, Mar. 19, 1918. Date of appl., Sept. 21, 1916.

AN acid-resisting mortar composed of melted sulphur and carbon is stiffened by the addition of glass in the form of fibrous wool.—A. B. S.

*Kieselguhr powder; Process of bonding* — and product thereof. C. S. Kinnison, Birmingham, Mich. U.S. Pat. 1,259,525, Mar. 19, 1918. Date of appl., June 27, 1917.

SODA ash is added to kieselguhr in the presence of moisture and the mixture is dried.—A. B. S.

*Grinding or disintegrating cement or other materials*. A. Smallwood, London. U.S. Pat. 1,262,115, Apr. 9, 1918. Date of appl., Apr. 18, 1916.

SEE Eng. Pat. 6329 of 1915; this J., 1916, 681.

*Cementitious bituminous emulsion*. U.S. Pat. 1,259,223. See IIA.

*Removal of soda from feldspars or artificial silicates or aluminates and manufacture of commercially pure caustic potash therefrom*. Eng. Pat. 114,743. See VII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Blast-furnace practice; Enquiry on* — in the United Kingdom. Report of Committee No. 2. Iron and Steel Inst., May, 1918. [Advance copy.] 18 pages.

THE report deals with the replies received to 14 questions circularised among British blast-furnace



owners. Questions 1—9 dealt with the influence of the mechanical and chemical conditions of the raw materials on furnace working. Except in the Cleveland district, where the ores are uniform and in lumps, ironstone of coarseness varying from lumps to fines has to be dealt with. The ores are rarely broken, and no great advantage is found thereby. Durham coke appears to be the best as regards hardness and uniformity. Limestone is rarely broken to less than 8 inches. It is generally agreed that variation of efficiency is traceable to changes in the mechanical condition of the burden, but opinions are divided as to the relative importance of chemical and mechanical conditions in their effect on regularity of working. Where the necessary storage capacity exists—as is rarely the case—and the ores are graded, this is usually found to be beneficial. The tenth question dealt with the influence on working of dimensions of the bell relative to stock line, and it was generally found that the area of bell should be about one-half that of the throat of the furnace at this point. Opinion is divided as to the merits of double bells in preventing loss of gas. Question 12 on the size and quality of firebricks used for the lining revealed a great diversity of practice in this connection, and also in the use of special devices for reducing wear at the upper part of the furnace as a result of the falling of the charge. Generally a less expensive but harder burnt brick is used in this part. Experience as to the use of spare gas for calcining ore is limited, but when tried advantage was generally claimed for it (Question 13). The cleaning of gas—wet and dry—was the subject of Question 11, but the replies will be dealt with in a separate report.

—H. J. H.

*Blast-furnaces; Fuel economy in —.* T. C. Hutchinson. Iron and Steel Inst., May, 1918. [Advance copy.] 15 pages.

PARTICULARS are given of the working of the blast-furnaces at the Skinningrove Ironworks since 1880. At that time the output was considerably less than the average of the furnaces in the Cleveland district. To discover the cause, experiments were made on a model furnace 1-48th full size, with all conditions, including the coarseness of the charge, reduced on the same scale. The model was cut vertically into two equal portions, each half being sheeted with glass so that the disposition of the charge could be followed. It was found that segregation of the charge occurred, coarser pieces collecting at the centre and periphery with an annular layer of fines between. On increasing the diameter of the bell equivalent to a change from 13 ft. to 15 ft. full size, the fines collected at the outside with lumps within. This would facilitate the passage of gases through the charge. On making the corresponding change on the actual furnace, the coke consumption fell from 23.94 cwt. to 22 cwt. per ton of pig iron. The fines formed an adherent carbonaceous lining to the furnace, which had a protective effect, and also contributed to fuel economy by diminishing radiation losses.—H. J. H.

*Coke hardness; Importance of — [in the blast-furnace].* G. D. Cochrane. Iron and Steel Inst., May, 1918. [Advance copy.] 12 pages.

The primary importance of coke hardness in blast-furnace working is affirmed and a machine—asccribed to Greville Jones—is described which permits of the measurement of the relative hardness. It consists of a drum turned at the rate of 18 revolutions per minute and having 2 angle-bars fixed inside, diametrically opposite, to turn the coke over. About 56 lb. of coke, in lumps up to the size of a man's fist, is dried on hot plates for 24 hours. Of this 28 lb. is charged into the drum, which is rotated for 1000 revolutions at 18 per

minute. The charge is then riddled over a  $\frac{1}{2}$ -in. mesh and the percentage left on the riddle noted. This is called the "hardness" of the coke. These hardness figures have been compared with the working of the furnace and the connection established. Satisfactory working can only be expected if the hardness number exceeds 74%, but no special advantage could be detected when hardness figures were above 76%. Cokes with a hardness of 70% have been found to lead to very unsatisfactory working. The soft coke is believed to react more readily with carbon dioxide in the upper part of the furnace where no useful purpose is served. Laboratory experiments on the effect of a current of carbon dioxide at 750° C. supported this view. Soft coke also becomes crushed, and as a result the working of the furnace is seriously impaired. (See also this J., 1918, 60 r.)—H. J. H.

*Blast-furnace bears.* J. E. Stead. Iron and Steel Inst., May, 1918. [Advance copy.] 42 pages.

THE "bears" or accretions of metal found below the hearth level of a number of blown-out blast-furnaces have been examined, and the nature and origin of the various bodies identified is discussed. Kish or graphite is prominent in all the bears examined, in considerable masses sometimes interstratified with metal. The conditions of slow cooling and absence of motion favour its separation and aggregation. Graphite in spheroidal particles is found in the mass of metal, probably having separated after solidification. Considerable bodies of mixed sulphides of manganese and iron have been found crystallised together in very varied proportions; in one case the proportion of manganese sulphide was as high as two-thirds. Slow cooling of the liquid metal is doubtless favourable to the settling out and collection of these mixtures. The red compound, titanium cyanonitride, is found in large crystals. A blue-coloured compound, titanium cyanide,  $Ti(CN)_2$ , has been identified in a bear at the works of Cochrane and Co., Ormesby. The compound was separated in an almost pure form, as cubical crystals harder than quartz or any steel. As a mineralogical name for this dicyanide or carbonitride of titanium, cochraneite is suggested. The discovery of iron oxides, iron silicate, and decarburised iron shows that oxidising conditions are possible even below the hearth level. The percolation of steam from the foundations is put forward as an explanation of the oxidation. The presence of such material would account for the discovery of metal closely resembling puddled iron in its malleability and ductility. The iron is frequently coarsely crystalline and columnar in structure. This columnar, dephosphorised iron, has been previously described (this J., 1914, 181). The phosphide eutectic has probably been squeezed out in the process of cooling of the partly liquid inferior owing to the contraction of the solid exterior. Minute crystals of the phosphide,  $Fe_3P$ , have been discovered in the body of the columnar ferrite crystals. Independent and idiomorphic crystals of phosphide and carbide of iron have been discovered in a drusy cavity of a bear. The existence of the carbo-silicide of iron and manganese (this J., 1901, 721) has been confirmed by the discovery of homogeneous idiomorphic crystals. The results of a crystallographic examination of these and the phosphide crystals, due to L. J. Spencer, are given.

—H. J. H.

*Blast furnaces; Copper tuyères for —.* A. K. Reese. Iron and Steel Inst., May, 1918. [Advance copy.] 6 pages.

FORGED copper tuyères for blast furnaces are recommended in preference to cast iron ones. The first cost is higher, but the advantages found fully warrant this.—F. C. Tu.

*Heavy steel industry in India; Technical aspects of the establishment of the —.* A. McWilliam. Iron and Steel Inst., May, 1918. [Advance copy.] 20 pages.

THE recent progress of the Tata Iron and Steel Co., Bengal, is described. The blast furnaces deal with an ore containing 63% of iron, a dolomite limestone as flux, and a local coke which contains 20% ash and 0.22% of phosphorous but only 0.5% of sulphur. The pig iron produced is therefore adapted for the basic process of steel manufacture, which is carried on in six 50-ton open-hearth furnaces. The company, which is now successfully supplying many of the Indian requirements in structural and rail steels, was slow at first to establish itself, mainly owing to the defective technical skill of the German staff prior to the war. It is now making "H.E. shell" steel of satisfactory quality. The addition of manganese is made while the metal is in the furnace rather than in the ladle. The firm owns its own manganese mines, and manganese losses are not a serious item. The author advocates higher limits in official specifications for manganese—up to 1%—which he finds beneficial rather than the reverse. Indian rail steels under wearing tests on the railways are proving satisfactory side by side with those from England. The author has made trials which encourage the view that it will be possible to produce acid steels when the opportunity is more favourable.—H. J. H.

[Steel] boiler plates; A cause of failure in —. W. Rosenhain and W. Hanson. Iron and Steel Inst., May, 1918. [Advance copy.] 12 pages.

A boiler plate, 1½ in. thick, broke after bending. The steel contained C 0.16, Si 0.08, Mn 0.62, S 0.03, P 0.048, and Ni 0.10%. The tests given by the steel as received and after annealing gave the following results:—

	Elastic limit. Tons per sq. in.	Yield point. Tons per sq. in.	Max. stress. Tons per sq. in.	Elongation. % on 1.3 in.	Reduction of area, %
As received ...	14.2	18.3	26.9	31.6	59.6
Annealed at 550° C.	15.4	18.7	27.6	34.5	59.1
Annealed at 900° C.	18.3	19.2	27.9	42.2	62.5

The modulus of elasticity was in each case practically identical at  $30 \times 10^6$  lb. per sq. in. The impact value of the steel as received, however, was only 0.75 kilogram-metre per sq. cm., which rose to 2.9 and 11.25 respectively after annealing at 550° C. and at 900° C. The fracture is ascribed to the low impact value of the plate as received, this being due to a coarse ferrite crystallisation in the carbonless bands of the plate. This crystallisation was the result of grain growth produced by slight deformation and subsequent low temperature annealing. This form of brittleness is eliminated by annealing at 900° C., and decreases as the thickness of the plate becomes less and the banded structure disappears. The brittleness produced by low temperature annealing after cold work increases as the degree of cold work decreases, as shown in the following table:—

Treatment.	Impact value. Kilogram-metres per sq. cm.
Normalised at 900° C. ...	10.46
Do. and annealed at 650° C. ...	9.04
Reduced 12.4%, and annealed at 650° C.	10.66
" 6.9% " " "	10.04
" 4.9% " " "	8.14
" 3.0% " " "	6.34

—F. C. TH.

[Steel;] Effect of mass on heat treatment [of —]. E. F. Law. Iron and Steel Inst., May, 1918. [Advance copy.] 15 pages.

To determine the effect of the mass of the sample of steel on heat treatment, heating and cooling experiments were made on 18-in. cubes weighing 14½ cwt. The steel used contained C 0.31, Si 0.19, Mn 0.74, S 0.022, P 0.032, and Ni 0.20%. The heating curve of this steel shows Ac1 at 730° C. and Ac3 at 820° C. On cooling, Ar3 occurs at 705° C. and Ar1 at 662° C. During the experiments three thermo-couples were inserted in each block, one ¼ in. deep, one half-way to the centre, i.e. 4½ in. deep, and the third at the centre. Each cube was heated to 900° C. in a furnace fired with Mond gas. The heating curves show that at 130 minutes from the time of charging, the metal at the centre and half-way to the centre was almost at the same temperature of 700° C.; but at this point the heat absorption due to Ac1 caused a greater lag at the centre, and the two temperatures did not again approximate until after another hour. After being in the furnace for about 4½ hours the cube attained a uniform temperature throughout. The cubes were then drawn and cooled respectively in air, in oil, in a water spray, and in water at 55° F. (13° C.). The air-cooled block cooled very slowly, and the heat evolution at Ar1 was clearly shown, especially at the centre. In the case of the oil-quenched mass the cooling was rapid, even at the centre, where the Ar1 point was very noticeable. The readings from the thermo-couples half-way to the centre, and ¼ in. deep respectively, gave not the slightest indication of any recalescence. The water-spray curves were similar to the oil-hardening ones, except in the lower ranges of temperature, where the cooling was in this case more rapid. The block quenched in water showed similar characteristics, but with a still greater rate of cooling. The essential difference between the oil and water quenching was found to lie in the greater time required in oil to cool down from about 500° C. Tensile tests were made on bars cut from the blocks. In the air-cooled block all yielded practically the same result, but in the other cases test-bars from the outer portion showed distinctly higher yield-points and maximum stresses than did test-bars from nearer the centre. The greatest difference was shown by the water-quenched block, the oil-quenched one, due to the "automatic tempering," showing the least. The properties of steels up to 250° C. are discussed in connection with an apparent transformation at 120° C.—F. C. TH.

*Damascene steel.* N. Belaw. Iron and Steel Inst., May, 1918. [Advance copy.] 22 pages.

DAMASCENE steel, which may contain from 1.1–1.8% carbon, is prepared in three ways: (1) by the old Indian method in a crucible from pure ore and charcoal; (2) by the Persian method from pure soft iron and graphite; (3) by a heat treatment like a very prolonged tempering. The characteristic "waterling" is referred to the original austenite dendrites distorted by the forming. Very slow cooling in the furnace from fusion gives a coarse crystallisation and a "milky way" of cementite globules. The spheroidal cementite comes from the slow cooling, or in the third process from the break-up of cementite cell walls and streaks at a very dull red heat. It is to this type of occurrence of the cementite that the remarkable ductile and elastic behaviour of this steel is ascribed. It is necessary that the forging should not be carried out at a temperature above the dull red.—F. C. TH.

*Steel springs; Brittleness produced in — by electroplating.* O. P. Watts and C. T. Fleckenstein. Amer. Electrochem. Soc., Apr.-May, 1918. [Advance copy.] 5 pages

BRITTLINESS was produced in steel watch springs



by electrolytic pickling in various electrolytes when hydrogen was evolved on the spring which served as cathode. After removing the grease from steel springs by treating with a hot alkaline solution, it was found possible to remove the "bluing" without causing brittleness by boiling in a 10% solution of ammonium citrate. When the springs were plated in cyanide baths, brittleness ensued whenever the evolution of hydrogen was detected; but the springs were not rendered brittle in silver or gold baths from which no hydrogen was evolved. Cyanide does not promote brittleness, which is probably caused, during either pickling or plating, by hydrogen. (Compare Thompson and Richardson, this J., 1917, 219.)—C. A. K.

*Steel; Determination of carbon in* —. L. Dufty. Analyst, 1918, 43, 167.

THE direct-combustion method (burning the drillings in oxygen) is more accurate and more rapid than the solution method (dissolving the drillings in copper-potassium chloride and burning the insoluble residue). The drillings must be thin, and the addition of an oxidising agent, such as bismuth oxide, is recommended, especially in the case of mild carbon steels. The solution method may be used for plain carbon steels, but is useless for high-speed steels and those containing molybdenum.

—W. P. S.

*Ferro-silicon; Methods for the commercial analysis of* —. E. M. Anger. Amer. Electrochem. Soc., Apr.—May, 1918. [Advance copy.] 8 pages.

CARBON is estimated by burning 0.5 gm. of the finely-ground sample, with addition of 2 grms. of fine mild steel turnings of known carbon content, to facilitate combustion. The solution of the finely-ground sample for analysis is effected by covering 0.5 gm. in a platinum dish with 10 c.c. of dilute nitric acid (1:1) and slowly adding hydrofluoric acid, drop by drop, until solution is complete. The solution is evaporated to dryness, and the residue dissolved in nitric acid. Manganese, calcium, magnesium, and sulphur are determined in this solution by known methods. Total phosphorus is precipitated by ammonium nitromolybdate; the small residue left undissolved when the solution is prepared as described above must be rendered soluble by fusing with sodium carbonate, as it contains a large proportion of the phosphorus. Phosphorus which can be liberated as hydrogen phosphide is determined by treating the ferro-silicon with 40% caustic soda solution, absorbing the hydrogen phosphide in bromine, and subsequently precipitating. For the determination of iron, hydrogen peroxide is used instead of nitric acid in preparing the solution, and the hydrochloric acid solution is reduced with stannous chloride and titrated with bichromate. When titanium and barium are to be determined, a small quantity of sulphuric acid must be added in the process of solution, otherwise these elements volatilise as fluorides. For the determination of silicon, ferro-silicon with up to 25% Si can be decomposed by carbonate fusion in a platinum crucible, but with higher proportions of Si, the platinum is rapidly attacked. Fusion with sodium peroxide in iron crucibles is recommended. An approximate method, accurate to 0.5%, is based on the evolution of silicon as tetrafluoride by the solution process described above, with subsequent ignition of the residue to oxides. A factor should be used to counteract the difference in the weight of aluminium and iron —C. A. K.

*Zinc or spelter; Effect of impurities on the hardness of cast* —. G. C. Stone. J. Amer. Inst. Metals, 1918, 12, 11.

THE hardness of a series of samples of commercial zinc of known composition was measured by the

load in lb. per sq. in. required to reduce the length of a test piece by 20%. Lead alone has practically no effect. Up to 0.1% iron has no effect, but in large quantities this metal renders spelter hard and very brittle. The hardness increases with the cadmium content, and it seems that cadmium is practically the sole cause of hardness in commercial spelter.—F. C. TH.

*Brass; Volatility of the constituents of* —. J. Johnston. J. Amer. Inst. Metals, 1918, 12, 15.

AFTER considering briefly the vapour pressures of some pure metals at temperatures up to 1150° C. (see this J., 1917, 1132), the relationship of the partial pressure of zinc in brasses of varying compositions to the vapour pressure of pure zinc at the same temperature is examined. This ratio is almost independent of temperature. The values of this ratio for the most important brasses are:—

Percentage of zinc ...	40	35	30	20
Ratio ...	0.27	0.21	0.15	0.05

The results indicate that, of the total zinc present in ordinary brasses, only a portion is present in solution as zinc itself, the remainder existing as a compound with copper. In the 70:30 brass half the zinc is free and half combined; in a 60:40 brass two-thirds is free. It is suggested that the compound is CuZn. Comparison of the partial pressures of the zinc in different brasses at their casting temperatures indicates that there is more danger of loss of zinc in pouring the high-zinc alloys than in those of lower zinc content, despite the higher temperature required in the latter case. With regard to the other constituents which may be present, the loss by volatilisation of copper and iron is negligible. In the case of lead the loss will be greater if it occurs free than if it is in solution. The loss of cadmium in practice is about four times that suggested by the volatilisation figures obtained, the discrepancy probably arising from the action of this metal as a deoxidiser.—F. C. TH.

*Bronze and brass; Non-metallic inclusions in* —. G. F. Comstock. J. Amer. Inst. Metals, 1918, 12, 5.

SPECIMENS were prepared by adding the element of which the oxide was desired to molten over-oxidised copper. Cuprous oxide, which is soluble in molten copper, occurs in the solid metal as an eutectic containing 3.5% Cu<sub>2</sub>O or 0.39% O. The other elements added all reduced the cuprous oxide. Tin oxide occurs in angular crystals or needles of very dark bluish-grey colour; it always appears to be pitted, especially after etching, and stands out in relief with dark black edges. It apparently does not occur in the form of cell walls. Zinc oxide in brass is distinguishable from tin oxide by its lighter colour, smoother surface, rounded form, and the absence of very dark outlines. This oxide may occur as films around the crystals. The oxides of manganese and phosphorus yield no typical inclusions. The phosphide present in phosphor-bronze may be distinguished from the  $\alpha\delta$  eutectoid by etching with 50% nitric acid, which attacks the eutectoid but leaves the phosphide perfectly bright. Etching with ferric chloride and ammoniacal hydrogen peroxide does not differentiate between these constituents, but in the unetched condition the phosphide is rather darker, and, being harder, stands out in relief. The sulphide present in phosphor-bronze has a bluish colour when unetched. No reagent has been found to attack it, whence it stands out clearly from the dark background of the etched alloy. This fact enables it to be distinguished from zinc oxide, which in colour and form it otherwise resembles. Alumina presents exactly the same appearance in bronze as in steel, i.e., small

black specks closely grouped without coalescing, irregular in shape, and with rough edges. Silicates occur in form similar to slag in steel. Sand occurs in smooth angular fragments, dark grey in colour, and with convex sides. Inclusions with concave sides consist of slag.—F. C. TH.

*Tungsten [in ores]: Rapid determination of —.*

F. W. Foote and R. S. Ransom, jun. Eng. and Min. J., 1918, 105, 836.

One gram. of finely powdered ore is mixed with 1.2 gm. of a mixture of calcium carbonate and sodium chloride (1:1) in a porcelain crucible and heated gently at first, then with the full heat of a Bunsen burner. The cold mass is transferred to a dish and digested on a water-bath with 40 c.c. of strong hydrochloric acid; 15 c.c. of nitric acid is added and the liquid concentrated to 15 c.c. after 15 minutes' digestion. It is diluted with 100–125 c.c. of boiling water, allowed to stand 2–3 hours, filtered, and the precipitate washed three times with 2% hydrochloric, and twice with 2% nitric acid. The residue of tungstic acid and silica is treated as usual.—W. R. S.

*Molybdenum ores and concentrates; Assay of —.*

H. C. Mabce. Eng. and Min. J., 1918, 105, 836–837.

From 0.5 to 1 gram. of the sample is treated with 2–3 c.c. of fuming nitric acid in a silica or platinum crucible of 50-c.c. capacity, and the mixture evaporated just to dryness. The residue is fused with 30 grms. of potassium bisulphate, the product leached with hot water and heated till completely dissolved, and the iron precipitated with ammonia. The washed precipitate should be dissolved and reprecipitated. Fifty c.c. of sulphuric acid (1:1) is added to the filtrate, which is passed through a 24-in. reductor into a 1-litre flask, using suction. The reductor contains 10-mesh zinc previously washed with hot sulphuric acid (1:1); 20 c.c. of 15% ferric alum solution and 20 c.c. of "titrating mixture" are placed in the receiver. After the molybdenum solution has been drawn through the reductor, the latter is washed at least four times with hot sulphuric acid (1:1); the solution is titrated warm with permanganate standardised against pure molybdenum trioxide, following the same procedure as in the actual determination.

—W. R. S.

*Chromium; Passivity of —.* A. H. W. Aten. Proc.

K. Akad. Wetensch. Amsterdam, 1918, 20, 812–823.

THE existence of chromium in active and passive forms is probably the cause of the divergent results which have been obtained in previous attempts to determine the electrode potential of chromium. New experiments made with electrolytic chromium and with chromium prepared by Goldschmidt's "thermit" process have given -0.47 volt for the potential of active chromium in contact with chromous sulphate solution with reference to the hydrogen electrode as standard. The attainment of this value requires the presence of hydrogen which acts as catalyst and produces the true contact equilibrium. (See also J. Chem. Soc., June, 1918.)—H. M. D.

*Sodium cyanide.* Sharwood. See VII.

*The lead electrode.* Getman. See XI.

*Electrolytic behaviour of manganese in sulphate solutions.* Van Arsdale and Maler. See XI.

PATENTS.

*High speed or special steels; Manufacture of tools from scrap of —.* Soc. Ind. des Métaux de Synthèse, Paris. Eng. Pat. 112,412, May 1, 1917. (Appl. No. 6156 of 1917.) Under Int. Conv., Dec. 30, 1916.

HIGH-SPEED steel scrap is heated in closed crucibles to about 1200° C., and potassium ferrocyanide added, preferably less than 5% of the mass treated. The temperature is then raised to 1800° C., and the metal cast, with or without a further small addition of ferrocyanide. It is claimed that the metal is free from blow holes. A small amount of barium or other suitable chloride may be added to aid the formation of slag.—C. A. K.

*Case-hardening of iron and steel articles; Carburisation and —.* W. Fennell, and The British Carbonizing Co., Ltd., Gloucester. Eng. Pat. 114,446, Jan. 27, 1917. (Appl. No. 1388 of 1917.)

THE articles embedded in carburising material are heated in a closed rotating chamber or muffle in presence of a gas (e.g., coal gas or carbon monoxide) under pressure (e.g., 15 lb. per sq. in.). The gas may be evolved from the charge of carburising material.—W. R. S.

*Case-hardening iron and steel; Process of —.* J. R. Messersmith, Cincinnati, Ohio. U.S. Pat. 1,260,787, Mar. 26, 1918. Date of appl., July 19, 1916.

THE case-hardening mixture is composed of potassium ferrocyanide, 1 part by vol., sodium chloride 1 part, and rosin,  $\frac{1}{2}$  part by vol.—C. A. K.

*Steel ingots; Casting of —.* A. Reynolds, Hove, Sussex. Eng. Pat. 114,466, Apr. 2, 1917. (Appl. No. 4746 of 1917.)

IN casting steel ingots, a reservoir for the molten metal, comparable in capacity with the mould, is interposed between the casting ladle and the mould. The reservoir is constructed with a depending spout of such length that the diminution of head under which the metal flows in casting is as small as is compatible with the conditions of casting. In casting a group of ingots, a reservoir is arranged above each mould, with its nozzle co-axial with the mould. In casting a large ingot, when the metal in the mould is at a level above the chill portion, the reservoir is raised, the spout throttled, e.g. by inserting a nozzle of smaller bore, and the casting completed at a slower speed. An inverted dish may be fixed at the lower end of the spout to prevent access of air to the contents of the mould.—T. H. B.

*[Iron] mill scale, and the like; Melting and de-oxidising of —.* A. Rollason, Long Eaton, Derby. Eng. Pat. 114,561, Aug. 7, 1917. (Appl. No. 11,307 of 1917.)

MILL scale or like material mixed with a suitable quantity of powdered carbon is added to 5–25% of its weight of molten pig-iron or steel scrap, and the mixture covered with a thick layer of non-oxidising material such as silicious slag. The temperature is then raised to melt the charge, the metal being de-carburised and converted into steel as usual.

—W. R. S.

*Iron; Process of smelting and purifying —.* G. R. Gehrant, Oak Park, Ill. U.S. Pat. 1,260,660, Mar. 26, 1918. Date of appl., Sept. 24, 1914.

THE ore is smelted and the smelted iron collected in the hearth of the furnace. Jets of air at sufficient pressure to penetrate the molten mass are



directed obliquely on to the surface of the iron. At the same time air at a lower pressure is blown into the furnace at a higher level.—C. A. K.

*Iron and steel; Treatment of — to prevent corrosion.* W. H. Allen, Detroit, Mich. U.S. Pat. 1,260,740, Mar. 26, 1918. Date of appl., Aug. 2, 1917.

ARTICLES of iron or steel the surfaces of which have been partly converted into iron phosphates, are immersed in an aqueous solution of a double cyanide of iron and potassium or other alkali metal.

—C. A. K.

*Iron, titanium, and silicon; Alloy of — and process for the production thereof.* N. G. Petinot, New York, Assignor to United States Alloys Corporation. U.S. Pat. 1,260,037, Mar. 19, 1918. Date of appl., Mar. 7, 1917.

AN alloy containing Fe 69–80%, together with titanium and silicon, in which the ratio of titanium to silicon is not less than 80:20 nor more than 85:15, is made in an electric furnace from a mixture containing excess of iron, and titanium and silicon in the above ratio, together with sufficient carbon to effect reduction: the silico-titanate slag is reduced by the iron of the bath.—W. R. S.

*Metals; Process for reducing — to a finely divided state.* Metals Disintegrating Co., New York, Assignees of E. J. Hall, Passaic, N.J., U.S.A. Eng. Pat. 109,258, Aug. 10, 1917. (Appl. No. 11,523 of 1917.) Under Int. Conv., Aug. 26, 1916.

METALS are ground whilst completely coated with or immersed in oil, in order to prevent oxidation or combustion of the metal. A modification includes the pulverisation of metals, under oil, in a ball mill, the oil being fed in and drawn off, intermittently or continuously, to prevent overheating, and to carry out with it material sufficiently ground.

—C. A. K.

*Spelter; Process for producing — and material employed for carrying the same into effect.* D. B. Jones, Chicago, Ill., Assignee of C. H. Fulton, St. Louis, Mo., U.S.A. Eng. Pat. 112,923, Aug. 10, 1917. (Appl. No. 11,498 of 1917.) Under Int. Conv., Jan. 22, 1917.

SEE U.S. Pat. 1,249,061 of 1917; this J., 1918, 95 A. Before distilling the zinc, the briquettes are preheated to expel volatile matter from the carbonaceous binding material. One or more of the briquettes may be interposed as a continuous resistor between the electrodes of an electric furnace, and a current passed in order to heat the material to a temperature sufficient to distil the zinc.

*Zinc-bearing material containing iron; Treatment of —.* A. G. Betts, Asheville, N.C. U.S. Pat. 1,259,594, Mar. 19, 1918. Date of appl., Apr. 21, 1916.

OXIDISED zinc ore containing iron is treated with an acid solution in such a manner as to leach out the greater portion of the zinc with a minimum of iron; the residue is again leached with an acid in presence of sulphur dioxide, the liquor treated with metallic iron, and electrolysed.—W. R. S.

*Gold alloys and the preparation thereof.* K. Shiga, Takachiho-mira, Japan. Eng. Pat. 114,447, Feb. 26, 1917. (Appl. No. 2814 of 1917.)

THE alloys contain gold (more than 80%) and nickel, or gold (more than 65), nickel (less than 20), and tungsten (less than 15%). The gold-nickel alloy is made by melting alternate layers of the metals in the form of thin sheets in a crucible. For the manufacture of the ternary alloy, a nickel-

tungsten alloy is produced first, and thin sheets of this are melted together with gold. Small quantities of cobalt, chromium, manganese, or silver may also be added. The alloys can be beaten into very thin leaves or drawn into fine wires. They are not attacked by acids (except *aqua regia*) or alkalis, and are not affected by high temperatures.—W. R. S.

*Furnaces for heating ingots.* G. J. Davies, Loughor, Glam. Eng. Pat. 114,570, Aug. 27, 1917. (Appl. No. 12,253 of 1917.)

THE "skid" or track along which ingots travel inside a heating furnace has a knee or bend at one end where the heat is greatest, and has imbedded within it a continuous pipe for the flow and return of cooling water.—T. H. B.

*Furnaces; Crucible and like —.* J. Gaunt, D. Brookfield, and J. Tylor and Sons, Ltd., London. Eng. Pat. 114,684, April 30, 1917. (Appl. No. 6093 of 1917.)

IN a crucible furnace the air blast is preheated by passing it through an annular chamber between the refractory wall and the casing of the furnace. This chamber is provided with baffles so as to give a circuitous up and down path to the air before it reaches the grate. A hinged tray of suitable construction is provided for the easy removal of the ashes.—C. A. K.

*Furnaces; Electric induction — [for melting and refining metals].* C. B. Foley, Ann Arbor, Mich., U.S.A. Eng. Pat. 114,853, Apr. 13, 1916. (Appl. No. 5442 of 1916.)

THE furnace is formed of a crucible, having in its lower part a circular hollow cross-member, the position of which is such, relatively to the bottom of the crucible, that a narrow passage is left between the lower part of the tubular member and the bottom of the crucible. The axis or axes of curvature of the inner walls of the crucible are different from that of the refractory cross-member, and the walls of the crucible slope outwards in an upward direction, so that the passage gradually widens on each side of the cross-member; the top of the crucible may be contracted inwards. A primary electrical winding, carried by a closed magnetic core, is arranged within the hollow member, which is completely surrounded by the molten secondary, and the plane of the core is so fixed that it includes the narrow passage corresponding to the smallest cross-section of the molten secondary. Several such cross-members may be provided to form channels, whereby the conditions of polarity and the temperature within the body of the secondary may be varied, the space between the different cross-members being greater than that between the members and the walls of the receptacle. The means for controlling the relative polarities of the electromagnet are adapted to produce zones of different temperatures on opposite sides of the inducing means, the hottest zone being at the bottom of the crucible, a relatively cooler zone between the cross-members and the walls, and the coolest zone between the inducing means. The induced or secondary current is made to circulate in series relation about the current inducing means, or about each inducing means by itself. The crucible is adapted to contain a high head of molten material, and the cross-section of the crucible area occupied by the secondary circuit is so proportioned that at any depth of secondary, measured vertically from the surface, except the greatest depth at the bottom of the crucible, the "pinch effect" forces tending to rupture the secondary will be substantially less

than the hydrostatic pressure at that depth. At the bottom of the crucible, the "pinch effect" forces are equal to the hydrostatic pressure when the apparatus is carrying its maximum input of current. The current density is therefore such as to obtain the most efficient circulation due to the so-called "pinch effect," and at the same time prevent the rupture of the molten material forming the induced secondary circuit.—B. N.

*Furnaces for roasting ores.* F. W. Harbord, Westminster. Eng. Pat. 114,953, Aug. 9, 1917. (Appl. No. 11,464 of 1917.)

Is a roaster of the tunnel type the hearth is divided into sections, each of which can be moved through the furnace supported on trolleys which travel on rails or water-cooled guides. The furnace may be horizontal, or slightly inclined to facilitate the movement of the hearths. The furnace itself is divided into two portions, one of which is muffled and heated externally. When once the furnace is heated up, the necessary heat is supplied to the other portion of the hearth by the combustion of sulphur in the ore. Both parts of the furnace form one continuous chamber and the sulphur gases produced are drawn through this chamber to the sulphuric acid plant, being unmixed with the products of combustion. During its passage through the furnace the ore is rabbled by the motion of the hearths past rakes fixed in the crown of the furnace and extending nearly to the hearth, preferably in the form of firebrick blocks or teeth set at suitable intervals. Air for the combustion of the sulphur may travel in the same direction as the hearths or in the opposite direction. The furnace is particularly suitable for sulphide ores such as blende, galena, or pyrites.—C. A. K.

*Roasting pyrites, ores, or the like in shelf-burners.* A. G. Bloxam, London. From Zellstoff-fabrik Waldhof, Mannheim-Waldhof, Germany. Eng. Pat. 114,954, Aug. 10, 1917. (Appl. No. 11,535 of 1917.)

The chief disadvantage of the shelf burner is that while a certain number of shelves are required for the complete roasting of one material, if a change is made to another grade of material the roasting may be complete at the middle shelf. This is overcome by fixing a second hopper to feed raw material to a lower shelf on which it mixes with partly burnt material arriving from the upper shelves.

—C. A. K.

*Melting furnace.* A. Fisher, Chicago, Ill. U.S. Pat. 1,258,666, Mar. 12, 1918. Date of appl., Dec. 4, 1916.

The furnace is in the form of a drum mounted to rotate on a horizontal axis. It is closed at one end by a removable door, and tapers from this end towards the opposite burner end, the portion adjacent the burner being cylindrical and forming a combustion chamber from which the heat is conducted by the lining of the drum underneath the metal resting on the sloping bottom. The lining of heat-resisting and heat-conducting material is in the form of parallel ring-shaped sections. The removable door carries a trunnion to form the axis of rotation, and from that end the rings forming the lining may be easily removed by tilting the furnace to a vertical position.—T. H. B.

*Annealing furnace.* I. W. Foltz, Chicago, Ill. U.S. Pat. 1,258,885, Mar. 12, 1918. Date of appl., Mar. 23, 1916.

The furnace comprises a fire-box and a combustion and annealing chamber, with air inlet conduits along the sides of the combustion chamber and

exposed to the combustion gases therein. These conduits communicate with the space formed between the side walls of the fire-box and false walls spaced apart from the side walls. Air-supply conduits are arranged at the opposite side of the combustion chamber with transverse distributing conduits at intervals along the top of the chamber communicating with the supply conduits. The roof of the fire-box has an air conduit with an outlet in the fire-box, and a baffle wall with a vertical passage therein depends from the roof, the passage communicating at its upper end with the fire-box, and at its lower end with the combustion chamber.—T. H. B.

*Electro-deposition of base metals (copper, zinc); Process for the —.* A. A. Lockwood, Merton Park, Surrey. Eng. Pat. 114,976, Oct. 3, 1917. (Appl. No. 14,286 of 1917.)

The base metals are deposited electrolytically from solutions derived from the treatment of ores and the like, in a vacuum or under reduced atmospheric pressure varying in degree, mainly according to the complexity and density of the electrolyte. By this means the need for clarification of the solution is generally obviated, polarisation is prevented, evanescent insulation (due to gas bubbles) is avoided, and the metal is deposited more rapidly and in a denser form.—B. N.

*Lead chloride and chlorine gas; Process of treating ores to produce —.* J. L. Malm, Denver, Colo. U.S. Pat. 1,258,800, Mar. 12, 1918. Date of appl., Dec. 12, 1914.

An ore containing lead and other sulphides is treated with chlorine gas in the presence of moisture at a temperature sufficient to convert the lead into sulphate, with hydrochloric acid as a by-product. The lead sulphate is converted into chloride and extracted by means of hot concentrated brine, and the solution cooled to precipitate lead chloride, from which lead and chlorine are recovered, the latter being used again in the initial stage of the process.—T. H. B.

*Concentrating ores; Process of and apparatus for —.* J. M. Callow, Salt Lake City, Utah, Assignor to Metals Recovery Co., Augusta, Me. U.S. Pat. 1,258,869, Mar. 12, 1918. Date of appl., Feb. 4, 1915. Renewed, Aug. 1, 1917.

The ore pulp is fed into a vessel provided with an overflow at its upper end, a tailings-separating chamber with an outlet in its lower portion, and a pipe through which a current of gas is passed upwards through the pulp, by which the coarse particles are separated from the fine; the latter overflow into a flotation cell, where they are subjected to flotation.—W. R. S.

*Copper alloys containing strontium; Composition of —.* F. C. Frary, Niagara Falls, N.Y., and S. N. Temple, St. Paul, Minn. U.S. Pat. 1,258,886, Mar. 12, 1918. Date of appl., Mar. 31, 1916.

An alloy, harder than copper, consists mainly of that metal, with a small amount of strontium. —T. H. B.

*Copper [sulphide] and like ores; Smelting —.* T. W. Cavers, Copperhill, Tenn. U.S. Pat. 1,259,467, Mar. 12, 1918. Date of appl., June 29, 1917.

A blast of air carrying a relatively small amount of carbonaceous fuel, and material containing matte-forming metal, is blown into the lower part of the blast-furnace at a point somewhat above the slag level.—T. H. B.



*Metals [lead, zinc, iron, and arsenic]; Process of extracting — from ores, &c.* C. C. Nitchie, Depue, Ill., Assignor to New Jersey Zinc Co., New York. U.S. Pat. 1,258,934, Mar. 12, 1918. Date of appl., Nov. 19, 1915.

ORES, fumes, or other metalliferous materials are heated with ammonium sulphate at such a temperature that zinc and iron are converted into sulphates and ammonia is driven off, with some of the arsenic, then heated to higher temperature (e.g., 600°–650° C.) to decompose iron sulphate and expel more arsenic without decomposing the zinc sulphate, the latter being afterwards dissolved out, leaving a residue containing lead, and practically free from zinc.—T. H. B.

[Vanadium.] *Method of recovering values from by-products and ores.* J. J. Boericke, Merion, Pa. U.S. Pat. 1,259,595, Mar. 19, 1918. Date of appl., July 20, 1914. Renewed Aug. 7, 1917.

VANADIFEROUS fume is condensed, roasted with an alkali, the melt leached, and the solution neutralised and precipitated with calcium hypochlorite.—W. R. S.

*Magnetic separator.* J. G. Sekinger, Ford City, Pa., Assignor to the Pittsburgh Plate Glass Co., Pittsburgh, Pa. U.S. Pat. 1,259,788, Mar. 19, 1918. Date of appl., July 21, 1915.

AN inclined discharge spout, open on its upper side, has a casing of magnetic material mounted on its end, the casing being provided with an opening in alignment with but of smaller cross-section than the outlet of the spout. The sides of the casing are substantially parallel to the axis of the spout. Surrounding the opening in the casing is an annular groove opening downwards, and dividing the casing into two annular pole pieces, and in the groove is a "winding." The lower face of the casing is at right angles to the axis of the winding and adapted to receive and support magnetic material which passes through the opening.—W. R. S.

*Tin-scrap and the like material; Art of dclanning —.* O. K. Zwingenberger, Tompkinsville, N.Y. U.S. Pat. 1,260,119, Mar. 19, 1918. Date of appl., June 8, 1911. Renewed Aug. 15, 1917.

TIN scrap is treated with chlorine in the presence of moisture, whilst being protected from the influence of the moisture by means of excess of another gas, e.g., sulphur dioxide, capable of reacting with the water and chlorine to form hydrochloric acid and another product.—T. H. B.

*Scrap material; Process of recovering metal from —.* J. W. Brown, Lakewood, Ohio. U.S. Pat. 1,260,312, Mar. 26, 1918. Date of appl., July 15, 1914.

SCRAP material which on heating forms distinct globules or masses of molten metal is subjected to pressure at a temperature above its melting point, thus squeezing out the molten metal from the dross. The dross may be mixed with a flux and heated and stirred.—C. A. K.

*Alloys; Method of recovering metals from —.* J. H. Gillis, Assignor to British America Nickel Corporation, Ltd., Toronto, Canada. U.S. Pat. 1,260,661, Mar. 26, 1918. Date of appl., Sept. 4, 1917.

AN electric current is passed from an alloy (e.g., nickel-copper) electrode to a suitable cathode through an electrolyte containing a salt of the metal (nickel) to be deposited, the polarity of the electrodes being reversed at such intervals and for

such periods as to avoid deposition on the cathode of any undesired components (e.g., the copper) of the alloy anode.—C. A. K.

*Tungsten ingots; Apparatus for welding —.* C. A. Pfanstiehl, Waukegan, Ill., Assignor to Pfanstiehl Co., Inc., North Chicago, Ill. U.S. Pat. 1,260,940, Mar. 26, 1918. Date of appl., July 12, 1915.

IN an electric furnace for sintering or welding tungsten ingots, the latter are supported between a pair of tungsten electrodes having squared and parallel end faces, to which powdered carbon may be applied. The electrodes, which are water-cooled, are of the same cross-section as the ingot, and are held firmly against the squared ends of the latter by springs or other resilient means.—C. A. K.

*Sheet lead and the like; Making —. Extruding lead and the like.* J. Stratton, Bowdon, and E. A. Claremont, High Legh, Cheshire, Eng. Pats. 114,902 and 114,903, Apr. 25, 1917. (Appl. Nos. 5838 and 5839 of 1917.)

*Sulphide ores; Separation of metallic —.* J. Hebbard and R. J. Harvey, Broken Hill, N.S.W., Assignors to Minerals Separation North American Corporation. U.S. Pat. 1,260,668, Mar. 26, 1918. Date of appl., Aug. 26, 1914.

SEE Eng. Pat. 19,373 of 1914; this J., 1915, 1017.

*Sulphide ores; Separation of mixed —.* J. Hebbard, Broken Hill, N.S.W., Assignor to Minerals Separation North American Corporation. U.S. Pat. 1,261,810, Apr. 9, 1918. Date of appl., Apr. 12, 1915.

SEE Eng. Pat. 5650 of 1915; this J., 1916, 639.

*Copper; Electrolytic deposition of — from acid solutions.* F. E. Studt, London. U.S. Pat. 1,260,803, Mar. 26, 1918. Date of appl., July 18, 1916.

SEE Eng. Pat. 108,688 of 1916; this J., 1917, 1100.

*Copper and other metals; Apparatus for electrolytic production of —.* M. Perreux-Lloyd, Boulogne-sur-Seine, Assignor to M. V. Bailly, Paris. U.S. Pat. 1,262,248, Apr. 9, 1918. Date of appl., Apr. 14, 1917.

SEE Eng. Pat. 110,474 of 1917; this J., 1917, 1249.

*Furnace for calcining copper and like ores.* J. G. Squire, Newcastle-upon-Tyne. U.S. Pat. 1,262,126, Apr. 9, 1918. Date of appl., Feb. 14, 1917.

SEE Eng. Pat., 101,998 of 1916; this J., 1916, 1264.

*Zinciferous ores; Treatment of —.* J. H. and P. McP. Gillies, East Camberwell, Victoria, Australia. U.S. Pat. 1,262,190, Apr. 9, 1918. Date of appl., Jan. 22, 1917.

SEE Eng. Pat. 112,336 of 1917; this J., 1918, 94A.

*Classifier.* U.S. Pat. 1,260,135. See I.

*Recovering sulphur dioxide or the like from gases containing the same. Apparatus for segregating and recovering gases.* U.S. Pats. 1,260,492 and 1,260,493. See VII.

*Making articles from blast-furnace slag.* U.S. Pat. 1,259,304. See IX.

*Cement for pickling-rats.* U.S. Pat. 1,259,651. See IX.

## XI.—ELECTRO-CHEMISTRY.

*Storage battery capacities; Calculation of —.* C. W. Hazlett. Amer. Electrochem. Soc., Apr.-May, 1918. [Advance copy.] 13 pages.

The capacity of any Faure or pasted-type starting battery cell between the limits of the 5-minute and 100-hour rates of discharge, and of all commercial-type vehicle plates between the 30-minute and 12-hour rates, is a definite single function of the time, i.e.,

$$\log I + f(T)(\log T) = K,$$

where  $I$  is the current,  $T$  the time,  $K$  the capacity in ampere-hours, and  $f(T)$  a definite function of the time. The formula

$$E = 1.85 - \frac{0.448}{\log T},$$

where  $E$  is the final voltage and  $T$  the time rate of discharge in minutes, is offered as a scientific and practical universal final-voltage equation. The charging and finish-charging rates of any cell should be a definite time rate of discharge, and the 7- and 18-hour rates respectively are recommended. The development of an alignment chart and circular slide rule for calculating storage battery capacities at various time rates of discharge is shown.—B. N.

*[Electric] cell; An experimental carbon —.* S. A. Reed. Amer. Electrochem. Soc., Apr.-May, 1918. [Advance copy.] 6 pages.

A primary battery is described in which the electrolyte is fused borax containing manganese dioxide, the negative is broken carbon in contact with a graphite grid floating on the melt, and the positive poles are strips of gold foil. The carbon is placed inside a cylindrical shell of silicious material (a crucible with the base cut off), and supported in a shallow clay roasting dish containing the melt. The electrolyte in contact with the outer gold strips is oxidised, whilst the inside electrolyte is reduced by the carbon anode, forming a mixture of carbon dioxide and monoxide. An average voltage of 0.8 is produced at a temperature of 935°–990° C.—B. N.

*Lead electrode; The —.* F. H. Getman. J. Amer. Chem. Soc., 1918, 40, 611–619.

Cast lead, electrolytic lead, and lead amalgam give the same potential, measured in a saturated solution of lead chloride at 25° C., but lead strips which had been immersed in Heller's solution (400 grms.  $\text{Pb}(\text{NO}_3)_2$ , 1000 c.c. water, and 100 c.c.  $\text{HNO}_3$  of sp. gr. 1.16) exhibited a potential 8 millivolts higher than the others. This confirms the existence of an allotrope of lead. From the results it is calculated that the heat of the reaction  $\text{Pb} + \text{Hg}_2\text{Cl}_2 = \text{PbCl}_2 + 2\text{Hg}$  is 21,840 cals. and the heat of formation of lead chloride is 84,400 cals. (See also J. Chem. Soc., 1918, ii., 184.)—J. F. S.

*Manganese in sulphate solutions; Electrolytic behaviour of —.* G. D. van Arsdale and C. G. Maier. Amer. Electrochem. Soc., Apr.-May, 1918. [Advance copy.] 16 pages.

The current efficiencies, voltages, and conditions affecting the nature of deposits formed during the electrolysis of manganese sulphate solutions have been studied, and it is shown that manganese may be deposited at the cathode from neutral solutions with high current efficiency (from 80–90%) at voltages from 3 upwards. The deposit is powdery, and ordinary expedients for improving deposition do not give better results to any extent. The deposi-

tion of manganese is diminished by increase of acidity, and is entirely prevented by 0.36% of free sulphuric acid. Manganese dioxide may be deposited at the anode at nearly 100% efficiency, and, though the effect of acid is to decrease the efficiency in cold solutions, there is much less effect on heating to 65°–75° C. Manganese dioxide is deposited from warm acid solutions containing more than 5% of manganese sulphate in the form of a dense, black, lustrous film, at current densities up to 275 amperes per sq. m. with very high deposition efficiency. Impurities such as ferrous iron and thionates cause a decrease in current efficiency according to the amount present.—B. N.

*Passivity of chromium.* Aten. See X.

## PATENTS.

*[Electrolytic] gas generators.* C. F. Euler, Topeka, Kans., U.S.A. Eng. Pat. 114,491, Apr. 13, 1917. (Appl. No. 5272 of 1917.)

SEVERAL horizontal electrodes are supported by a non-conducting tank near its base, and are spaced vertically apart, extending substantially the length of the tank. A wire screen electrode is supported a suitable distance above, extending over substantially the entire area of the tank. (See also U.S. Pat. 1,234,319; this J., 1917, 1053.)—B. N.

*Gas; [Electrolytic] process of making —.* F. A. Osowski, Detroit, Mich. U.S. Pat. 1,259,898, Mar. 19, 1918. Date of appl., Sep. 28, 1917.

The electric circuit is provided with spaced terminals forming a gap, and a measured quantity of acidulated water is fed, drop by drop, between the terminals to bridge the gap. Each drop, forming a conductor for the current, is decomposed by the current flowing through it.—B. N.

*Depolariser; Battery —.* M. E. Holmes, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,259,099, Mar. 12, 1918. Date of appl., Sep. 10, 1914.

PIROLUSITE is purified and its depolarising action, when used in electric batteries, increased, by heating it in a hydroxide solution under pressure.—B. N.

*Electrode; Storage battery —.* T. Spencer, Philadelphia, Pa., Assignor to National Carbon Co., Inc., Cleveland, Ohio. U.S. Pat. 1,260,472, Mar. 26, 1918. Date of appl., Aug. 21, 1915.

A porous framework of a phenolic condensation product is formed as a filling in a grid, and active material is introduced into the pores of the filling.—B. N.

*Electrode holder for electric furnaces.* P. Fischer, Chicago, Ill. U.S. Pat. 1,259,842, Mar. 19, 1918. Date of appl., June 8, 1917.

A HOLDER for an electric furnace electrode is provided with a tubular shank surrounded by a water jacket, the cooling water passing around and through the bore of the tubular shank. To one end of the electrode is fastened a metal reinforcing member comprising a tubular portion surrounding one end of the electrode and another portion extending beyond the end of the electrode, both the latter portion and the adjacent end of the electrode being provided with a continuous tapering bore. The end of the holder is tapered to correspond with the bore and fits into the latter, the tip of the holder-end being provided with a raised thread engaging adjacent portions of the electrode.—J. F. B.



*Furnaces; Phase distribution for electric* —. T. F. Baily and F. T. Cope, Assignors to The Electric Furnace Co., Alliance, Ohio. U.S. Pat. 1,260,298, Mar. 26, 1918. Date of appl., Jan. 31, 1918.

A HIGH-TEMPERATURE heating and a low-temperature reheating furnace are each furnished with a pair of resistance elements and supplied with current from a 3-phase supply. One of the supply wires is connected to each of the resistance elements in the heating furnace, the second supply wire to one of the resistance elements in the reheating furnace and to both elements in the reheating furnace, whilst the third wire is connected to the other resistance element in the heating furnace and to both elements in the reheating furnace.—B. N.

*Electric furnaces.* J. L. Dixon, Detroit, Mich., U.S.A. Eng. Pat. 111,103, Oct. 15, 1917. (Appl. No. 14,932 of 1917.) Under Int. Conv., Oct. 14, 1916.

SEE U.S. Pat. 1,214,763 of 1917; this J., 1917, 389.

## XII.—FATS; OILS; WAXES.

*"Acid oils" and soap stocks; Determination of moisture and total fatty matter in* —. E. R. Bolton and C. Revis. Analyst, 1918, 43, 158—161.

"SOAP stock" is the product obtained when edible fats and oils are treated with alkali to remove free fatty acids; it contains a varying proportion of neutral oil entrained in the soap. If this product is decomposed with a mineral acid or nitric acid, the mixture of neutral oil and fatty acids obtained constitutes "acid oil." To determine moisture in "acid oil," a strip of filter paper, 15 in. long and 1.25 in. wide, is pleated in  $\frac{1}{2}$  in. folds, loosely curled, placed in a weighing bottle, and dried for 48 hours over sulphuric acid in a vacuum desiccator. The bottle and coil are then weighed, 20 to 30 drops (0.7 to 1.0 gm.) of the oil are dropped on the upper edges of the coil, and, after re-weighing, the bottle and its contents are again dried in the desiccator as before. The desiccator should be kept at such a temperature (say 30° C.) that the oil remains liquid. To determine total fatty matter in soap stock, from 1 to 2 grms. of the sample is mixed with 20 c.c. of hot water and sufficient hydrochloric acid to decompose the soap, the mixture is heated gently under a reflux condenser until the fatty acids separate in a clear layer, the mixture then cooled, and the fatty layer separated by means of a mixture of equal parts of ether and petroleum spirit. The ethereal extract is evaporated, the residue of fatty substance dried in the water-oven, and weighed. A special flask is described in which the soap stock is decomposed and extracted.—W. P. S.

*Shark liver oil; A highly unsaturated hydrocarbon in* —. M. Tsujimoto, Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 953—1017, 1069—1098. (See also this J., 1916, 609, 1121; 1918, 98; also Chapman, this J., 1917, 392.)

SEVERAL samples of shark liver oil obtained from 27 species of sharks caught in Japanese waters have been investigated. The oils have been classified in two groups: (1) those with sp. gr. below 0.9000 at 15°/4° C., and (2) those with sp. gr. above 0.9000 at 15°/4° C. The shark liver oils of the first group, most of which are derived from sharks of the family *Squalidae*, are all rich in the hydrocarbon squalene (*loc. cit.*),  $C_{30}H_{50}$ , whereas the oils of higher sp. gr. are free from squalene, with the exception of Karasuzame oil (10%) and Fujikujira (*Elmopterus lucifer*) oil (1%). The hexahydrohalogen compounds of squalene are easily

obtained by passing the dry hydrogen halide into a cooled ethereal solution and recrystallising from acetone. The hexahydrochloride sinters at 110° C. and melts clear at 125° C. with separation of hydrogen chloride; this compound may be used for the detection of squalene, even as little as 1%. The hydrobromide is very similar; the hydriodide is not so easily obtained, and melts to a pink liquid at 108°—109° C. The oils of the first group are characterised by high iodine values and low saponification values. By eliminating the elements of hydrogen chloride from the hexahydrochloride by treatment with zinc and acetic acid, a viscous liquid corresponding to  $C_{30}H_{50}O_2$  is obtained. On destructive distillation a hydrocarbon resembling myrcene is obtained. The specific viscosity of squalene in Ostwald's viscosimeter is 14.3 at 15° C. and 12.2 at 25° C., and the time of flow by Redwood's viscosimeter is 91 secs. at 15° C. and 73 secs. at 25° C. For the determination of hydrocarbon (squalene) in shark liver oil, 50 grms. of the oil is distilled under 10 mm. pressure, and the acid value of the distillate, calculated as fatty acid, is deducted from the weight of distillate, the remainder being the approximate quantity of hydrocarbon. Aizame (*Centrophorus* sp., described previously as *Squalus mitsukurii*) oil is exceptionally rich, containing 84.8% of squalene. —J. F. B.

*Dog-fish liver oil.* A. C. Chapman. Analyst, 1918, 43, 156—158.

THE liver of the common dog-fish (*Squalus acanthias*) yields, when minced and steamed, about 40—50% of its weight of a pale yellow oil, which becomes semi-solid at 10° C. owing to the separation of solid glycerides. Two specimens of the oil prepared from different batches of liver had the following physical and chemical characters, the analyses having been made after the oils had been cooled and the solid substance separated:—Sp. gr. at 15°/15° C., 0.9175, 0.9186; refractive index at 20° C., 1.4755, 1.4749; optical rotation in 100 mm. tube, —, —1.45; saponif. value, 161.0, 168.3; iodine value (Wijs), 123.3, 123.0; free fatty acids (as oleic acid), 0.33%, 0.42%; unsaponifiable matter, 32.94%, 34.8%; brominated glycerides insoluble in ether, 19.25%, 24.95%. The proportion of unsaponifiable matter in the oil appears to depend on the age and condition of the fish.—W. P. S.

*Lipase; Action of acids on castor seed* —. G. Kita, Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 1—4.

ARMSTRONG (this J., 1905, 1242) found that the lipolytic activity of castor seed from which the fat had been removed was destroyed by a quantity of acid equal to that which exercised the most favourable effect on the enzymic hydrolysis of fats, and hence concluded that the favourable effect of the acid cannot be due merely to its stimulating action on the enzyme. According to the author the explanation of this apparent anomaly lies in the fact that whilst lipase rendered active by a small quantity of acid loses its activity in presence of an additional quantity of acid, in presence of fats the sensitiveness of the activated enzyme to acid is markedly diminished.

*Gossypol, the toxic principle of cotton seed.* Carruth. See XIXA.

### PATENTS.

*Oily materials; Process of purifying* —. W. P. Schuck, Assignor to Superior Oil and Process Co., Portland, Oreg. U.S. Pat. 1,250,072, Mar. 19, 1918. Date of appl., Aug. 31, 1916.

FATTY oils are purified by distilling off at least some of the impurities in a current of hydrogen, in

absence of hydrogenising catalysts, whilst maintaining the oil at about 300° C., a free exit for the gas carrying off the volatilised impurities being maintained.—A. de W.

*Press plates for oil expressing presses and analogous apparatus.* A. W. French, Plqua, Ohio, U.S.A. Eng. Pat. 111,855, Dec. 3, 1917. (Appl. No. 17,894 of 1917.) Under Int. Conv., Dec. 2, 1916.

*Soap; Apparatus for and method of rapidly ageing* —. De N. W. B. Young, Narberth, Pa., U.S.A. Eng. Pats. 114,878 and 114,879, Apr. 13, 1917. (Appl. Nos. 5242 and 5243 of 1917.)

See U.S. Pats. 1,246,515 and 1,250,540 of 1917; this J., 1918, 47 A, 130 A.

*Separating liquids of different specific gravity insoluble or difficultly soluble in each other, such for instance, as an emulsion or mixture of grease and water.* Eng. Pat. 114,921. See I.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Sulphite turpentine* Schorger. See V.

#### PATENTS.

*Sulphide of zinc; Process for the preparation of anhydrous* —. P. Comment, Dijon, France. Eng. Pat. 114,407, May 9, 1917. (Appl. No. 6596 of 1917.) Under Int. Conv., Mar. 28, 1917. Addition to Eng. Pat. 106,489 (this J., 1918, 273A).

Zinc persulphide to be used for reducing anhydrous zinc sulphate to sulphide as described in the chief patent may be obtained by dehydrating the zinc pentasulphide resulting from the action of an alkali or alkaline-earth pentasulphide on a zinc salt. Sulphur may also be present, with the persulphide, and effects a similar reduction. The addition of an alkali sulphate may be avoided by calcining the mixture for 3 hours at 500°–600° C., and then for 1 hour at 800°–900° C. The process may also be modified by the substitution of zinc sulphite for zinc sulphate. Barium sulphate may also be added in any desired proportion to obtain lithopones.—W. F. F.

*Red pigment ["Crimson antimony" or "antimony vermilion"]; Manufacture of* —. E. F. Morris, Roby, and T. N. C. Nevill, St. Helens, Lancs. Eng. Pat. 114,581, Oct. 17, 1917. (Appl. No. 15,025 of 1917.)

Basic antimony sulphate is used in place of the normal sulphate in the preparation of "antimony vermilion" or "crimson antimony" by precipitation with sodium thiosulphate.—A. de W.

*Lampblack [and hydrochloric acid, from natural gas]; Process of manufacturing* —. W. R. Mott, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,259,121, Mar. 12, 1918. Date of appl., June 24, 1916.

Chlorine and methane or natural gas are admitted into a heater to form carbon tetrachloride and hydrochloric acid. The resulting products are admitted to a mixer with more methane or natural gas and forced into a heater to form lampblack and hydrochloric acid, the hydrochloric acid and excess of natural gas being withdrawn through a condenser to condense the former, and the excess of natural gas then led into the stream of natural gas entering the mixer.—A. de W.

*Ink; Solid* —. P. Akashi, Tokyo, Japan. Eng. Pat. 114,601, Dec. 21, 1917. (Appl. No. 18,941 of 1917.)

A PLASTIC or pasty composition forming a permanent solution when dissolved in water and which will

not dry up or harden on keeping, is prepared with the aid of a mixture of thymol and lactic acid. A suitable composition for ink may be made from dye (e.g., Methylene Blue), 25 parts; dextrin, 5 parts; gum tragacanth mucilage, 4 parts; thymol, 2–3 parts; and lactic acid, 3–4 parts, all by weight. —A. de W.

*Phenolic condensation products; Composition containing* —. Process of moulding articles. L. H. Bakeland, Yonkers, N.Y., Assignor to General Bakelite Co., New York. U.S. Pats. (A) 1,259,472 and (B) 1,259,473, Mar. 19, 1918. Dates of appl., Dec. 2, 1909, and May 25, 1917.

(A) AX infusible composition, particles or fragments of which can be welded by heat and pressure to compact coherent masses, consists of a phenolic condensation product in solid solution with a fusible cyclic hydrocarbon of high boiling point, insoluble in water. (B) Coherent masses or articles are produced from a final infusible phenolic condensation product by compounding it with a material imparting to the mass the capability of welding, subdividing this product, and then consolidating the fragments by heat and pressure.—A. de W.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Caoutchouc; Depolymerisation of raw* —. S. Takeuchi, Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1917, 20, 1297–1309.

The degree of depolymerisation of raw caoutchouc which takes place during mastication has been studied by measuring the absolute viscosity of solutions of 1 grm. of caoutchouc in 70 c.c. of benzene. Samples from *Urecola* and *Hevea*, wild and cultivated, were investigated. This determination serves as a very convenient control of the time of mastication and other operations. The mastication should be carried on until the absolute viscosity of the solution reaches about 0.1. The solubility in acetone of the resin or other acetone-soluble matter changes during mastication, and shows a minimum value when the viscosity reaches about 0.1.—J. F. B.

#### PATENTS.

*Rubber coagulum or similar material and process for preparation of the same for vulcanisation.* E. M. Slocum, Medan, Sumatra, Assignor to General Rubber Co. U.S. Pats. (A) 1,259,793 and (B) 1,259,794, Mar. 19, 1918. Date of appl., Jan. 8, 1917.

(A) LATEX is treated with a naphthol, all the solid constituents of the latex being thereby substantially conserved. (B) Rubber coagulum, before the incorporation of sulphur, is subjected to the action of  $\beta$ -naphthol so as to form a semi-permeable layer over the nitrogenous material in the rubber and thus prevent its migration.—D. F. T.

*Plastic [rubber] composition.* F. J. Tone, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,259,806, Mar. 19, 1918. Date of appl., Sep. 23, 1915.

A PLASTIC composition is made containing rubber mixed with silicon monoxide.—D. F. T.

### XV.—LEATHER; BONE; HORN; GLUE.

*Tanning; Lyotropic series and theory of* —. H. G. Bennett, J. Soc. Leather Trades' Chem., 1917, 1, 130–136 and 169–182.

DISSOLVED substances fall into a definite sequence when arranged in the order of their effects on the physical properties of water and on its properties



as solvent and as a dispersion medium for colloid sols. This sequence is known as the lyotrope series, and its influence in various processes of leather manufacture is pointed out. Lyotrope influence has a marked effect on the imbibition of the hide gel, and a considerable though less effect on the gelation or diffusion of the tannin sol. The active surface of swollen hide or hide powder is of predominant importance for the adsorption of tannin, and largely depends on the lyotrope influence of salts, etc., present. Consequently the addition of non-tanning substances (such as glucose or magnesium sulphate) to a tannin solution causes considerable variations in the amount of tannin shown by analysis. The first stage of the vegetable tanning process is adsorption, and the amount of tannin adsorbed is a function of the specific surface of the adsorbing hide. The next stage consists in the diffusion of the tanning sol into the hide gel (there also lyotrope influence exerts an effect), and the gelation of the tanning sol at the interface of sol and hide fibre. Either of these two phenomena may predominate. Another essential part of tanning is the co-precipitation of the two oppositely charged colloids, the positive hide (in acid) and the negative tannin. Chrome tannage is essentially the same as vegetable tannage. Lyotrope influence is more marked, but exerted in the same sense, i.e., the imbibition of the hide gel is reduced. There is the same sequence of lyotrope influence, adsorption, gelation, and diffusion of the sol, and precipitation of the sol at the hide fibre surface. The author does not regard tanning as an "irreversible" phenomenon.—F. C. T.

*Gelatin; The swelling of* —. H. G. Bennett.  
J. Soc. Leather Trades' Chem., 1918, 2, 40—51.

OWING to the contractile force of surface-tension the surface layer of a liquid is a zone of compression. If the surface is greatly increased by the presence of a large number of small particles the compression of the water becomes correspondingly greater. In the case of a colloid sol, such as a gelatin solution, there is a zone of compression surrounding each particle, and increased concentration causes the approach of these zones and final overlapping. Thus at first there is increase in viscosity, and subsequently setting. A jelly swells by reason of the mutual repulsion of similarly charged particles, the charges being due to the adsorption of ions. The swelling force varies inversely as the square of the distances separating the particles, i.e., inversely as the cube root of the square of the volume. Thus the contractile force, or force tending to resist swelling, and the swelling force both diminish as swelling proceeds and the volume increases, but the swelling force diminishes with the greater rapidity, though much the larger at the outset. Finally the force of repression equals the swelling force and there is no further increase in volume.—F. C. T.

*Gelatin; The swelling of* —. H. R. Procter.  
J. Soc. Leather Trades' Chem., 1918, 2, 73—76.

This paper is in reply to one by Bennett (see preceding abstracts). The use of terms such as adsorption, lyophile, lyotrope, etc., does not explain anything, since Bennett does not show their relation to understood physical or chemical processes. Further, Bennett's explanations are purely qualitative, whereas Procter and Wilson (this J., 1916, 645; 1917, 92) account quantitatively for the phenomena of swelling. Bennett's contention that jellies are two-phased structures is merely a matter of terms, since everything is two-phased in a molecular sense. In a gelatin jelly, however, even the ultramicroscope gives no proof of structure. The

statement that the internal pressure of a liquid is due to surface-tension is vigorously contested. Actually the surface-tension is a consequence of the internal pressure, which in its turn is caused by molecular attraction. The attraction drawing a particle of liquid inwards is a maximum at the surface, but the compression is a minimum as there is no superincumbent liquid. At interfaces of contact with solid or other liquid particles the tensions may be zero or negative. No experimental condensations of liquids by pressure or addition of soluble salts have resulted in large increases of viscosity. Whereas Procter and Wilson attribute the swelling of gelatin to the osmotic pressure or kinetic energy of ions associated with the colloid, Bennett ascribes it to static electrical repulsion of similarly charged colloid particles. Such particles, however, will not repel each other, as their charges will be balanced by a surface layer of ions of opposite charge, the whole complex being electrically neutral. Agreement is expressed with Bennett's view of the practical importance of the lyotrope series in tanning.—F. C. T.

*Chrome liquors; Alteration in basicity during tanning of hides in one-bath* —. C. F. Barber and P. R. Barker. J. Soc. Leather Trades' Chem., 1917, 1, 142—144.

FOUR series of liquors were used, the first made from chrome alum, the second by the reduction of bichromate with glucose, the third from a ready-made one-bath liquor, and the fourth made from bichromate by reduction with thiosulphate. Each series consisted of several liquors of different basicities. During tannage the basicity sometimes remained constant, sometimes diminished, and occasionally increased. No general rule appears to be followed. The results are given in detail in the paper.—F. C. T.

*Leather; Wear resistance of* — from different parts of the hide. F. P. Veitch and J. S. Rogers.  
J. Amer. Leather Chem. Assoc., 1918, 13, 86—90.

A LARGE number of soles were tested in actual wear on boys' sandals. Leather from butts wore twice as long as leather from bellies and one and one-third times as long as that from shoulders.—F. C. T.

*[Tannery] lime liquors; Determination of ammonia in used* —. F. C. Thompson and K. Suzukl.  
J. Soc. Leather Trades' Chem., 1917, 1, 140—142.

CYSTINE is decomposed by magnesia at 100° C., yielding ammonia, and its probable presence in used lime liquors points to a likely source of error in the ordinary method of determining ammonia. Distillation of the neutralised liquor with magnesia *in vacuo* at any temperature below 40° C. (when cystine is unattacked but ammonium salts easily decompose) gives lower results than the ordinary method, the difference being usually about 5% of the ammonia found.—F. C. T.

*Egg yolks; Estimation of fat in commercial* —. M. C. Lamb and A. Harvey. J. Soc. Leather Trades' Chem., 1917, 1, 186—187.

ADAM's method for the estimation of fat in milk is adopted. The egg yolk is spotted on an Adam's paper, or on a strip of coarse fat-free filter paper. The paper is dried overnight at the ordinary temperature and then extracted as usual in a Soxhlet apparatus. The advantage of the method is in its convenience. Accurate results are obtained.

—F. C. T.

## PATENTS.

*Hides and skins; Process of tanning* —. L. T. Hall and F. Tuttle, Houston Heights, Tex. U.S. Pat. 1,256,789, Feb. 19, 1918. Date of appl., Mar. 25, 1916.

Hides are tanned by an infusion of tillandsia. —F. C. T.

*Leather; Process of producing artificial* —. C. E. Arnold, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,257,665, Feb. 26, 1918. Date of appl., Sep. 3, 1914. Renewed July 9, 1917.

ONE or more coats of a plastic nitrocellulose composition are applied to a fabric with the aid of pressure.—F. C. T.

*Leather; Process for producing artificial* —. F. Sparre, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,257,756, Feb. 26, 1918. Date of appl., Sep. 3, 1914. Renewed July 10, 1917.

ONE or more coats of nitrocellulose composition in the form of a jelly are applied to a fabric and each coat subsequently dried. A coat of cementing material is then applied, and before this is dry a coat of nitrocellulose plastic material is applied with the aid of pressure.—F. C. T.

*Glue; Process of manufacturing vegetable* — [from starch]. V. G. Bloede, Catonsville, Md. U.S. Pat. 1,257,307, Feb. 26, 1918. Date of appl., Oct. 29, 1917.

A STARCH base is treated with caustic alkali, then partially or completely neutralised, and subsequently reduced to the required consistency either by the action of ferments or enzymes or by dilution with a thin vegetable glue produced from starch by such ferment action.—F. C. T.

*Hides, skins, and the like; Machine for facilitating the handling and treatment of* — in tanning and like operations. The Vulcan Engineering Co. (Whitehaven), Ltd., and A. Dickson, Whitehaven. Eng. Pat. 115,009, Jan. 29, 1918. (Appl. No. 1665 of 1918.)

## XVI.—SOILS; FERTILISERS.

*Zeolitic silicic acid in soils; Determination of* —. K. K. Gedrovic. Reprint, pp. 400–406.

THE following method for determining zeolitic silicic acid, i.e., that liberated when a soil is treated with concentrated hydrochloric acid, gives good results, is rapid, and obviates the necessity for using large platinum dishes. Five grms. of the soil is treated in a small platinum dish with 10% hydrochloric acid and then calcined over a gas burner furnished with a movable mushroom-shaped top perforated over its whole surface; the temperature of the soil should not exceed 625° C. When the organic matter has been completely destroyed, the soil is cooled, mixed in a beaker with water and with 100 c.c. of 5% potassium hydroxide solution. The liquid is then heated for half an hour on a water-bath and subsequently filtered, the first opaque portion of the filtrate being returned to the filter. The contents of the latter are washed ten times with 1% potassium hydroxide solution and the total filtrate acidified with hydrochloric acid and evaporated to dryness on a sand-bath at 125°–150° C., this requiring 1–1½ hour. The silicic acid, thus rendered quite insoluble, is washed and filtered; it should be free from chlorine.—T. H. P.

*Bracken as a source of potash*. R. A. Berry, G. W. Robinson, and E. J. Russell. J. Rd. Agric., 1918, 25, 1–11.

Young bracken yields the purest ash, containing just over 50% K<sub>2</sub>O, but the total yield of potash per acre is only about 20 lb. Fully-grown bracken yields a less pure ash, containing from 30–40% K<sub>2</sub>O, but the yield of potash per acre may be 200 lb. or more. When the plant dies and shrivels, however, the potash becomes lost, part returning to the root and part being washed away by the rain. At the fully-grown stage the yield of potash per acre will vary somewhat with the class of soil, but is largely controlled by the relative luxuriance of growth and close packing on the ground. The best time to harvest is July or August, and it is estimated that one ton of bracken ash would be obtained from 7–10 acres of land. Making approximate estimates, the cost of collection of one ton of ash would be from 12l. to 15l., and its value, taking potash at 10s. per unit, would be 15l. to 20l., assuming that all the ash was saved and none exposed to rain. Probably the best way wherever possible is to use the bracken for litter or bedding, thus obtaining the potash and also nitrogen and organic matter, and at the same time liberating straw for other purposes.—W. G.

## PATENTS.

*Cyanamide and like silos; Construction of* —. O. Svendsen, London. Eng. Pat. 114,487, Apr. 11, 1917. (Appl. No. 5119 of 1917.)

A SILO, when of rectangular form, is constructed with a series of sloping shelves, one above the other, along two opposite sides, and a double series along the centre, arranged so as to slope towards the shelves on the sides at an angle slightly greater than the angle of repose of the material to be stored. Hoppers are provided at the top, opening over the space between the sloping shelves. The silo is emptied from the bottom. When the silo is of cylindrical construction the central shelves are of circular form, supported on a central pillar, and placed intermediately with the shelves on the wall. The spaces under the shelves permit of expansion of the material, e.g., crude calcium cyanamide, being stored.—J. H. J.

*Calcium cyanamide; Process of making* —. G. B. Cox, Niagara Falls, N.Y., Assignor to American Cyanamid Co., New York. U.S. Pat. 1,258,747. Mar. 12, 1918. Date of appl., Dec. 19, 1917.

FINELY-DIVIDED calcium carbide is heated to a reacting temperature and nitrogen passed over it at a constant rate. When the reaction begins, the heat applied is decreased and is maintained at the lower level until the reaction is self-propagating. The reaction is allowed to go on until practically all the mass is converted into cyanamide.

—J. H. J.

*Phosphate fertilizer containing nitrogen; Manufacture of a* —. F. W. Howorth, London. From Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. Eng. Pat. 114,873, Apr. 10, 1917. (Appl. No. 5062 of 1917.)

AN intimate mixture of raw phosphate and calcium carbonate is treated with sufficient dilute nitric acid to render the bulk of the phosphate soluble and to form a product which solidifies to a hard, dry mass on cooling. For example, by treating a mixture of apatite (920 kilos., containing 72% of triacalcic phosphate) and limestone (80 kilos.) with nitric acid of 62% strength (790 litres), a product (1850 kilos.) is obtained containing over 15% of soluble phosphoric acid and about 6.5% of nitrogen. Conservation of the nitrous gases is effected by con-



ducting the process in a closed vessel and subjecting the liquid product to the action of a vacuum or compressed air.—W. E. F. P.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar; Balance of some constituent principles of the sugar-beet during the manufacture of —.*

E. Saillard. *Comptes rend.*, 1918, 166, 697–699.

AVERAGE figures are given for twenty factories over a period of fifteen years for the distribution of the dry matter, nitrogen, potash, soda, and phosphoric acid of the beetroot, in the diffusion pulps and water, the carbonatation cakes, the molasses, and the condenser water. The nitrogen in the condenser water is about 17% of the total and is lost. That in the molasses is about 30% of the total and is generally lost in the manufacture of alcohol from the molasses. Almost the whole of the potash and soda are found in the molasses, whilst most of the phosphoric acid is found in the carbonatation cakes.

—W. G.

*Sugar boiling in Hawaii.* A. Giacometti. *Intern. Sugar J.*, 1918, 20, 182–185.

IN the two-boiling system, which is now most generally used in Hawaii, a magma of second sugar and syrup is charged into the pan; syrup is boiled upon it until the pan is full; one half is transferred to a second pan; and the two "strikes" finished with more syrup and molasses. After curing the massecuites with very little or no washing, the sugar is bagged without drying. By the regulation of the quantity of "seed" in the first charge, the desired size of the finished grain is determined, and much care must be exercised in preparing this "seed footing" before proceeding to the actual boiling, since any neglect in eliminating false grain at this stage of the process will result in a poor sugar. When boiling second-grade sugars, a charge of first molasses is drawn into the pan to about one-fourth of its capacity, boiled to string-proof, and dropped into a tank. As soon as enough crystals are formed, which generally occurs after 18–24 hours, the charge is taken back into the pan, and finished with the same grade of molasses. Alternatively, this period of crystallisation at rest may be dispensed with, the molasses being grained directly in the pan; but in either case boiling must be very slow to ensure good exhaustion, and cooling should be carried out in crystallisers alone or in crystallisers and tanks for about equal periods. Complaints have been made by refineries in the United States against the small and uneven grain of Hawaiian sugar, so troublesome in refining, but by insisting upon the careful elimination of false grain in the early stage of boiling this defect has been remedied. Attention is now being given to the high ash content of the sugar. Contrary to the general belief, the greater part of the ash is not present in solution in the molasses adhering to the crystals, but is held by the sugar in the solid form. Washing the sugar in centrifugals and afterwards remelting is insufficient, and the suspended solids must be eliminated by decantation or filtration. Sugars of lower ash content would be obtained by sublimating the thick-juice to neutrality and filtering to remove the calcium salts; the formation of incrustation in the pan would also thus be prevented, or at least much diminished.—J. P. O.

*Molasses; Geerligns' theory of the formation of — and experiments upon the precipitation of sucrose from exhausted molasses by acetic acid.* H. C. Prinsens Geerligns. *Intern. Sugar J.*, 1918, 20, 168–170.

ACCORDING to the author's theory (*Archief Java Suiker-ind.*, 1895, 297; this J., 1895, 667), final

molasses may be regarded as a hydrated combination of sucrose, reducing sugars, and salts, analogous to the known double compounds of sodium chloride and sucrose or calcium chloride and levulose, from which the constituents do not separate out on further concentration of the solution. Experiments are now described controverting the view that molasses is simply a solution of sucrose in non-sugars. Alcohol was added to a solution of sucrose and sodium chloride in water, in such proportion that the sucrose present would have remained dissolved and the salt would have been precipitated had either of these constituents been present alone in the solution. It was found, however, that the solution separated into two layers, both consisting of sucrose, sodium chloride, water, and alcohol, with the same relative proportions of sucrose and sodium chloride in both. A similar result was obtained in the case of cane molasses; whereas were molasses simply a solution of sucrose in non-sugars, it would seem certain that the relative proportions of sugars and salts would thereby have been changed. The observation of Friedrich (this J., 1917, 1187) that on mixing beet molasses with an equal weight of glacial acetic acid, about 36% of the sucrose of the molasses (or about 72% of the total sugar present) is precipitated in the form of a tolerably white powder, is confirmed, and this result is regarded as proof of the existence of a sucrose-salts combination, decomposable on the addition of an acid. Cane molasses failed to yield a parallel result, possibly because in this case the reducing sugars are too strongly combined with the salts to be liberated by acetic acid. If a stronger acid, e.g., hydrochloric, be added, the combination appears to break up, for the alcohol was found to precipitate the salt fraction, or at least part of it, from cane molasses, the sucrose and reducing sugars remaining dissolved.—J. P. O.

*Decolorising carbons; Vegetable — [for sugar refining].* M. A. Schneller. *Louisiana Planter*, 1917, 64, No. 10, 154–156.

A MIXTURE of three parts of sawdust and two of lime when charred and heated to 600°–1000° C. or over produced a carbon of the efficiency of "Norit" (see this J., 1918, 160A), but the process is a costly one, owing to the fact that the product contains much calcium carbonate, which must be eliminated by solution in hydrochloric acid and washing. Processes using water-soluble substances as potassium carbonate or calcium chloride in the mixture that is carbonised are cheaper, and these were found to give carbons having 70–80% of the decolorising power of "Norit." In Louisiana rice hulls or leaves are carbonised with the exclusion of air in a closed iron retort at red heat, when a product is obtained containing about 50% of mineral matter, mostly silica. By dissolving out the silica enveloping the carbon, using about 20% of caustic soda, a material is produced having about 1½ times the decolorising power of "Norit." (See also this J., 1917, 1041.) None of the other methods tried, however; approaches the zinc chloride process of Zelnicek (Austrian Patent 3718), the underlying principle of which appears to be the carbonisation of the organic matter with the aid of dehydrating substances at a low temperature before the furnace heat can destroy the porosity of the material. Thus, a mixture of 2 parts of zinc chloride and 1 of sawdust was evaporated with water and heated in a retort to the volatilisation point of the salt, when a carbon was obtained having a decolorising power three times as great as that of "Norit" (the decolorising power of which is 10–30 times that of ordinary animal charcoal); while a 3 to 1 mixture of these two ingredients produced a carbon five times more active than "Norit."—J. P. O.

*Corrosion in an evaporator of a sugar factory in Java caused by organic acids formed from the juice.* T. A. Jentink. *Archief Suiker-ind. Ned.-Indië*, 1917, 25, 29–31. *Intern. Sugar J.*, 1918, 20, 194–195.

In a factory in Java operating according to the sulphitation-defecation process the brass tubes of the multiple-effect evaporator were strongly corroded, especially in the second and the third effects. Suspecting that this was due to sulphurous acid volatilised from the juices, daily titrations of the condensed waters from the several effects with  $N/50$  iodine and  $N/50$  sodium hydroxide solutions were made during a fortnight, parallel observations being also carried out in a factory working normally. In the factory troubled with corrosion, the acidity of the condensed waters was considerably higher in the case of the fourth or last effect and lowest in the second; whereas normally it is constant in each effect. On the other hand, the iodine titration was highest in the water from the second effect and lowest in the fourth. If the corrosion had been caused by sulphurous acid, the iodine titration would have been highest in the case of the fourth effect, in which by far most corrosion had occurred. It is suggested that, probably owing to insufficient clarification, there were present in the juice volatile organic compounds of ketonic or aldehydic nature, methylglyoxal, for example, which became oxidised during evaporation. As concentration proceeded the content of these compounds in the condensed water (as indicated by the iodine titration) fell, while concurrently acids, probably mostly glyoxylic acid, were formed, and accumulated principally in the last effect of the multiple evaporator.—J. P. O.

*Alcohol from waste molasses in the Philippine Islands.* Brill and Thurlow. See XVIII.

*Erythrodertrin in starch hydrolysis. Digestibility of bread.* III. Blake. See XIXa.

*Influence of suspended matter on colorimetric determinations.* Van der Linden. See XXIII.

#### PATENTS.

*Sugar solutions; Material for clarifying — and means for regenerating the material after use.* T. Hayashi, U. Emura, and Kwanto Sanso Kabushiki-Kaisha, Ltd., Tokyo, Japan. Eng. Pat. 108,303, May 18, 1917. (Appl. No. 7175 of 1917.) Under Int. Conv., July 22, 1916.

A solution of commercial phosphoric acid of 13–20% strength is freed from excess of free sulphuric acid by heating with 3–10% of aluminium or 5–13% of aluminium hydroxide. The greater part of the water is then removed by vacuum evaporation, and the concentrated phosphoric acid is mixed with powdered bone-black to a pasty consistence. The product is used for the clarification of sugar solutions after defecation with lime. The carbon may be regenerated by washing with a solution of phosphoric acid, either directly or, if highly charged with impurities, after burning or boiling with caustic soda.—J. F. B.

*Bagasse, etc.; Process of preserving — and making fuel therefrom. Process for economic steam production in cane-sugar factories.* W. Geveke, New York. U.S. Pats. (a) 1,258,888 and (b) 1,258,889, Mar. 19, 1918. Date of appl., July 3, 1917.

(a) Moist bagasse is tightly baled and the bales are allowed to dry and the bagasse then made into briquettes, the adhesive material contained in the bagasse itself acting as binding agent. (b) Bagasse

is prepared for combustion in boiler furnaces, etc., by mixing it with dry vegetable material, e.g., dry cane leaves cut into pieces.—J. H. L.

*Sugars [and cattle food]; Process of producing — [from spent grains].* R. L. Uman, Baltimore county, Md. U.S. Pat. 1,259,568, Mar. 19, 1918. Date of appl., Sept. 28, 1916.

SUGARS and stock food are produced from waste materials such as brewers' or distillers' spent grains, by heating with dilute mineral acid, e.g., at 115°–120° C., to saccharify the carbohydrates present. The liquid, containing the sugars, may be neutralised after separation from the solid matter, and the latter, still containing the unaltered proteins, may be obtained free from acid and other deleterious substances and suitable for cattle food.—J. H. L.

*Process of manufacturing vegetable glue [from starch].* U.S. Pat. 1,257,307. See XV.

#### XVIII.—FERMENTATION INDUSTRIES.

*Starch [in barley]; Determination of —.* R. Kutzcha. *Woch. Brau.*, 1917, 34, 277–281, 290–291, 294–295, 304–306, 313–316, 323–325, 332–334, 339–341, 349–352, 359–360, 368, 375, 381–384, 391–392, 398–399, 406–408.

The author gives an historical summary of the researches on the determination of starch in barley, and recommends Lintner's polarimetric method (this J., 1912, 653). There is no constant relation between the starch-content of barley and the percentage yield of extract from the corresponding malt (both referred to dry substance); the results obtained by different investigators indicate that the amount by which the latter exceeds the former may vary considerably, average values ranging from 14.8 to 17.5 having been suggested (cp. Lintner, this J., 1911, 1080). The sum of the starch-content and the protein-content of barley is not constant, but may range between 70 and 74% for barleys of the same season. For the valuation of brewing barleys the starch-content is as useful as the extract-content (cp. Lintner, *ibid.*).—J. H. L.

*Sorghum or millet [as malt adjunct].* J. Raux. *Brasserie et Malterie*, 1918, 7, 372–375.

BREWERS in France have recently been offered a new malt adjunct, viz., *domra* or millet, the seeds of *Sorghum vulgare*. It is used in distilleries and yields very pure spirit of pleasant flavour. It is sold in the form of whole grains, sometimes under-corticated, and also as flour. Its range of composition is as follows:—Moisture, 12–15%; starch, 62–66%; extract, 64–70%; fatty matter, 3–5%; total proteins, 10–13%. The yield of extract is about 8–10% lower than that from maize and 10–15% lower than that from rice; it is less than might be expected from the starch-content because the fatty matters present, as in the case of raw maize, impede conversion of the starch to some extent. The removal of the fatty matters would enhance the value of the material not only on this account, but also because they render it liable to become rancid. Millet appears to be well suited for brewing purposes provided it is in good condition, but it is subject to rapid deterioration when abnormally moist. Like maize and rice, it requires cooking to gelatinise the starch. Its use has sometimes given rise to difficulty in the filtration of the wort, but this is chiefly because it has been very finely ground. This trouble may be avoided by cooking under pressure, for in this case the grain need only be coarsely crushed.—J. H. L.



*Alcoholic fermentation; Influence of variations of barometric pressure on the course of —.*

A. Rippel. *Centralbl. Bakt.*, 1917, **11**, 47, 225—229.

In the fermentation of nutrient liquids by yeast it was observed that when fermentation was very slow, extending over 1—2 weeks, the rate of evolution of carbon dioxide, as measured by the loss in weight of the flasks containing the cultures, fluctuated from day to day. The fluctuations showed a certain correspondence with the variations of barometric pressure, low pressures favouring the evolution of gas, and *vice versa*.—J. H. L.

*Fermentation; Influence of air pressure on —.*

H. Zikes. *Allg. Z. Bierbrau. u. Malzfabr.*, 1917, **38**, 229. *Woch. Bran.*, 1917, **34**, 410.

BEER wort was fermented completely in flasks, some of which were closed by means of a sulphuric acid trap and others only with a plug of cotton wool. It was found that in the former case the wort attenuated more completely than in the latter case, though the evolution of gas was more rapid in the flasks closed with cotton wool. The more complete attenuation is attributed to exclusion of air, in consequence of which the yeast is compelled to obtain oxygen by increased decomposition of sugar. The author noted also fluctuations in the amount of gas evolved from day to day, as observed by Rippel (see preceding abstract), especially in the flasks closed only with cotton wool.—J. H. L.

*Enzyme formation.* H. Euler. *Biochem. Zeits.*, 1918, **85**, 406—417.

ATTENTION is called to the fact that the formation of an enzyme (invertase) in developing yeast is not necessarily parallel with the rate of growth. A series of experiments was carried out with culture media containing, in addition to sucrose and necessary inorganic salts, the following substances as sources of nitrogen: glycine, glycine *plus* cystine, glycine *plus* tyrosine, asparagine, alanine, yeast water. There was no marked difference in the rate of growth with different amino-acids, but it was less than in the presence of yeast water. In yeast water the rate of formation of invertase was greater than in the presence of pure amino-acids, but here again there was no marked difference in the action of the individual acids.—S. B. S.

*Molasses; Alcohol from waste — in the Philippine Islands.* H. C. Brill and L. W. Thurlow. *Philippine J. Sci.*, 1917, **12**, A, 267—292.

THE quantity of waste molasses produced in the Philippines in 1915 was probably about 30 million kilos. A typical sample contained 1.39% of potash, 0.38% of phosphorus, and 0.21% of nitrogen. The proportions of potash and nitrogen are low compared with those in Hawaiian molasses, because in many cases no fertilisers are applied to sugar lands in the Philippines and no attempt is made to return the fertiliser constituents in the ash of the bagasse and in the molasses to the soil. Before many years, however, fertilisers will have to be employed to maintain the yield of cane, and the authors consider that the most profitable method of disposing of the waste molasses is to produce alcohol and utilise the valuable constituents of the vinasses for fertilising. This is already being done to some extent, but the methods employed are very crude and the yield of alcohol is therefore low. The experiments described, made with a strongly-fermenting top-fermentation yeast isolated from nipa juice (cp. Pratt and others, this J., 1914, 656), indicate the lines on which improvements may be realised. Wherever practicable without excessive expense, the diluted molasses before inoculation should be

sterilised, e.g., by heating at 70° C., but where this is impossible only good water must be used for diluting. The diluted molasses should have a density of about 16.5° Brix (sp. gr. 1.0678), and should be treated with 2 grms. of sulphuric acid and at least 0.4 gm. of ammonium sulphate per litre. Ammonium fluoride has no greater stimulating action on fermentation than the sulphate. Sea water has a deleterious influence and reduces the yield of alcohol. Addition of rice polishings does not appear to improve the fermentation, but tends to increase the production of acids. The yeast employed should be free from infection; it may be cultivated from a pure culture in diluted molasses, 1 part of the fermenting liquid being seeded into 100—150 parts of unfermented solution. By beginning with a 10% solution of molasses and making each successive solution slightly more concentrated until a density of 16.5° Brix is attained, the ability of the yeast to ferment in more highly concentrated solutions is strengthened. The temperature of the fermenting liquid should be maintained between 28° and 30° C. by artificial cooling. By operating in this way the authors obtained yields of about 90% of the theoretical quantity of alcohol.

—J. H. L.

*Spirit; Revision of the [Dutch] tables for the strength of —.* N. Schoorl and A. Regenbogen. *Proc. K. Akad. Wetensch. Amsterdam*, 1918, **20**, 831—837. *Pharm. Weekblad*, 1918, **55**, 390—409.

THE authors have redetermined the densities of mixtures of ethyl alcohol and water at 15° C. Alcohol prepared from "supra-grain spirit" and from "prime grain (3) and molasses (3) mixed," when distilled over lime, shaken with freshly precipitated silver oxide, and finally distilled over metallic calcium gave concordant values for sp. gr. at 15°/4° C. = 0.79360. The densities of mixtures of this alcohol with water differ very appreciably in certain cases from those recorded by von Baumhauer (1860), on which the present Dutch official tables are based, but are in very close agreement with the values obtained by Osborne and MacKelvey (this J., 1913, 303), on which are based the tables issued by the U.S. Bureau of Standards. These tables are recommended by the authors as affording the most accurate compilation of the densities of mixtures of ethyl alcohol and water.—H. M. D.

*Wood alcohol poisoning.* A. O. Gettler and A. V. St. George. *Amer. J. Pharm.*, 1918, **90**, 280—284.

DETAILS are given of six fatal cases of poisoning by whiskey adulterated with methyl alcohol. The presence of methyl alcohol was detected in the distillates from portions of the brains, by oxidising the liquid and applying the tests for formaldehyde. In view of the danger of direct introduction or inhalation of methyl alcohol into the body, it is suggested that its sale for domestic purposes should be prohibited, and denatured ethyl alcohol used in its place.—C. A. M.

*Action of acids on castor seed lipase.* Kita. *Sec XII.*

*Erythrodertrin in starch hydrolysis.* Blake. *See XIXa.*

*Preservation of moist yeast.* Völtz. *See XIXa.*

*Studies in carbonating. II. Carbonating of distilled water.* Patten and Mains. *See XIXa.*

## PATENTS.

*Germs [lactic bacteria]; Process for compounding — with an enveloping protective medium.* I. F. Harbils, Bronxville, and P. W. R. Eschmann, Assignors to The Arlington Chemical Co., Yonkers, N.Y. U.S. Pat. 1,260,899, Mar. 26, 1918. Date of appl., Dec. 10, 1914.

GERMS, such as lactic bacteria, are prepared in a state of dormant vitality by enveloping them in a medium of a fatty composition. The germs are developed as a culture in a matrix fluid, the culture is filtered through a layer of inert material, the residue on the filter is dried and pulverised, the fatty enveloping medium is melted, and the pulverised residue incorporated therein. The enveloping medium should have a melting point below the temperature of the human body; it should be insoluble in water, and capable of protecting the germs against the action of air, moisture, and the gastric juice and body fluids.—J. F. B.

*Rectification of fermented musts, methyl alcohol, and other liquids; Process and apparatus for —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 14,041, June 10, 1914. Under Int. Conv., June 10, 1913.

SEE Fr. Pat. 470,242 of 1913; this J., 1915, 194.

*Process of producing sugars [and cattle food from spent grains].* U.S. Pat. 1,259,568. See XVII.

## XIXA.—FOODS.

*Wheat or its milled or baked products; Determination of the residue indigestible in vitro by pancreatin acting on —.* L. Devilliers. Comptes rend., 1918, 166, 700—702.

FIVE grms. of the product is digested with 0.025 gm. of Defresne pancreatin, 1 gm. of sodium borate, and 0.30 gm. of crystallised calcium chloride in 100 c.c. of water at 55° C., with thorough agitation, for three hours. The temperature is then slowly raised to 70° C. and more rapidly to 120° C. in an autoclave. The liquid is allowed to cool to 55° C., a further 0.025 gm. of pancreatin is added, and the digestion continued until the whole of the starch has disappeared. To the liquid hydrochloric acid is added until its acidity is equivalent to 0.175% of the acid, and the digestion is continued at 35°—40° C. for one hour, when the residue is filtered off, washed, dried, and weighed. Results are given for a number of samples of wheat, bran, flour, and bread made from the latter.—W. G.

*Bread; Digestibility of —. III. Erythrodextrin in starch hydrolysis.* J. C. Blake. J. Amer. Chem. Soc., 1918, 40, 623—636.

The author has previously shown that the digestion of erythrodextrin is a unimolecular reaction, and might serve, therefore, in determinations of amylolytic activity. With the aim of devising a method of preparation of pure erythrodextrin for such purposes, the degradation of starch by heating at 85° C. after moistening with dilute hydrochloric acid and by boiling with dilute hydrochloric acid has been studied. The course of the decomposition was followed by measuring the colorations produced by iodine water, in a Duboscq colorimeter, a full account of the method being given. It appears that erythrodextrin is the final polysaccharide produced by the "roasting" process if the acid used to moisten the starch is sufficiently dilute. (See further J. Chem. Soc., 1918, i., 254.)—J. C. W.

*Milk; Artificial colouring matter in —.* H. D. Cox. Analyst, 1918, 43, 166—167.

MILK coloured with methyl orange loses its colour within 24 to 48 hrs., but annatto and turmeric give a relatively more permanent colour to the milk, fading only taking place after 6 to 14 days, or longer in the case of annatto. The presence of formalin retards the reduction of methyl orange, but accelerates the decolorisation of the vegetable dyes.—W. P. S.

*Desiccated skim milk; Comparison of the proximate and mineral composition of — with that of normal cows' milk.* E. P. Harding and H. Ringstrom. J. Ind. Eng. Chem., 1918, 10, 295—297.

The proximate composition of commercial samples of desiccated skim milk shows but little variation except in the proportion of moisture, which ranges from 2 to 9% or more. The amount of fat rarely exceeds 2%. A typical American sample contained: Ash, 7.89; fat, 1.42; proteins, 32.86; moisture, 5.60; lactose 48.49; and acidity, 1.58%. Calculated on the basis of milk containing 9% of solids-not-fat, the mineral constituents corresponded closely with those of normal milk, except that the calcium oxide, magnesium oxide, and phosphoric anhydride were higher, possibly owing to the addition of phosphate as an emulsifying agent. Two other samples of different origin gave similar results, whilst in the case of a fourth, the colour, emulsifying power, high protein, low lactose, and high content of calcium and phosphorus indicated that the sample was not a genuine desiccated skim milk powder.

—C. A. M.

*Gossypol, the toxic principle of cotton seed.* F. E. Carruth. J. Amer. Chem. Soc., 1918, 40, 647—665.

Gossypol (see this J., 1917, 1285) is present in glands in all but the woody tissues of the cotton plant. The very sparingly soluble aniline salt, of the average composition,  $2C_{30}H_{30}O_2 \cdot 5C_6H_5NH_2$ , may be used for the separation and determination of gossypol. A more yellow substance, "B" gossypol, is formed by heating at 186°—190° C., a white product, "C" gossypol, by fusion with alkalis, and another yellow compound, "D" gossypol, by cooking the cotton seed for oil. All four substances give blood-red solutions in concentrated sulphuric acid and fine blue colorations when their alkaline solutions are exposed to the air. By suitably conducted cold pressing the oil cells of cotton seed may be emptied without the oil dissolving the contents of the resin glands, and no gossypol then enters the cotton-seed oil, but in the usual cold pressing processes the seed is heated to a considerable extent and the glands ruptured, so that commercial "cold pressed" oil contains about 1.5% of gossypol, that is about three-fourths of the whole. In the "hot pressing" process, the gossypol is exposed to oxidising influences and changes largely into the less toxic "D" gossypol, most of which remains in the cake. Consequently, a "hot pressed" oil should lose less on refining with alkali than a "cold pressed" oil from the same seed. (See further J. Chem. Soc., 1918, i., 236.)—J. C. W.

*Yeast; Preservation of moist —.* W. Völtz. Woch. Bran., 1917, 34, 414.

LACTIC acidification affords a means of preserving moist yeast for feeding purposes in cases where drying is impracticable, though the latter method is to be preferred. Fresh yeast treated with 0.5—1% of sugar, e.g., in the form of molasses or pulped beets, may be mixed with chopped straw and inoculated with a pure culture of lactic bacteria. If stored in trenches impermeable by water, the yeast



will remain for a long time in good condition. Lactic acidification may also be applied to the preservation of other feeding-stuffs, *e.g.*, insufficiently dried green fodder, and products such as distillery vinasses. The addition of 0.5–1% of sugar is necessary to enable the lactic bacteria to develop.

—J. H. L.

*Carbonating; Studies in* —. II. *Carbonating of distilled water*. H. E. Patten and G. H. Mains. J. Ind. Eng. Chem., 1918, 10, 279–288.

In experiments to test the rate of evolution of carbon dioxide from aqueous solutions bottled under pressure, a quart bottle was charged with 770 c.c. of distilled water, and, after cooling to 0° C., the liquid was impregnated with carbon dioxide by means of an apparatus in which the gas was introduced from a gas cylinder while the contents of the bottle were agitated by a rotating stirrer (J. Ind. Eng. Chem., 1917, 9, 787). The gas layer above the liquid was withdrawn and analysed and the time required for the initial pressure to be recorded again on the gauge was noted. This process was repeated several times, simultaneous readings of the time and pressure being recorded, until practically all the gas obtainable at 0° C. at atmospheric pressure had been removed. The bottle was then removed from the ice, further portions of gas withdrawn at the ordinary temperature, and it was finally placed in the steam bath, and the remainder of the gas expelled by boiling. The "pressure recovery" curves, constructed from these data, showed a striking regularity except in the case of the first curve corresponding with the first opening of the bottle, when an excessive quantity of gas was withdrawn. This was contrary to expectation, since a sudden rise of curve and rather sharp decrease of pressure were anticipated. In practice the saturation of aerated liquids is incomplete owing to the pressure of foreign gases (air), which reduce the partial pressure of the carbon dioxide. The effective pressure is that of the partial pressure of the carbon dioxide and not the total pressure of the gaseous layer as indicated on the pressure gauge. Complete equilibrium between the gas in the gaseous layer and in the liquid was not attained at the end of 19 hours, after the first opening of the bottle, but was practically complete after 40 hours. The fact that the length of time a bottle of aerated water is allowed to stand before opening increases the stability of the supersaturated solution after opening of the bottle, may be attributed to the gradual solution of gas bubbles retained by the walls of the bottle. The foregoing results also explain why a badly carbonated product containing a large proportion of air, or one which has stood for an insufficient length of time, may show a high initial pressure in the gaseous layer without a correspondingly great quantity of gas dissolved in the liquid. It also explains why the practice of removing the foreign gases by "sniffing" gives a better product. The time of carbonating may be shortened and the degree of impregnation increased by increasing the speed at which the liquid is stirred during the introduction of the gas.—C. A. M.

*Estimation of fat in commercial egg yolks*. Lamb and Harvey. See XV.

#### PATENTS.

*Fruits and vegetables; Processes and apparatus for drying wet or watery materials, especially* —. J. R. van Musschenbroek, The Hague, Holland. Eng. Pat. 114,442, Jan. 2, 1917. (Appl. No. 105 of 1917.)

Hot air is supplied to the drying chamber through a number of passages which are subdivided so that separate channels are formed, each leading to a

separate rectangular area beneath a perforated tray containing the material to be dried. Means are provided for conducting the spent air to a regenerating chamber, and steam pipes are fitted between the layers of trays for maintaining the temperature of the air. Continuously moving tissues or cloths of hygroscopic material are also passed through the chamber to control the humidity of the air, and then through a separate drying chamber.—W. P. S.

*Vegetables; Method of preparing evaporated* —. *Method of preparing evaporated fruit. Method of preparing evaporated apples. Evaporated vegetable product. Evaporated apples. Evaporated fruits*. R. W. King, The Dalles, Assignor to Pacific Evaporator Co., Portland, Oreg. U.S. Pats. (A) 1,259,631, (B) 1,259,632, (C) 1,259,633, (D) 1,259,634, (E) 1,259,635, (F) 1,259,636, (G) 1,259,637, Mar. 19, 1918. Dates of appl. (A) to (D) June 27, 1916, and (E) to (G) June 29, 1917.

(A) VEGETABLES are treated with sodium chloride solution, steamed at a temperature at which the cells are not burst, and then dried in a current of air below 156° F. (69° C.). (B) Vegetables are subjected to a current of air at 125°–156° F. (52°–69° C.). (C) Fruit is treated with a bleaching agent and steam and dried at a temperature which will not disrupt the cells. (D) Apples are sliced, treated with 3% sodium chloride solution and a bleaching agent, and then dried below 156° F. (69° C.). (E) A steamed uncooked vegetable product which has been treated with sodium chloride solution and dried. (F) Apples which have been treated with sodium chloride and a bleaching agent and then evaporated. (G) An uncooked fruit product of the *Prunus* family which has been evaporated after treatment with a "fuming agent" and steam.—W. P. S.

*Butter; Method of making composition* —. *Artificial butter*. S. M. Dick, Assignor to F. E. Mick, Minneapolis, Minn. U.S. Pats. 1,258,996 and 1,258,997, Mar. 12, 1918. Dates of appl. Jan. 8 and Mar. 15, 1917.

CREAM is separated from milk, the separated milk is condensed, mixed with the cream, and the mixture sprayed into heated air so as to produce a mass of a buttery consistence.—W. P. S.

*Meats; Process for curing* —. *Pickling liquor and process of making the same*. G. F. Doran, Omaha, Nebr. U.S. Pats. (A) 1,259,376 and (B) 1,259,377, Mar. 12, 1918. Dates of appl. Dec. 13, 1915, and Nov. 28, 1916.

(A) MEAT is treated with a pickling liquid containing small amounts of soluble nitrites in addition to sodium chloride and other salts. (B) Old or used pickling liquid containing nitrites is purified and sterilised, suitable amounts of curing agents being added to restore it to the desired strength.

—W. P. S.

*Process of producing sugars [and cattle food from spent grains]*. U.S. Pat. 1,259,568. See XVII.

#### XIXE.—WATER PURIFICATION; SANITATION.

*Bactericidal properties conferred on the blood by the intravenous injection of diaminoacridine sulphate*. C. H. Browning and R. Galbransen. Proc. Roy. Soc., 1918, 90, B, 136–144.

DIAMINOACRIDINE sulphate when injected into rabbits renders their sera bactericidal to *Staphylococcus aureus* and *B. coli*. (See also J. Chem. Soc., 1918, i., 282.)—S. B. S.

*Arsenic; Determination of — in insecticides by means of potassium iodate.* G. S. Jamieson. *J. Ind. Eng. Chem.*, 1918, 10, 290—292.

For the determination of arsenious oxide in Paris green or other arsenite, 0.15 to 0.4 gm. of the sample is treated with 30 c.c. of hydrochloric acid (sp. gr. 1.19), 20 c.c. of water, and 6 c.c. of chloroform, and the liquid titrated with standard potassium iodate solution (3.241 grms. per litre; 1 c.c. = 0.003 gm. As<sub>2</sub>O<sub>3</sub>), in a glass-stoppered bottle. The reagent is added rapidly at first, until the iodine liberated has largely disappeared from the solution. The stopper is then inserted and the bottle thoroughly shaken, and the titration slowly completed, with vigorous shaking after each addition, until the iodine colour of the chloroform has disappeared, and does not appear again after standing for five minutes. It is essential that the acidity of the solution at the end of the titration should not be less than 11%, to prevent hydrolysis of the iodine monochloride formed, nor more than 20%, or the reaction will take place too slowly. For the determination of total arsenic in an insecticide the official (American) distillation process is used, the arsenic being distilled as arsenious chloride by means of cuprous chloride and strong hydrochloric acid, and received in Erlenmeyer flasks chilled with ice. An aliquot portion of the distillate is then titrated as described above.—C. A. M.

*Wood alcohol poisoning.* Gettler and St. George. *See XVIII.*

#### PATENTS.

*Softening and purification of water; Apparatus for the —.* J. J. Lassen and V. F. Hjort, London. Eng. Pat. 114,485, Apr. 11, 1917. (Appl. No. 5082 of 1917.)

The process may be conveniently carried out in an old Lancashire boiler shell, upon the top of which are mounted the water and reagent tanks. After receiving the reagent the water passes to the flues of the boiler, the ends of which are blocked in order to make them suitable for settling tanks. From the top of the flues, pipes lead to the main shell of the boiler, which has a filtering material compartment at one end through which the water passes in an upward direction and thence to a reservoir. When it is desired to flush out the flues, their contents are run into a tank below the boiler. The exit pipe from this tank has a floating arm, which enables the water to be decanted from the surface and raised by a pump to the supply tank on the top of the boiler to be re-treated.—J. H. J.

*Zinc compound [for treating boiler feed water].* F. O. Paige, Assignor to Paige and Jones Chemical Co., Inc., New York. U.S. Pat. 1,260,987, Mar. 26, 1918. Date of appl., July 31, 1917.

The material is composed of zinc dust, vegetable acid, alkali salts, and carbohydrates.—W. E. F. P.

*Drying and deodorising night soil, slaughter house refuse, and other analogous materials; Apparatus for —.* J. W. Stamp, Melbourne, Australia. Eng. Pat. 114,700, May 21, 1917. (Appl. No. 7275 of 1917.)

A closed rotating cylinder supported horizontally on hollow trunnions is enclosed in a casing through which gases from a furnace are passed. The material to be dried is fed into a hopper fitted with a splash shield and discharging into a valve-controlled pipe leading into one of the hollow trunnions and thence into the dryer. Vapour is withdrawn

through the other trunnion and thence through a condenser, by means of a vacuum pump beyond the condenser. The dried material is discharged through a door in the cylinder and a corresponding discharge opening on the lower side of the casing. Material to be dried may be charged directly into the cylinder through the discharge door, and the cylinder may be provided with Galloway tubes.

—W. E. F.

*Garbage disposal; Method of —.* E. C. Wallace, West Roxburg, and S. C. Prescott, Brookline, Assignors to The Bio-Chemic Fertilizer Co., Boston, Mass. U.S. Pat. 1,260,103, Mar. 19, 1918. Date of appl., July 5, 1917.

COMMIXTURED garbage is mixed with a dry bulky material in sufficient quantity to produce a mass containing spaces free from liquid and filled with air. The mass is inoculated with bacteria and kept under optimum conditions for bacterial growth. As the intensity of the bacterial action slackens more garbage is added.—J. H. J.

*Insecticides.* W. H. Hyatt, Long Ditton, Surrey, and E. N. Fellowes, London. Eng. Pat. 114,602, Dec. 22, 1917. (Appl. No. 19,008 of 1917.)

THE insecticides consist of (1) a mixture of copper sulphate, 32 parts, neutral sodium sulphate, 36 parts, and ammonium carbonate, 24 parts, prepared by mixing together the powdered ingredients, or by evaporating to dryness a solution of the copper and sodium sulphates and adding the powdered carbonate, and (2) a mixture of copper sulphate, 32 parts, and ammonium carbonate, 36 parts, in powdered form.—J. H. J.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Ipecacuanha; Alkaloids of —. Part III.* F. L. Pyman. *Chem. Soc. Trans.*, 1918, 113, 222—234.

O-METHYLPSYCHOTRINE (this J., 1917, 1192), on reduction, gives a mixture of emetine and isoemetine. It was therefore to be expected that isoemetine, like emetine, would yield on oxidation methylpsychotrine and rubremetine, and this was proved to be the case. Thus emetine and isoemetine are proved to be stereoisomerides, but it has not so far been possible to establish an equilibrium between the two bases to form a racemic modification. Psychotrine gives cephaeline and isocephaeline on reduction, and methylpsychotrine gives emetine and isoemetine; it is now shown that isoemetine is the methyl ether of isocephaeline, and it has been prepared by the methylation of the latter. When methylated under suitable conditions, isoemetine gives a well crystallised N-methyl derivative; this is the O-methyl ether of N-methylisocephaeline previously obtained (this J., 1914, 764). Complete methylation of isoemetine yields a well crystallised N-methylisoemetine methiodide, which is accompanied by an amorphous salt, probably a mixture of two stereoisomeric methiodides. Both products give N-methylisoemetinemethine when converted into the corresponding methohydroxides and evaporated in a vacuum. By the complete methylation of emetine a stereoisomeric  $\beta$ -N-methylemetine methiodide, m. pt. 262° C. (corr.) and  $[\alpha]_D = +68^\circ$ , has now been isolated in addition to the original  $\alpha$ -methiodide, m. pt. 225°—226° C. (corr.) and  $[\alpha]_D = -10^\circ$ , both yielding the same methine; these two isomerides are due to the asymmetry of the tertiary N atom common to two-ring complexes. Considerable differences in the physiological action of emetine and isoemetine have been observed: the hydro-



chloride of isometine is less than half as toxic as that of emetine. When administered in cases of amoebic dysentery emetine is nauseating but effective, whereas isometine is well tolerated but ineffective towards the amoebæ.—J. F. B.

*Atropine sulphate; Melting point of* —. H. D. Richmond. *Analyst*, 1918, **43**, 168.

ACCORDING to the B.P., atropine sulphate melts at 189°–190° C., whilst the U.S.P. gives the m. pt. as 188°–191° C., or 181°–183° C. when the substance is dry and free from hyoscyamine. These statements are erroneous, since the true m. pt. of atropine sulphate is 194° C. In determining the m. pt. the temperature of the bath should at first be 25° C. below the m. pt. and then be raised gradually: if the temperature of the bath is near the m. pt. when the capillary is introduced, traces of water in the sulphate are expelled rapidly, but at the same time cause the salt to sinter together and appear to melt.—W. P. S.

*Homatropine and the Vitali test.* H. D. Richmond. *Analyst*, 1918, **43**, 167–168.

THE Vitali test cannot be used to distinguish homatropine sulphate from atropine, hyoscyamine, or hyosine, since the sulphuric acid of the sulphate so intensifies the action of the nitric acid that a violet coloration is produced. If, however, the test is applied to the homatropine itself, after separation from the sulphate, a violet coloration is not obtained.—W. P. S.

*Digitalis: Effect of cleaning on the inorganic constituents of* —. C. H. Rogers and E. H. Newcomb. *Amer. J. Pharm.*, 1918, **90**, 239–252.

THE normal ash of digitalis amounts to from 8–10%, but commercial samples may yield over 25%. The U.S. Pharmacopœia fixes the limit at 15%. By sifting the dried digitalis through a 14-mesh sieve the adhering foreign matter may be removed, leaving a residue with the normal amount of ash. For example, a composite sample of the original crop yielded 11.36 to 11.59% of ash, and, when screened, was separated into two portions yielding respectively 9.28% and 26.24% of ash. By further treatment the latter portion (about 20%) was separated into fine siftings yielding over 56% of ash, and coarser portions yielding about 9 to 10%, the loss of actual digitalis being less than 3½%. Only 4.35% of the ash of the screened digitalis was insoluble in hydrochloric acid. Manganese is a constant constituent of digitalis leaves, but varies in amount with the origin of the sample. Digitalis grown in Minnesota normally contains iron, calcium, magnesium, manganese, potassium, and sodium. The ash from the leaves of cultivated plants is usually higher than that of wild plants.

—C. A. M.

*3,4-Dihydroxyphenanthrene (morphol) and 3,4-phenanthraquinone; Synthesis of* —. G. Barger. *Chem. Soc. Trans.*, 1918, **113**, 218–221.

3-PHENANTHROL-4-ALDEHYDE (*Chem. Soc. Trans.*, 1916, **109**, 568) was converted into 3,4-dihydroxyphenanthrene (morphol) by Dakin's reaction. 1.11 gm. of the hydroxy-aldehyde was dissolved in 10 c.c. of pyridine, the air was displaced from the flask by a current of hydrogen, and 0.55 c.c. of 30.8% hydrogen peroxide was added; the addition of 0.45 c.c. of 12.5 N-potassium hydroxide solution then caused a considerable rise of temperature. After boiling for a few seconds, the liquid was cooled, acidified with hydrochloric acid, and the morphol extracted with ether. The yield of recrystallised morphol amounted to 80% of the theoretical. The substance was identified by its m. pt., 142°–143° C. by its

diacetate, m. pt. 159° C., and by conversion into its dimethyl ether. 3,4-Phenanthraquinone was prepared by oxidation of the diphenol in ethereal solution by dry silver oxide in presence of silver sulphate; it was obtained in brilliant red, short, pointed prisms with not very definite melting point, about 132°–133° C. on rapid heating; it did not yield Laubenheimer's reaction.—J. F. B.

*Phenylethylbarbituric acid; Preparation of* —. M. Rising and J. Stieglitz. *J. Amer. Chem. Soc.*, 1918, **40**, 723–730.

SOME practical notes are given on the synthesis of phenylethylbarbituric acid, which was introduced as a specific in epilepsy under the name "luminal," and is now officially termed "phenylbarbital" in America, as veronal is designated "barbital." Methyl phenylacetate obtained by saturating a solution of benzyl cyanide in 90% methyl alcohol with hydrogen chloride, and methyl oxalate obtained by saturating a solution of anhydrous oxalic acid in methyl alcohol with hydrogen chloride, are mixed together, treated with sodium wire and ether and a few drops of alcohol and the resulting sodium compound is decomposed by dilute sulphuric acid, giving dimethyl oxalylphenyl-acetate,  $\text{CH}_3\text{O}^-\text{CO}^-\text{CO}^-\text{CH}(\text{C}_6\text{H}_5)\text{CO}^-\text{CH}_3$ . This is heated *in vacuo* when it loses carbon monoxide and gives methyl phenylmalonate,  $\text{C}_6\text{H}_5\text{CH}(\text{CO}_2\text{CH}_3)_2$ , b.pt. 147°–159° C. under 10 mm. This ester is warmed with ethyl iodide and a solution of sodium in methyl alcohol, and the methyl phenylethylmalonate so formed is heated with urea and a solution of sodium in methyl alcohol, in a sealed tube at 105°–108° C. for 6 hours.—J. C. W.

*Acetylsalicylic acid; Examination of American-made* —. P. N. Leech. *J. Ind. Eng. Chem.*, 1918, **10**, 288–290.

PURIFIED acetylsalicylic acid melted at 131.5°–132.5° C. (corr.), whilst 14 samples of American origin melted between 128° and 133° C. It is suggested that this latter range of m. pts. is more correct than those of the British Pharmacopœia (133°–135° C.), the German Pharmacopœia ("about 135° C."), and the French Pharmacopœia (135° C.). With one exception all the commercial samples examined contained less than 0.1% of free salicylic acid, as determined colorimetrically, and it is proposed that this limit should be adopted as a standard.—C. A. M.

*Crotonisation of acetaldehyde; formation of butanol and hexanol from ethyl alcohol.* P. Sabatier and G. Gaudion. *Comptes rend.*, 1918, **166**, 632–636.

THE conversion of ethyl alcohol into butanol and hexanol is effected in three stages, viz., reduction to acetaldehyde by passing the alcohol vapour over reduced copper at 300° C.; dehydration of the aldehyde by passing its vapour over the oxides of thorium, titanium, or uranium, preferably the last-named, at 360° C.; hydrogenation of the products of crotonisation by passing their vapours with hydrogen over reduced nickel at 170°–180° C. butanol and hexanol with small amounts of higher isomerides being thereby obtained. Stages one and two may be carried out simultaneously by passing the ethyl alcohol vapour slowly over uranium oxide at 360° C. The best yield in stage two is obtained by starting with paraldehyde, and the products from this process are roughly fractionated, the fractions boiling at 90°–130° C. and 130°–220° C. being used for the third stage.—W. G.

*Sulphite turpentine.* Schorger. *See* V.

[Determination of] lead in commercial cream of tartar. Jones. *See* VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## PATENT.

*Sensitised paper or like flexible material for use in obtaining X-ray photographs or radiograms; Preparation of —.* G. W. K. and T. P. K. Crosland, Huddersfield. Eng. Pat. 114,333, June 4, 1917. (Appl. No. 7961 of 1917.)

SENSITISED paper or other flexible material is coated on both sides with a preparation of Venetian red, chrome yellow, or other material opaque to light and which can be easily removed by washing.

—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

## PATENT.

*Manufacture of ammonium perchlorate.* Eng. Pat. 110,544. See VII.

## XXIII.—ANALYSIS.

*Colorimetric determinations; Influence of suspended matter on —.* T. Van der Linden. Archief Suikerind. Ned.-Indië, 1917, 24, 1871—1874. Intern. Sugar J., 1918, 20, 195.

In experiments on the effect of clarifying sugar syrup by boiling and settling, it was found that although there had been an appreciable increase in the purity of the product, the reading in a colorimeter indicated that the syrup was much darker after treatment than before. Further examination showed that the boiled syrup had about the same colour intensity as the unboiled product, the anomalous results being due to the fact that the readings were taken with samples that had not been filtered and contained a small amount of suspended matter. In order to obtain trustworthy results in colour determinations, it is necessary to operate only with solutions that are quite clear and bright.

—J. P. O.

*New reaction of formic acid and hydrosulphites.* Comanducci. See VII.

*Detection of carbon dioxide in the analysis of carbonates and oxalates.* Stafford. See VII.

[*Determination of*] *lead in commercial cream of tartar.* Jones. See VII.

*Sodium cyanide.* Sharwood. See VII.

*Oxidation of sulphides with potassium iodate.* Dean. See VII.

*Determination of carbon in steel.* Dufty. See X.

*Methods for the commercial analysis of ferrosilicon.* Anger. See X.

*Rapid determination of tungsten [in ores].* Foote and Ransom, jun. See X.

*Assay of molybdenum ores and concentrates.* Mabee. See X.

*Determination of moisture and total fatty matter in "acid oils" and soap stocks.* Bolton and Revis. See XII.

*Determination of ammonia in used [tannery] lime liquors.* Thompson and Suzuki. See XV.

*Estimation of fat in commercial egg yolks.* Lamb and Harvey. See XV.

*Determination of zeolitic silicic acid in soils.* Gedrovie. See XVI.

*Determination of starch [in barley].* Kutzcha. See XVIII.

*Determination of the residue indigestible in vitro by pancreatin acting on wheat or its milled or baked products.* Devillers. See XIXa.

*Determination of arsenic in insecticides by means of potassium iodate.* Jamieson. See XIXb.

*Homatropine and the Vitali test.* Richmond. See XX.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

## I.—GENERAL; PLANT; MACHINERY.

## APPLICATIONS.

Balcombe. Making extracts of soluble matter. 8571. May 25.

Barbet et Fils et Cie. Tubular evaporating, condensing, and cooling apparatus. 8821. May 25. (Fr., June 21, 1917.)

Barbet et Fils et Cie. Apparatus for continuous rectification of liquid air. 8954. May 30. (Fr., Oct. 9, 1917.)

Batchelor. Furnaces. 8285. May 17.

Böckman. Furnaces, etc. 8271. May 17. (Norway, Nov. 27, 1917.)

Calvert. Apparatus for catalytic reactions. 7997. May 13.

Carrier Engineering Corporation, and Carrier. Drying, conditioning, and regulating moisture content of hygroscopic materials. 8464. May 21.

Crowcroft and Morris. Furnaces. 8137. May 15.

Fennell. Rotary furnaces or kilns. 9022. May 31.

Hersey, and Kirkham, Hulett, and Chandler. Apparatus for effecting intimate contact between liquids and gases. 9005. May 31.

Johnston. Dehydrating or drying machine for extracting moisture from vegetable, animal, or mineral matter. 8266. May 17.

Kuehnrich. Furnaces. 8611. May 24.

Moreton. Mixing, stirring, or agitating apparatus. 8908. May 30.

Seanes. Apparatus for maintaining vacuum in steam-condensers, evaporating-plant, etc. 8253. May 16.

Soc. Gallot et Cie. 8529. See XI.

Turner. Process for fractional or destructive distillation of liquids. 8011. May 13.

Vacuum Oil Co. Fractional condensing heat-exchanger. 8458. May 21.

Wilson. Filter presses. 8482. May 22.

Wise. Distilling. 8624. May 24.

## COMPLETE SPECIFICATIONS ACCEPTED.

1277 (1917). Marschall. Manufacture of thermoclements having copper sulphide as the positive constituent. (103,671.) June 5.

6939 (1917). Bowrey. Apparatus for fractional distillation. (115,504.) May 23.

7068 and 13,925 (1917). West. Continuous or series kilns. (115,696.) May 29.

7259 (1917). Soc. de Stearinerie et Savonnerie de Lyon, and Berthon. Production of catalysts for reduction or hydrogenation, and apparatus therefor. (107,001.) June 5.



7612 (1917). Thompson (Elmore). Centrifugal drying-machines. (115,909.) June 5.  
 8219 (1917). Cleworth. Wet filters, water coolers, etc. (115,521.) May 23.  
 8295 (1917). Lane. Preparation of catalytic material employed for promoting synthetic chemical reactions. (115,924.) June 5.  
 10,150 (1917). Furse and Neill. Crushing and pulverising machines. (115,542.) May 23.  
 10,860 (1917). Heenan and Froude, and Walker. Apparatus for treating and cooling air and gas. (115,546.) May 23.  
 1035 (1918). Meihuizen. Drying apparatus. (114,620.) May 29.

#### H.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

##### APPLICATIONS.

Abrahams and Parker. Apparatus for generation, from incandescent carbonaceous material, of fuel gas for internal combustion engines. 8296. May 17.  
 Anderson. Apparatus for producing oil fuel. 8490. May 21.  
 Davies. Preparation of fuel briquettes and utilising them in carbonisation, combustion, gasification, etc. 8369. May 31.  
 Davis. Treatment of flue gases. 8440. May 31.  
 Freer and Schauer mann. Liquid fuel. 8079. May 14.  
 Grange Iron Co., and Hoyle. Apparatus for washing coal, etc., and separating it from impurities. 8539. May 22.  
 Hackford, Smith, and Pearson & Son. Treatment of coal, coal products, etc. 8470. May 21.  
 Helps. Gas manufacture. 8569. May 23.  
 Lynn and Rambush. Gas producers. 8258. May 17.  
 Marks (Soc. Franco-Belge de Fours à Coke). Horizontal coke-ovens. 8583. May 23.  
 Nelson. Desulphurising hydrocarbon oils. 8899. May 29.  
 O'Donnell. Powdered fuel, and process of treating same. 8310. May 17.  
 Perry. Apparatus for distilling carbonaceous material. 9010. May 31.  
 Reed. Extraction of hydrocarbon products from shales and coals. 8330. May 17. (U.S., Sep. 23, 1917.)  
 Rew. Carbonisation of coal, etc. 8438. May 21.  
 Turner. 8011. *See* I.  
 Wade (China Sugar Refining Co., and others). 9037. *See* XVII.  
 Walster and Walster. Generating coal-gas in retorts. 8479. May 22.  
 Webb. Incendiary compositions. 8245. May 16.  
 Whittaker. 8100. *See* III.

##### COMPLETE SPECIFICATIONS ACCEPTED.

13,749 and 13,800 (1916). Aktiebol. Ingeniörsfirma F. Egnell. *See* XXIII.  
 4219 (1917). Heyl. Obtaining a distillate of shale free from sulphur. (115,452.) May 23.  
 5081 (1917). Heyl. Obtaining a distillate from shale, free from sulphur. (115,867.) June 5.  
 7813 (1917). Simmance. *See* XXIII.  
 8525 (1917). Bates. Incandescence gas mantles, and method of making same. (115,720.) May 29.  
 10,283 (1917). Toogood, and Dempster & Sons. Construction of vertical retorts. (115,738.) May 29.  
 12,834 (1917). Goodall, Clayton & Co., and Settle. Manufacture of coal gas. (115,997.) June 5.  
 12,855 (1917). Shannon. Liquid fuel. (114,411.) May 23.

13,485 (1917). Justice (Pirani). Obtaining motor fuels and light paraffin oils from shale, and benzene, toluene, and solvent naphtha from coal. (115,573.) May 23.  
 1114 (1918). Soc. d'Exploit. des Appareils Rateau. Apparatus for the production of water-gas. (112,942.) June 5.  
 2059 (1918). Pearson and Meredith. Manufactured fuel. (115,790.) May 29.  
 3455 (1918). Snowden. Repair of coke ovens and the like. (116,059.) June 5.

#### III.—TAR AND TAR PRODUCTS.

##### APPLICATIONS.

Whittaker. Apparatus for distilling lower boiling constituents from oil, etc. 8400. May 21.  
 Wilton. Continuous dehydration and distillation of tar, etc. 8813. May 28.

##### COMPLETE SPECIFICATIONS ACCEPTED.

7485 (1917). Brotherton & Co., Porteous, and Whitelock. Apparatus for extracting naphthalene salts and similar substances from coal tar distillates. (115,906.) June 5.  
 13,485 (1917). Justice (Pirani). *See* III.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATION.

Baner. Colouring-materials. 8515. May 22.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Aktiebolaget Cellulosa. Production of soda cellulose, particularly wood cellulose. 8750. May 27. (Sweden, June 1, 1917.)  
 Cross and Bevan. Treatment of fibrous cellulose for the production of hydrated derivatives. 8312. May 17.  
 Deuts. Gasglühlicht A.-G. Method of loading silk. 8517. May 22. (Ger., May 22, 1917.)  
 Deuts. Gasglühlicht A.-G. Production of artificial silk. 8518. May 22. (Ger., May 22, 1917.)  
 Dydzinski. Retting. 8956. May 30.  
 Farbenfabr. vorm. F. Bayer und Co. Adhesive or agglutinating substances for cellulose derivatives. 8977. May 14. (Ger., Apr. 20, 1917.)  
 Hill. Wood fibre or artificial silk. 8616. May 24.  
 Hyndman. Treatment of tow of flax, etc. 8729. May 27.  
 Knight. Manufacture of paper, etc. 8665. May 24.  
 Lovell. Colloid-treated fabrics. 8527. May 22. (U.S., June 27, 1917.)  
 McKee. Process of obtaining alcohol from sulphite liquors. 8158. May 15.  
 Masters. 8917. *See* IX.  
 Morris. Applying powdered material to surface of fabrics. 8840. May 29.  
 Poulson. Rendering textile fabrics, etc., water-proof and fireproof. 9055. June 1.  
 Privett, and Thames Paper Co. Apparatus for drying paper, paper board, etc., during manufacture. 9026. May 31.  
 Radcliffe. Manufacture of acetylcellulose. 8216. May 16.

##### COMPLETE SPECIFICATIONS ACCEPTED.

9045 (1917). Galloway and Stratton. Utilising waste paper for remaking into white paper. (115,949.) June 5.  
 10,999 (1917). Cowan and Brown. Apparatus for treating paper pulp and other fibrous materials. (115,972.) June 5.  
 13,679 (1917). Stewart, Hall, and Readie. Treatment of certain plants for paper-making. (116,005.) June 5.  
 18,952 (1917). Cowan and Brown. Apparatus for treating paper pulp and other fibrous material. (116,040.) June 5.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

- Barrett and Pennington. Manufacture of carbonic acid gas. 8072. May 14.  
 Bequevort. Manufacture of metabisulphites, bisulphites, sulphites, etc. 8198. May 16.  
 Bertoya. Reduction of oxides and other salts. 7989. May 13.  
 Cloke and Cloke. Rendering arsenic contained in arsenic works, refining furnaces, residues, etc., recoverable by volatilisation. 8045. May 14.  
 Cocksedge and Freeth. Removal of sodium sulphate from solution. 8986. May 31.  
 Field, and Metals Extraction Corporation. Purification of zinc solutions. 8335. May 22.  
 Field, Sulman, and Metals Extraction Corporation. Purification of zinc solutions. 8831 and 8832. May 28.  
 Gulman. Production of sulphuric acid. 8322. May 17.  
 Rivett. Production of ammonium nitrate. 8228. May 16.  
 Rollason. Utilisation of atmospheric nitrogen in the manufacture of ammonia and other compounds. 8614. May 24.  
 Sheard. Manufacture of sulphate of ammonia. 7972. May 13.  
 Uthelm. Manufacture of acetic acid. 8025. May 13. (Norway, June 1, 1917.)  
 Wilton. Manufacture of sulphate of ammonia. 8812. May 28.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 117 (1917). Levin. *See* XI.  
 3399 (1917). Ore. Manufacture of ammonia or its salts. (115,449.) May 23.  
 6579 (1917). Jones (Willing). Preparing water-soluble compounds of aluminium with organic acids. (115,463.) May 23.  
 7014 (1917). Levin. Manufacture of pyrophosphoric acid and acid pyrophosphates. (115,693.) May 29.  
 12,370 (1917). Steiger and Steiger. *See* IX.  
 17,543 (1917). Steiger. *See* IX.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

- Dean, Gasch, and Kettel. Platinising pottery, earthenware, etc. 8003. May 13.  
 Koizumi. Lacquer decoration on glass. 9000. May 31.  
 McCauley. Conveyance of molten glass. 9030. May 31.

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

- Culls. Concrete, etc. 8243. May 16.  
 Hartner. Manufacture of mortar-forming material from anhydrite. 8395. May 17. (Ger., July 18, 1917.)  
 Kiddie. Machines for mixing concrete ingredients, etc. 8828. May 28.  
 Masters. Waterproofing mixture for making Portland cement non-absorbent when kept in water. 8916. May 30.  
 Masters. Waterproofing solution for timber, linen, flannelette, paper, etc. 8917. May 30.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 9210 (1917). Charlton. Manufacture of cementing materials. (115,946.) June 5.  
 12,370 (1917). Steiger and Steiger. Shaft furnaces for burning cement, lime, etc. (109,266.) May 23.

- 17,543 (1917). Steiger. Kilns for burning cement, lime, or the like. (111,851.) May 23.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

- Adam and Stevenson. Removal of tin coating from tinned iron. 8463. May 21.  
 Bertoya. 7989. *See* VII.  
 Böckman. Reduction and smelting of ores. 8271 and 8272. May 17. (Norway, May 29 and June 29, 1917.)  
 Burgan and Winder. Machine for testing hardness of metals. 9045. June 1.  
 Carpmuel (A.-G. f. Autogene Aluminium Schweißung). Autogenous welding of aluminium or its alloys. 8874. May 29.  
 Clarke, and Edison Swan Electric Co. Treatment of refractory metals. 9028. May 31.  
 Gibbons Bros., and Masters. Furnaces for heating metal articles, etc. 8040. May 14.  
 Hickling. Puddling ball and mill furnaces for manufacture of wrought iron. 8967. May 31.  
 McDermott. Apparatus for concentrating crushed ores. 8076. May 14.  
 Mackie. Gold concentrators. 8829. May 28.  
 Milliken. Alloys, and process of making same. 8289. May 17.  
 Sarron and Simon. 8761. *See* XI.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 14,059 (1916). Giesecke. Process for sintering fine ores, fine dust, purple ore, iron filings, etc. (115,441.) May 23.  
 5694 (1917). Maconochie and De Ros. Oxidising or volatilising metals, such as tin and zinc. (115,870.) June 5.  
 7078 (1917). Vogelsang. Treatment of ores. (115,697.) May 29.  
 8000 (1917). Eldridge. Process of making copper alloys. (115,917.) June 5.  
 8233 (1917). Pozzo and Colonnetti. Apparatus for testing iron. (107,759.) May 29.  
 10,396 (1917). Connolly. Crucible furnaces. (115,543.) May 23.  
 11,102 (1917). Gillespie and Sheather. Acid-resisting metal and articles made thereof. (115,759.) May 29.  
 11,499 (1917). Jones. Distilling metallic ores and other materials containing metals. (112,924.) May 29.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

- Böckman. Electric induction furnaces. 8273. May 17. (Norway, Oct. 12, 1917.)  
 British Thomson-Houston Co. (General Electric Co.). Electric furnaces. 8509. May 22.  
 Davies. Dry non-acid accumulator. 8047. May 14.  
 Levin. Electrolytic gas-generators. 8207. May 16. (U.S., May 22, 1917.)  
 McLeod. Preventing rapid exhaustion of primary batteries. 8740. May 27.  
 Oldham and Oldham. Galvanic batteries. 8162. May 15.  
 Sarron and Simon. Electro-converter furnaces and process of treatment for manufacture of steels thereby. 8761. May 27.  
 Soc. Gallot et Cie., and Soc. Poussin, Rondeau, et Cie. Apparatus for electrical separation of substances in suspension in gaseous or fluid media. 8529. May 22. (Fr., May 22, 1917.)  
 Wallace. Primary batteries, and their manufacture. 9025. May 31.



## COMPLETE SPECIFICATIONS ACCEPTED.

- 117 (1917). Levin. Electrolytic apparatus for generating oxygen and hydrogen. (107,366.) May 23.  
 3585 (1917). Schuster. Galvanic cells. (115,653.) May 29.  
 4885 (1917). Wade (Flanders). Manufacture of plates for electric storage batteries. (115,865.) June 5.  
 5972 (1917). Greaves and Etchells. Electrically-heated resistance furnaces. (115,866.) June 5.  
 7519 (1917). Hamilton and Holland. Preventing oxidation or wasting of electrodes in electric furnaces. (115,705.) May 29.  
 8478 (1917). Sahlin. Electric furnaces. (115,719.) May 29.  
 11,079 (1917). Chloride Electrical Storage Co., and Dean. Secondary electric batteries or accumulators. (115,551.) May 23.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

- Hilliam. Cleaning-preparations, etc. 8935. May 30.  
 Pink. Separating solid and liquid fatty or fat-like bodies. 8209. May 16.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 7210 (1917). Turner and Leighton. Extraction of coconut oil. (115,877.) June 5.  
 7259 (1917). Soc. de Stearinerie et Savonnerie de Lyon. *See I.*  
 8347 (1917). French. Presses for expressing oil and the like. (107,200.) June 5.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 5694 (1917). Maconochie and De Ros. *See X.*  
 9572 (1917). Burke. Composition for use as an adhesive or varnish. (115,537.) May 23

## XV.—LEATHER: BONE; HORN; GLUE.

## APPLICATION.

- Walker. Treating hides during soaking, tanning, etc. 8391. May 21.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 3143 (1918). Heyl. Process for drying japanned or patent leather. (113,622.) June 5.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

- Bottomley. Treatment of peat. 8434. May 21.  
 Fry and de Wolf. Treatment of seeds. 9039. May 31.

## XVII.—SUGAR; STARCHES; GUMS.

## APPLICATION.

- Wade (China Sugar Refining Co., Shaw, and McTavish). Decolorising-carbon, and preparation thereof. 9037. May 31.

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATIONS.

- Block. Manufacture of non-alcoholic malt beer. 8925. May 30.  
 McKee. 8158. *See V.*  
 Pascal. 9019. *See XX.*

## COMPLETE SPECIFICATION ACCEPTED.

- 7051 (1917). Kanegafuchi Boseki Kabushiki Kwaisha. Production of an extract of proteolytic enzymes. (106,501.) May 29.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

- Fyers and Watkins. Preservative treatment of fish. 8891. May 29.  
 Jones and Stolt. Aeration of sewage, etc. 8041. May 14.  
 Molassine Co., and de Whalley. Manufacture of food for cattle, etc. 8705. May 25.  
 O'Brien. Treatment of wood flour to render it suitable for food, etc. 8061. May 14.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 3215 (1917). Mackay. Cooling and preserving food and other substances. (115,850.) June 5.  
 7064 (1917). Clifford, and Jones and Attwood. Separating and settling solids and semi-solids from sewage liquids, etc. (115,872.) June 5.  
 7070 (1917). Widner. Production of a water softener. (107,197.) June 5.  
 8785 (1917). Ames. Apparatus for aerating sewage and other foul liquids. (115,923.) June 5.  
 15,744 (1917). Widner. Production of a water-softening material. (116,023.) June 5.  
 15,738 (1917). Hampshire. Food compound or mixture. (116,024.) June 5.  
 775 (1918). Scully. Manufacture of potatoes into potato flour. (115,607.) May 23.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

- Pascal. Synthetic manufacture of ethyl alcohol, its ethers, and other alcohols, glycols, and hydrocarbons from acetylene. 9019. May 31. (Fr., Apr. 3, 1917.)  
 Utheim. 8025. *See VII.*  
 Utheim. Manufacture of chloroform. 8026. May 13. (Norway, May 25, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

- 6943 (1917). Imray (Soc. Chem. Ind. in Basle). Manufacture of metal albumin combinations. (115,686.) May 29.  
 7259 (1917). Soc. de Stearinerie et Savonnerie de Lyon, and Berthon. *See I.*  
 7378 (1917). Dreyfus. Manufacture of acetic aldehyde. (115,899.) June 5.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATION.

- Barron and Barron. Method of coating photographic sensitised glass. 8851. May 29.

## XXII.—EXPLOSIVES; MATCHES.

## COMPLETE SPECIFICATION ACCEPTED.

- 3813 (1917). Segay. Explosives. (113,083.) May 23.

## XXIII.—ANALYSIS.

## APPLICATION.

- Burgan and Winder. 9045. *See X.*

## COMPLETE SPECIFICATIONS ACCEPTED.

- 13,749 (1916). Aktiebolaget Ingeniörsfirma F. Egnell. Gas-analysing apparatus. (103,812.) June 5.  
 13,800 (1916). Aktiebolaget Ingeniörsfirma F. Egnell. Apparatus for analysing gas. (104,160.) June 5.  
 7813 (1917). Simmance. Gas calorimeters. (115,915.) June 5.  
 8233 (1917). Pozzo and Colonnetti. *See X.*

**I.—GENERAL ; PLANT ; MACHINERY.**

*Combustion of coal and design of furnaces.* Krelsinger and others. See 11a.

**PATENTS.**

*Vapour condensers and like apparatus; Tubulous* —. W. L. Flinn, Elizabeth, N.J., U.S.A. Eng. Pat. 114,945, July 12, 1917. (Appl. No. 10,086 of 1917.)

Each horizontal pipe section of a condenser or like coil is provided with a separate trough for the cooling liquid, the pipe and trough being spaced apart by a web having an opening for the passage of liquid. The trough has one upturned end to accommodate the corresponding end of the pipe, and this end is provided with lugs by means of which it may be supported in a frame. The trough supports the pipe at that end of the latter which is connected to the section next above, and the trough is supported by the pipe at the opposite end by means of a suitable bridge-piece spanning the trough and held against the pipe by inclined bolts carried by a collar surrounding the pipe. The bridge-piece is provided with an opening to maintain the desired liquid level, and to direct the liquid over the end of the pipe into the upturned end of the trough below. The apparatus is specially adapted for use with pipes having liquid-sealed joints as described in Eng. Pat. 113,907.—W. F. F.

*Jacketed vats for heating and cooling liquids.* A. G. Enock and Co., Ltd., and A. G. and E. C. Enock, London. Eng. Pat. 115,126, June 1, 1917. (Appl. No. 7867 of 1917.)

The vat is constructed with an inner vessel and an outer casing, a coil or series of tubes being placed in the intermediate space adjacent to the inner vessel, and surrounded by insulating material held in position by the outer casing. A cooling or heating fluid is circulated through the coil or tubes. In the interior of the vat a swinging coil is mounted to agitate the contents, and a cooling or heating fluid is circulated through it.—J. H. P.

*Oil and similar liquids; Apparatus for cleansing and filtering* —. J. S. Highfield, London. Eng. Pat. 115,140, July 3, 1917. (Appl. No. 9497 of 1917.)

The liquid to be treated is pumped into a vertical cylindrical vessel, through an inlet tube a short distance above the bottom, and passes upwards through a horizontal layer of powder, e.g., fullers' earth, supported between metal gauze on a perforated plate. Above is a clear space in which some settlement takes place. The upper part of the cylinder is occupied by a filter-press consisting of a series of horizontal wheels with perforated plates covered with filter-cloth between. The liquid enters the press by means of a central hollow shaft, passes through holes into alternate wheels, through the cloth both upwards and downwards into the other set of wheels, and, leaving these, passes upwards through a space between the circumference of the press and the sides of the apparatus, and out through a pipe at the top of the vessel. The lid of the apparatus can be screwed down in case it is necessary for the liquid to leave the apparatus under pressure.—L. A. C.

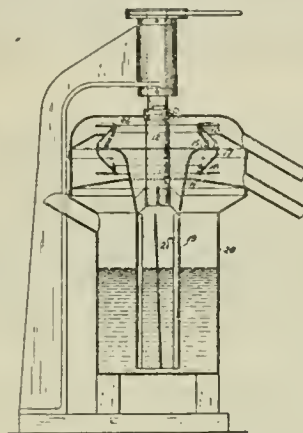
*Heat-insulating compounds.* N. Ito, Tokyo, Japan. Eng. Pat. 115,163, Aug. 8, 1917. (Appl. No. 11,419 of 1917.)

A HEAT-INSULATING compound, suitable for covering boilers, steam-pipes, etc., is made by adding 4 to 7

parts of sodium bicarbonate to 100 parts of a solution of calcium bicarbonate prepared from lime water and mixing this solution with 50 parts of kieselguhr, 20 parts of fibrous asbestos, 40 parts of magnesium carbonate, and 40 parts of anhydrous calcium sulphate.—A. B. S.

*Separator; Centrifugal* —. T. H. Parker, San Francisco, Cal. U.S. Pat. 1,262,384, Apr. 9, 1918. Date of appl., May 26, 1917.

A ROTARY distributor, 14, 19, is connected by radial webs to a shaft, 12, supported by an overhead bearing. The liquid to be separated is raised from the vessel, 20, by the rotation of the inclined ribs,



21, and discharged at the flared edge, 15, of the distributor into the annular shell, 17. This shell is open at the top and bottom, and is provided with lips, 18, 26, projecting inwards so as to confine a mass of heavy liquid within the shell to a level above the rim of the distributor. The lighter liquid is thereby prevented from passing below the rim, 15, and is discharged over the upper edge of the shell, while the heavier liquid is discharged over the lower edge. Separate discharge outlets are provided as shown.—W. F. F.

*Drying apparatus; Powder* —. F. L. Caskey, Buffalo, N.Y. U.S. Pat. 1,262,448, Apr. 9, 1918. Date of appl., Mar. 14, 1917.

A VERTICAL cylindrical drying chamber is provided with axial supply and discharge pipes at the top and bottom respectively, and is surrounded by a heating coil, the whole being enclosed in a casing. The pipes at their free ends carry discs, provided with annular channels on their outer faces which engage with corresponding ribs on other discs so as to close the pipes. The lower closure disc carries a short tube sliding within the discharge pipe, and having a side opening for discharge, and this disc may be moved axially by a hand-operated screwed rod so as to vary or close the discharge opening.—W. F. F.

*Separating and dewatering device.* F. W. Beardslee, Assignor to M. T. Beardslee, San Francisco, Cal. U.S. Pat. 1,262,738, Apr. 16, 1918. Date of appl., Sept. 25, 1916.

THE inflowing material is directed against the buckets of a floating turbine wheel in a container divided by baffles into inner and outer zones. The light material and liquid are withdrawn from the inner zone near the upper end of the container,



whilst the heavier material, which is projected outwards, sinks to a valved discharge outlet at the bottom. The discharge valve is connected with the turbine wheel, so that the consistency of the heavier material discharged is varied according to the height of the material in the tank.—J. H. P.

*Thickening process; Continuous* — A. L. Genter, Assignor to General Engineering Co., Salt Lake City, Utah. U.S. Pat. 1,263,226, Apr. 16, 1918. Date of appl., Sept. 12, 1917.

In a continuous filtering and thickening process, in which filtration is effected under pressure, the filter medium is cleansed automatically and continuously *in situ* by directing alternately, in any desired succession, the filter current against one side of the filter medium simultaneously with the release of pressure on the filtrate side, and a cleansing current against the opposite side of the filter simultaneously with the release of the filtration pressure.—J. H. P.

*Gases; Process for the removal of — from gaseous mixtures.* P. Beck, Cöln-Ehrenfeld. Ger. Pat. 302,092, Oct. 16, 1915.

THE gaseous mixture is passed through a cooled layer of lignite previously heated until no further evolution of gas takes place. It is an advantage to have the lignite in granular form or as briquettes, and not as a fine powder. The process may be applied to the removal of valuable or injurious gases from waste gases.—B. V. S.

*Gaseous mixtures; Process for the separation of —.* Ges. für Linde's Eismaschinen A.-G., Hüllriegelskreuth, and F. Pollitzer, Munich. Ger. Pat. 302,674, Sept. 12, 1916. Addition to Ger. Pat. 301,941.

FOR the separation of a mixture of, *e.g.*, air, alcohol, and ether, the cooled gas is brought into intimate contact with a well-cooled mixture of alcohol and ether similar to that produced by condensation of the gas.—L. A. C.

*Lubricant [from sulphite waste liquor].* Kirchbach'sche Werke, Coswig. Ger. Pat. 302,188, May 30, 1915.

THE composition consists of graphite, talc, or other minerals and sulphite waste liquor.—L. A. C.

*Separating water, oil, or impurities from steam or air; Device for —.* Device for separating oil, moisture, and impurities from exhaust steam or air. W. H. Taylor and C. Shaw, Sheffield. Eng. Pats. 115,103 and 115,104, May 3, 1917. (Appl. Nos. 6273 and 6274 of 1917.)

*Condensers.* C. J. Snow, Redwood City, Cal., U.S.A. Eng. Pat. 115,141, July 3, 1917. (Appl. No. 9564 of 1917.)

*Classifiers.* A. L. Blomfield, Denver, Colo., U.S.A. Eng. Pat. 115,184, Oct. 10, 1917. (Appl. No. 14,684 of 1917.)

*Organic chemical reactions; Apparatus for performing —.* A. M. Aylsworth, and Savings Investment and Trust Co., East Orange, N.J., U.S.A. Eng. Pat. 103,665, Jan. 17, 1917. (Appl. No. 884 of 1917.) Under Int. Conv., Jan. 24, 1916.

SEE U.S. Pat. 1,213,143 of 1917; this J., 1917, 382.

*Filters.* C. D. Burchenal, New York. Eng. Pat. 104,673, Nov. 15, 1916. (Appl. No. 16,372 of 1916.) Under Int. Conv., Mar. 10, 1916.

SEE U.S. Pat. 1,194,949 of 1916; this J., 1916, 1000.

*Drying solid bodies containing liquid; Process and apparatus for —.* J. W. Æ. Elling, Assignor to A./S. Myrens Verksted, Christiania, Norway. U.S. Pat. 1,263,392, Apr. 23, 1918. Date of appl., Mar. 20, 1917.

SEE Eng. Pat. 105,072 of 1917; this J., 1918, 280 A.

*Evaporating apparatus.* J. A. Reavell and W. G. Mann, London. U.S. Pat. 1,263,467, Apr. 23, 1918. Date of appl., Nov. 12, 1917.

SEE Eng. Pat. 110,921 of 1916; this J., 1917, 1262.

## II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Combustion of coal and design of furnaces.* H. Kreisinger, C. E. Augustine, and F. K. Orvitz. U.S. Bureau of Mines. Bull. 135. 144 pages.

PREVIOUS investigations (this J., 1917, 919) have shown that the reactions in the fuel bed of hand-fired furnaces are essentially those of the formation of producer gas. In the present experiments the combustion of these gases and the volume of combustion space required have been studied on a working scale. The furnace consisted of a Murphy stoker with a grate area of 25 sq. ft., built at the end of a horizontal tunnel, 40 ft. long and 3 × 3 ft. in section, which formed the combustion chamber. Arrangements were made for measuring temperatures and for taking samples of gas at intervals of 5 ft. along the combustion chamber. One hundred experiments were made on three types of bituminous coal (Pocahontas, Pittsburgh, and Illinois) at rates of combustion varying from 20 to 60 lb. per sq. ft. of grate area per hour. In general the results showed that the volume of combustion space necessary for complete combustion depends chiefly on the kind of coal, the rate of consumption of fuel, and the amount of excess air. On leaving the fuel bed the gases contain from 10 to 28% of combustible constituents, representing 40 to 60% of the heating power of the coal burned. Heat is developed by combustion of these constituents with the secondary air, at first rapidly and then with diminishing speed as the combustion space is traversed. Increased rate of firing leads to less complete combustion, while increase of combustion space and excess of oxygen favour its completion. The volume of combustion space required appears to be directly proportional to the product of the percentage of volatile matter and a factor of quality—volatile carbon divided by the available hydrogen. The difficulty of burning the volatile matter rises with increase of contained carbon. The gases travel along the combustion space in stratified layers, and, by delaying admixture, this retards combustion, so that the mode of introducing secondary air is important. Hydrocarbons are rapidly destroyed above the fuel bed, and only very small quantities are present in the gases after travelling 1 or 2 ft. On the other hand, soot persists for a long time in the atmosphere of rarefied oxygen, even when excess of air is present. Cooling surfaces such as boiler tubes do not cause the formation of soot, but merely collect it. The relation of various factors is shown graphically in a series of curves, and it is concluded that the requisite dimensions of furnaces for given conditions can be forecast from the data given.—H. J. H.

*Coke and coke breeze; Mechanical stokers for —.*  
B. Lepsien. J. Gasbeleucht., 1918, 61, 3–9.

Coke and even coke breeze can be used in hand-fired boiler furnaces where moderate rates of steaming are required, although the latter demands unusual attention from the fireman. Sprinkler mechanical stokers do not give a uniform distribution of breeze over the fuel bed. Breeze can be successfully burned on a so-called "Pluto" stoker. The breeze is fed from a hopper on to a series of inclined iron grates, resembling ladders with horizontal steps, down which the fuel slides until the clinker falls over the end. A slight reciprocating motion is imparted to the ladders, adjacent units always moving in opposite directions. The difficulty of ignition of coke and breeze is met by using forced draught and a special form of arch. The latter is built over the back part of the grate, thus deflecting the flame towards the front of the grate and thence through an opening to the boiler. This and the absence of draught above the grate cause a rapid heating of the coke on entering the furnace. Chain grates, with forced draught and suitable construction of the arch, have also been used successfully for burning coke breeze. The arch reaches two-thirds the length of the grate and slopes downwards towards the back. The radiation from the arch then raises the breeze rapidly to the ignition temperature. Lump coke can be burned on a chain grate with natural draught if the coke is pre-heated in a small chamber like a miniature gas producer, from which it is fed hot upon the grate. At the rear end a water-cooled bridge is fitted over the grate to retain unburnt coke, preventing it from falling over with the clinker into the ash-pit.

—II. J. II.

*Coal; Combustion of the volatile matter in —.*  
S. H. Katz. U.S. Bureau of Mines. Tech. paper, 183. 15 pages.

THE literature of the chemical and physico-chemical features of carbonisation of coal and decomposition and combustion of liquid and gaseous products has been collated, and a scheme is deduced to represent the supposed process of combustion in coal fires. It is proposed to use it as a guide in the further study of combustion.

—H. J. II.

*Steaming horizontal [gas] retorts.* R. J. Rew. Gas J., 1918, 142, 280–281.

It has been found possible to increase the make of gas per ton in a small gasworks by steaming the coal in horizontal retorts of a special pattern. The settling contained three retorts, one or more of which could be steamed. The make of "straight" coal gas per ton was 11,500 cb. ft., and by steaming one retort for 14 hours this could be increased 20% with a loss of coke amounting to 15½ lb. per 1000 cb. ft. of water-gas produced. Trials with boiling burners showed that the gas was apparently satisfactory for use in ordinary domestic appliances.—II. J. II.

*Paraffin, aromatic, naphthene, and olefine hydrocarbons; Solubility of — in liquid sulphur dioxide.* R. J. Moore, J. C. Morrell, and G. Egloff. Met. and Chem. Eng., 1918, 18, 396–402.

THE utility of liquid sulphur dioxide as a solvent in the refining and analysis of liquid hydrocarbons has been investigated. The solubility of individual hydrocarbons of different types, of binary mixtures, and of a ternary mixture of paraffin, aromatic, and naphthene hydrocarbons has been studied. Solubilities were determined by bringing the liquid sulphur dioxide and the hydrocarbons together in a burette immersed in a freezing mixture of ice, salt, and calcium chloride at temperatures down

to  $-18^{\circ}\text{C}$ . Benzene, toluene, xylene, mesitylene, and olefines were miscible with the solvent in all proportions at this temperature. Light paraffins up to decane, gasoline, and kerosene were practically insoluble. Naphthenes showed a limited solubility at  $-18^{\circ}\text{C}$ ., more marked at  $-45^{\circ}\text{C}$ . There is evidence of the formation of a molecular compound with the solvent. The separation of different types of hydrocarbon by liquid sulphur dioxide is limited by the partition of the hydrocarbon between the two phases, but by attention to the relative proportions some approximation to quantitative separation can be effected. In general, when the constituent to be extracted is in small proportion, aromatic hydrocarbons and olefines, together or separate, may be separated from paraffins and naphthenes. Naphthenes may be separated from paraffins. Over methods of sulphonation and nitration this process has the advantage of permitting recovery of both hydrocarbons and solvent, and also avoiding the production of undesirable by-products.—II. J. II.

*Benzines and benzols; Examination of —.* J. Formaeck, J. Knop, and J. Korber. Chem.-Zeit., 1917, 41, 713–714, 730–731. Z. angew. Chem., 1918, 31, Ref., 62–63.

*Detection of aromatic hydrocarbons (benzene) and of unsaturated hydrocarbons in benzine.* The dracorubin test for the detection of aromatic hydrocarbons in benzine must be cautiously employed, for dracorubin is soluble, with a deep-red colour, in methyl, ethyl, and amyl alcohols, carbon bisulphide, ether, acetone, and chloroform, all of which, with the exception of the last, may be present in benzine and its substitutes. Two vat dyes, Indanthrene Dark Blue BT and Indanthrene Violet RT, are superior to dracorubin for the purpose of this test. Both of these are practically insoluble in benzine, but dissolve in benzene, toluene, xylene, carbon bisulphide, and chloroform with a bluish-red colour and yellow fluorescence; in ether and acetone the solubility is considerably less; in the alcohols, petroleum, and turpentine both are almost or quite insoluble. The colorimetric estimation of aromatic hydrocarbons by means of Indanthrene Dark Blue BT is practicable after two hours' standing, and compares favourably with results obtained by the Kraemer-Böttcher method. *Solidification points of benzene hydrocarbons and benzine.* The following solidification points are given: Benzene,  $+5.5^{\circ}$ ; toluene,  $-88^{\circ}$ ; *p*-xylene,  $+13^{\circ}$ ; *m*-xylene,  $-54^{\circ}$ ; *o*-xylene,  $-28^{\circ}$ ; technical xylol,  $-115^{\circ}$ ; benzines: fraction 200° to 220° C.,  $-93^{\circ}$ ; fraction 24° to 40° C.,  $-203^{\circ}$ ; light benzine (sp. gr. 0.680 to 0.700),  $-135^{\circ}$  to  $-170^{\circ}$ ; medium benzine (sp. gr. 0.700 to 0.740),  $-125^{\circ}$  to  $-150^{\circ}$ ; heavy benzine (sp. gr. 0.740 to 0.760),  $-95^{\circ}$  to  $-120^{\circ}$ ; crude petroleum,  $-58^{\circ}$ ; refined petroleum,  $-85^{\circ}$  to  $-90^{\circ}\text{C}$ .—L. A. C.

*Mineral oils; Sulphur and oxygen compounds of —.* J. Marcusson. Mitt. k. Materialprüf., 1917, 374–380. Z. angew. Chem., 1918, 31, Ref., 62.

THE sulphur content of crude petroleum fluctuates considerably, heavy oils usually containing more than light oils. For the same oil the sulphur content increases in the separate fractions with increasing b. pt. and sp. gr., the residues being the richest in sulphur. Light oils rich in benzine contain least sulphur. The oxygen content also increases with rising b. pt. Compounds containing both sulphur and oxygen are found principally in dark oils, among the solid resinous and asphaltic constituents. The so-called resins form the first transition stage from the petroleum hydrocarbons to the asphaltenes, the latter being formed from the resins either by intramolecular change or by



further action of sulphur or oxygen. Both asphalt-  
enes and resins are polycyclic bodies which contain  
sulphur or oxygen linkings, possibly as ring com-  
pounds or as sulphides or ethers. Asphaltenes con-  
tain a higher percentage of sulphur and oxygen  
than resins.—L. A. C.

*Intermittent tar-dehydrating plant.* Wimhurst.  
See 111.

#### PATENTS.

*Artificial fuel and method of making same.* J.  
Schaub, Newark, N.J., Assignor to American  
Linsed Co. U.S. Pats. (A) 1,262,267 and  
(B) 1,262,268, Apr. 9, 1918. Dates of appl.,  
July 17 and Oct. 4, 1917.

(A) CELLULOSE pentanitrate is dissolved in methyl  
alcohol, and ethyl alcohol is then added till the  
solution is sufficiently viscous to be moulded  
through a die into a "tubulous envelope," the  
tubules of which are of capillary size. Ethyl  
alcohol is then injected into the tubules. (B) Cellu-  
lose pentanitrate is dissolved in methyl alcohol,  
and the colloidal material is solidified by the addi-  
tion of water.—W. F. F.

*Gas-making apparatus.* E. B. Benham, New  
London, Conn., Assignor to Hydrocarbon Con-  
verter Co., New York. U.S. Pat. 1,262,576,  
Apr. 9, 1918. Date of appl., July 14, 1911.

A SPHERICAL chamber is arranged within an outer  
spherical chamber, and is provided with a neck  
projecting through an opening in the side of the  
larger chamber, the latter being closed by a plate  
having openings which are normally closed. The  
space between the chambers is filled with porous  
material, and a mixture of air and oil is supplied  
to it. The outer casing is surrounded by a con-  
centric heating jacket, and the air and gasified oil  
pass through perforations into the central space of  
the inner chamber, from which they are withdrawn  
by a pipe passing through the projecting neck of  
the inner chamber.—W. F. F.

*Gas producers; Operation of — with previous  
drying of moist fuels.* A. Eckardt, Zwickau.  
Ger. Pat. 302,827, Mar. 14, 1914.

THE apparatus is so arranged that the moisture  
given off from a previous drying of the moist fuel  
is utilised to supply the steam under the grate of  
a gas producer of the Mond type. The degree of  
drying is regulated according to the quantity of  
steam it is desired to use in the producer.

—A. de W.

*Gas-purifying apparatus.* H. N. Cheney, Boston,  
and D. S. Reynolds, Brookline, Mass. U.S. Pat.  
1,261,767, Apr. 9, 1918. Date of appl., Feb. 23,  
1917.

A CASINO with vertical side and end walls and  
having its top and bottom inclined but parallel to  
one another, is provided with a number of tiers  
of inclined beams parallel to the top and bottom.  
The purifying material is carried between grids  
supported by adjacent beams so as to permit the  
passage of gas but prevent leakage of purifying  
material. The purifying material is charged  
through openings in the upper ends of the inclined  
chambers formed by the grids and the side walls of  
the casing, and discharged by gravity through open-  
ings at the lower ends. Gas is supplied to the top  
and bottom chambers formed by the top and bottom  
walls and the adjacent grids and to alternate  
spaces between the grids, and discharged from the  
remaining alternate spaces.—W. F. F.

*Tar extractor.* G. M. S. Tait, Washington, D.C.,  
Assignor to H. M. Marble, Newark, N.J. U.S.  
Pat. 1,262,571, Apr. 9, 1918. Date of appl.,  
Mar. 24, 1911. Renewed Aug. 11, 1917.

THE gas passes through a chamber containing a  
steam jet to the extractor, which comprises a series  
of adjacent annular chambers of successively in-  
creasing diameter, communicating with one another  
and arranged with their common axis horizontal.  
Each chamber contains a rotating extractor wheel,  
also of successively increasing diameter, mounted  
on a common shaft, the wheels having blades which  
force the gas from one chamber to the next. Each  
chamber is provided with a discharge conduit for  
tar leading from its lower portion to a water  
chamber below, and terminating below the water  
level. The chamber containing the steam jet com-  
municates directly with the space above the water,  
so that gas escaping through the tar outlets may  
return to the inlet of the extractor.—W. F. F.

*Hydrocarbon; Art of converting —.* W. A. Hall,  
New York, Assignor to Motor Petrol Co., Brattle-  
boro, Vt. U.S. Pat. 1,261,930, Apr. 9, 1918. Date  
of appl., May 24, 1913.

THE vapour of a heavy hydrocarbon is subjected  
to the action of steam at a temperature approach-  
ing red-heat and a pressure above that of the  
atmosphere, in the presence of a finely divided metal  
catalyst which is neither oxidised nor carbonised  
under the above conditions. Carbon separates in  
the solid form, and on condensing the vapours a  
hydrocarbon of lower specific gravity is obtained.

—L. A. C.

*Hydrocarbon liquid; Semi-solidified — and  
method of producing and utilising the same.*  
M. H. Keyt, Chicago, Ill. U.S. Pat. 1,262,808,  
Apr. 16, 1918. Date of appl., Feb. 4, 1916.

A DILUTE aqueous solution of albumin is added to  
the hydrocarbon oil to be solidified.—L. A. C.

*Petroleum; Process of treating —.* M. J. Trumble,  
Los Angeles, Cal., Assignor to Simplex Refining  
Co. U.S. Pat. 1,262,875, Apr. 16, 1918. Date of  
appl., Oct. 10, 1910.

A MIXTURE of hydrocarbons is heated under pressure  
to a temperature sufficient to vaporise the lighter  
constituents at atmospheric pressure. The pressure  
is reduced, and the portion not vaporised is heated  
in the form of a thin film in order to supply the  
latent heat absorbed by the vapours.—L. A. C.

*Gasoline fractions containing aromatic hydrocar-  
bons; Purifying —.* F. C. Ruff, Los Angeles,  
Assignor to By-Products Manufacturing Co., San  
Francisco, Cal. U.S. Pat. 1,263,289, Apr. 16, 1918.  
Date of appl., Jan. 15, 1917.

TO remove unsaturated aliphatic hydrocarbons from  
a gasoline fraction containing an aromatic hydro-  
carbon, it is treated with sulphuric acid containing  
not more than 1% of nitric acid, i.e., an amount  
insufficient materially to attack the aromatic com-  
pound.—L. A. C.

*Naphthenic acids; Preparation of technically pure  
—.* H. Bauer, Kolin, Bohemia. Ger. Pat.  
302,210, Dec. 12, 1916.

CRUDE naphthenic acid, a by-product of the petro-  
leum industry, is well stirred with aniline or  
o-toluidine, and, after standing for some time, the  
lower layer is separated and the solvent distilled  
off. Naphthenic acids of 98–99% purity remain in  
the still.—L. A. C.

*Mineral oils, tar, etc.; Process for the distillation of —.* R. Neumann, Brilau. Ger. Pat. 302,911, Mar. 27, 1917. Under Int. Conv., Mar. 20, 1917.

A TUBULAR still is connected with an auxiliary vessel by means of an overflow pipe at the top and a tube containing a circulating pump at the bottom. These are both filled with the oil or tar, and the fluid kept in circulation by means of the pump, which conveys the liquid from the auxiliary vessel to the still. When the former is empty the pump is shut off and distillation continued until the level of the liquid in the still is within a few cm. of the top of the fire tubes.—L. A. C.

*Lubricating oil and glycerin; [Dicrosyl carbonate as] substitute for —.* Chem. Fabr. von Heyden A.-G. Ger. Pat. 302,361, Feb. 27, 1917.

DICRESYL carbonate is recommended as being cheaper than the tricresyl phosphate described in Ger. Pat. 288,448 (this J., 1916, 415) as a substitute for lubricating oil and glycerin.—L. A. C.

*Lubricating oil; Substitute for —.* E. Stern, Hannover. Ger. Pat. 302,986, Mar. 31, 1916.

A SUBSTITUTE for mineral lubricating oil consists of chlorination products of naphthalene, especially those prepared in the presence of a catalyst (e.g., aluminium chloride or the like), or under pressure, or under the combined action of pressure and a catalyst at a high temperature. The product is only slightly inflammable, neutral, and inactive.—L. A. C.

*Apparatus for cleansing and filtering oil and similar liquids.* Eng. Pat. 115,140. See I.

*Preparation of concentrated ammonia from gas liquor.* Ger. Pat. 302,195. See VII.

*Manufacture of chlorinated hydrocarbons from natural gas.* U.S. Pat. 1,262,769. See XX.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Retort for dry distillation; Experimental —.* F. E. Coombs. Met. and Chem. Eng., 1918, 48, 425.

THE retort consists of a cylindrical container of metal closed at the bottom. A similar cylinder, of slightly larger diameter so as to slide loosely over the retort, forms the lid, to which a bent delivery tube is fitted. The lid is secured to the retort by screws. The apparatus is heated in a bath of liquid—e.g., fused metal or caustic soda—the level of which is below the top of the container, which is thus sealed by the liquid entering the annular space inside the lid. The contents of the retort can be distilled at a known and regulated temperature, while the apparatus permits great accessibility for cleaning and charging.—H. J. H.

*"Perocide" liquor as a substitute for copper-lime liquor.* Appel. See XIXb.

### PATENTS.

*Dry distillation of coal; Process for the —.* T. Limberg, Halle. Ger. Pat. 302,322, Nov. 3, 1916.

DISTILLATION is carried out in three sharply defined phases, i.e., up to 250° C., from 250° to 400° C., and above 400° C. The tar obtained in the second

phase from bituminous lignite has a high paraffin content; that obtained in the third phase has a high oil content.—L. A. C.

*Hydrocarbons; Production of — from bituminous coals.* Chem. Fabr. Buckau, Magdeburg. Ger. Pat. 302,397, Jan. 16, 1914.

THE finely-divided coal is worked up into a homogeneous mass with concentrated alkali solution, either alone or with the addition of alkaline-earth hydroxides. The mass is heated in autoclaves at a high temperature and pressure (30–50 atmos.) and then distilled, at first by means of superheated steam at 400°–450° C., and finally by direct heat until all the liquid has distilled over. The distillate is purified, and the alkali and alkaline-earth carbonates in the residue are converted into hydroxides and used again in the process. A total yield of 25–30% of liquid and gaseous hydrocarbons is obtained, including 4% distilling at 100° C., and 12% up to 200° C.—L. A. C.

*Absorbent material [charcoal] and process of producing the same.* J. B. Garner, Pittsburgh, Pa., Assignor to Metals Research Co., New York. U.S. Pat. 1,262,770, Apr. 16, 1918. Date of appl., June 30, 1915.

WOOD charcoal is heated in an inert gas for a prolonged period at about 250° to 275° C., and subsequently under reduced pressure at 100° to 125° C. for sufficient time to remove occluded gases and volatile constituents.—L. A. C.

*Heating stills, boilers, and the like; Electrical apparatus for —.* C. O. Lorenz, Port Arthur, Tex., Assignor to The Texas Co. U.S. Pat. 1,262,072, Apr. 9, 1918. Date of appl., May 8, 1917.

THE greater portion of the heating element is composed of sand and iron, fused and cast.—B. N.

*Crystalline non-metallic bodies; Production of —.* [Manufacture of filaments for incandescence electric lamps composed of a solid solution of thorium in tungsten.] J. Pintsch A.-G., Berlin. Ger. Pat. 304,857, May 28, 1916. Addition to Ger. Pat. 291,994 (see Fr. Pat. 469,212; this J., 1915, 36).

THE process described in the chief patent is applied to non-metallic substances. The substance used may be one which during the process will be converted by heat or by interaction with the surrounding atmosphere into another substance; or a crystal of the substance may be heated in a gas from which the substance of which the crystal is composed is deposited, so that by regulating the conditions growth of the crystal in a desired direction can be obtained. As a special case the application of the process to the production of wires of a solid solution of thorium in tungsten is described. This solid solution forms long crystals, and is particularly suitable for the manufacture of filaments for electric incandescence lamps.

*Dry distillation of untreated or concentrated cellulose waste lyes.* Ger. Pat. 303,653. See V.

*Production of an insecticide.* Ger. Pat. 302,238. See XIXb.

*Photometry and apparatus therefor.* Eng. Pat. 112,614. See XXIII.



### III.—TAR AND TAR PRODUCTS.

*Tar-dehydrating plant; Intermittent* —. H. R. Wimhurst. Eastern Counties Gas Managers' Assoc., May 2, 1918. Gas J., 1918, 142, 282—283.

THE tar is distilled in two horizontal cylindrical stills of 1500 gallons capacity, direct fired with coke and worked alternately. The stills are built side by side in a setting over a common flue system, so arranged that the products of combustion from the one in use pass under the other on their way to the stack, thereby preheating a charge of crude tar. Distillation of water and light oil is continued until the tar reaches a temperature of 110° C. Each still has an independent condenser communicating with a common separating tank, which in turn is connected to a small purifier box to deal with the waste gas.—H. J. H.

*Solubility of paraffin, aromatic, olefine, and naphthene hydrocarbons in liquid sulphur dioxide.* Moore and others. See IIa.

*Examination of benzines and benzols.* Formanek and others. See IIa.

#### PATENTS.

*Tar oils; Process for the production of — resistant to cold.* L. Singer, Düsseldorf. Ger. Pat. 303,783, Oct. 6, 1916.

TAR oils which contain readily deposited solid matter, particularly the so-called "fatty tar oils," are mixed with hydrocarbons obtained by the extraction of mineral oils with liquid sulphur dioxide or acetone, as described in Ger. Pats. 216,459 (this J., 1910, 144) and 166,452 (this J., 1906, 634). A permanently clear oil is obtained of high viscosity and temperature of ignition.—L. A. C.

*Toluol; Manufacture of* —. F. Thuman, Westminster. From O. B. Evans, Philadelphia, U.S.A. Eng. Pat. 115,066, Apr. 20, 1917. (Appl. No. 5567 of 1917.)

SEE U.S. Pat. 1,230,087 of 1917; this J., 1917, 866.

*Manufacture of  $\beta$ -aminoanthraquinone.* O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 115,259, Apr. 28, 1917. (Appl. No. 6035 of 1917.)

SEE U.S. Pat. 1,255,719 of 1918; this J., 1918, 202 A.

*Substitute for lubricating oil.* Ger. Pat. 302,986. See IIa.

*Benzol varnishes.* Ger. Pat. 302,741. See XIII.

*[Naphthol-resin as] shellac substitute.* Ger. Pat. 302,742. See XIII.

### IV.—COLOURING MATTERS AND DYES.

*Genus Coprosma (Family Rubiaceæ); Tinctorial properties of* —. B. C. Aston. New Zealand J. Sci. Tech., 1918, 1, 3—4.

THREE species of the genus *Coprosma*—*C. grandifolia*, *C. Unarifolia*, and *C. areolata*—are widely distributed in New Zealand and their bark, especially that of the roots, possesses marked tinctorial properties. These plants are related to madder, the natural source of alizarin. The alcoholic extract from *C. grandifolia* is coloured

a brilliant purple in alkaline and yellow in acid solution. By extraction of the acid solution with ether orange-coloured crystals were isolated in quantity equal to 0.068% of the wet bark. The possibility of using the extracts in the local textile industry is suggested.—H. J. H.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### PATENTS.

*Wool substitute; Process for the preparation of — from Eriophorum fibres.* E. L. Fegraeus. Lerum, Sweden. Ger. Pat. 302,261, Feb. 25, 1917. Under Int. Conv., May 19, 1916.

THE fibres are treated for 4—6 hrs. in a cold weak alkaline bath, and then for  $\frac{1}{2}$ —1 hr. in a stronger bath, then rinsed and impregnated with olein or other oil emulsion in a press. The material is hydro-extracted and dried.—L. A. C.

*Fibres and fabrics; Preparation of — as substitutes for wool.* P. Kraiss, Tübingen. Ger. Pat. 302,611, Feb. 1, 1917.

WOOL, hair, horn, or leather refuse is treated with solutions of gelatin, glue, acetylcellulose or other viscous solutions of cellulose or its compounds, with the addition of glycerin or its substitutes, esters such as triphenyl phosphate, oils, or fats in order to render the material pliable. The material is formed into fibres, and these are spun into yarn and cloth in the same manner as paper yarn. In order to render the gelatin or glue insoluble, the solution is treated with a chromium compound, e.g., a bichromate or chrome alum. By after-treatment with formaldehyde, tannin, or their substitutes, or with aluminium salts, the material is hardened and rendered more resistant to the action of hot water.—L. A. C.

*Textile fibres from plants of the lupin family; Process of obtaining* —. Pflanzenfaser Patent Ges.m.b.H., Berlin. Ger. Pat. 302,803, Mar. 26, 1916.

PLANTS of the lupin family are treated with hot strong alkali for such time as to separate the bast tissue from the other constituents whilst insufficient to break up the former into its individual cells. The lye is then rapidly removed and the fibres washed with sprays of water.

—A. de W.

*Cellulose waste lyes; Dry distillation of untreated or concentrated* —. W. Schacht, Weissenfels. Ger. Pat. 303,053, June 24, 1915.

THE apparatus consists of a battery of vertical retorts connected with one another and fitted internally with interchangeable rings or plates to hold the material, which is spread on in relatively thin layers. These rings or plates are connected to one another by pipes and are quickly and easily removable, thus permitting the bottom layer to be removed for lixiviation or further treatment. The distillation in thin layers, it is claimed, results in a larger yield of acetone, oils, and nitrogenous compounds.—A. de W.

*Vegetable fibres; Process of removing pectic matter from* —. B. Leech, Macclesfield. U.S. Pat. 1,263,685, Apr. 23, 1918. Date of appl., Aug. 21, 1917.

SEE Eng. Pat. 104,202 of 1916; this J., 1917, 451.

*Lubricant [from sulphite waste liquor].* Ger. Pat. 302,188. See I.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Tinctorial properties of the Genus Coprosma (Family Rubiaceae). Aston. See IV.*

### PATENT.

*Dyeing apparatus and the like. L. G. Grabe, St. George, N.Y. U.S. Pat. 1,261,921, Apr. 9, 1918. Date of appl., Apr. 7, 1917.*

In the upper part of a dye-vat is a mixing chamber open at the bottom and with a screen extending across the opening, and below this is another screen extending across the vat. A helical vane is attached to the wall in the lower part of the vat. A hollow conoidal vessel with a corrugated wall pierced by numerous openings at different levels in the spaces between the corrugations, and connected at its upper end with the mixing chamber, acts as a circulating device, causing the liquid in the vat to enter the mixing chamber. Water condensed on the cover of the vat falls into a condensing chamber adjacent to the mixing chamber, and thence is delivered on to the middle portion of the lower screen.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Water sprayer for lead sulphuric acid chambers. W. Szlgeti. Chem.-Zelt., 1918, 42, 115.*

TANTALUM, owing to its chemical indifference to chamber gases, combined with mechanical strength, can replace platinum with advantage in the construction of water sprayers for lead chambers. A sprayer is described in which water at 60–75 lb. pressure passes through a plate by three holes and then along three grooves, whence it passes tangentially into a small chamber, one side of which is a tantalum plate pierced at the centre; the water emerges through the perforation in a fine spray. The body of the sprayer is of hard lead. The tantalum plates have shown great durability in practice.—H. J. H.

*Salt; Dissociation of —. H. V. Thompson. Ceramic Soc., Refractories Section, May 18, 1918. [Advance proof.]*

The degree of dissociation of salt in steam at 1100° C. was found to be too small and the reversal of the reaction on cooling too rapid to permit of determining the equilibrium by measurement of the hydrogen chloride formed. An estimate was obtained by volatilising salt at 1100° C. in a current of air for 6 hours—first in dry air and then in air saturated with water vapour at 21.7° C. The condensed products from the air passed were collected and the difference between the two was ascribed to salt formed by recombination of soda and hydrochloric acid formed by the action of steam on the salt. The salt was heated in a platinum tube through which the air passed at the rate of 2 litres per hour. The salt recovered in the condensed product was 0.0554 gm. per litre of dry air and 0.0782 gm. per litre of air saturated at 21.7° C., i.e., 1 gm.-mol. of water vapour had decomposed 0.37 gm.-mol. of salt at 1113° C. Chips of quartz were exposed to the action of salt vapours in dry and moist air at 1100° C. In moist air the effect was more pronounced and the quartz was superficially attacked with the formation of a silicate, apparently  $\text{Na}_2\text{O} \cdot \text{SiO}_2$ , which, however, was insoluble in water. The result of heating commercial ferric oxide in the current of salt vapours

was to cause the formation of magnetic oxide in a black crystalline form. Alumina was also superficially attacked with the formation of an aluminate of the empirical formula about  $2\text{Na}_2\text{O} \cdot 9\text{Al}_2\text{O}_3$ .—H. J. H.

*Radium from American pitchblende ores; Experiments on the extraction of — by chlorination. R. Cable and H. Schlundt. Met. and Chem. Eng., 1918, 13, 460–462.*

The pitchblende ores of Gilpin County, Colorado, contain uraninite very intimately associated with iron pyrites. By ordinary concentration, products containing from 1 to 25% of uranium oxide ( $\text{U}_3\text{O}_8$ ) are obtained; and these are blended to produce high-, medium-, and low-grade concentrates, which, in the present case, contained 17.45, 2.92, and 1.20%  $\text{U}_3\text{O}_8$  and 49.27, 8.312, and 3.43 parts per billion of radium, respectively. Each concentrate was heated in a current of chlorine in a glass tube, producing sulphur monochloride (which was collected separately) and a crystalline sublimate of anhydrous metallic chlorides which was found to contain nearly all the radium. By treating the ore residue and the sublimate together with water, a solution was obtained from which the radium was separated almost quantitatively as radium-barium sulphate. In this way 93.68, 91.78, and 92.34% of the radium originally present was recovered from the high-, medium-, and low-grade concentrates respectively, the radium content of the sulphates being well within the limit required for further concentration by the usual methods.

—W. E. F. P.

*Precipitation of phosphoric acid in the form of ammonium phosphomolybdate. Clarens. See XXIII.*

*Estimation of phosphoric anhydride as ammonium phosphomolybdate. Clarens. See XXIII.*

*Iodine titration of silver nitrate. Scheider. See XXIII.*

### PATENTS.

*Hydrofluoric acid; Process for the preparation of — from bisulphate and fluorspar. B. Teisler, Dohna. Ger. Pat. 302,278, Aug. 21, 1915.*

To a mixture of fluorspar and bisulphate is added a solid such as a metallic sulphate, or carbon in the form of graphite, retort-coke, anthracite, or soot, which is not attacked by sulphuric or hydrofluoric acid. Fusion of the bisulphate is thereby inhibited.—L. A. C.

*Sodium cyanide briquette. A. Kaufman, Assignor to Air Reduction Co., New York. U.S. Pat. 1,262,057, Apr. 9, 1918. Date of appl., Nov. 7, 1916.*

A BRIQUETTE, composed of or containing sodium cyanide, having a porous interior and a relatively impervious exterior, and provided with relatively small supporting ribs on one face, is made by compressing the material into the required shape and heating the block until surface fusion occurs.

—W. E. F. P.

*Sulphurous fumes; Process for treating —. S. W. Young, Palo Alto, Cal., Assignor to The Thlogen Co., Wilmington, Del. U.S. Pat. 1,262,295, Apr. 9, 1918. Date of appl., Oct. 18, 1915. Renewed Nov. 15, 1917.*

In a cyclic process for treating sulphurous fumes, the latter are brought into contact with water and



the solution is treated with an alkaline-earth (barium) sulphide to produce a mixture of sulphur and oxygen-sulphur compounds. This mixture is heated to expel the free sulphur and then reduced with carbon to regenerate the alkaline-earth sulphide.—W. E. F. P.

*Hydrogen peroxide; Process of producing* —. O. Liebknecht, Frankfurt, Germany. U.S. Pat. 1,262,589, Apr. 9, 1918. Date of appl., Jan. 3, 1918.

A CONCENTRATED mineral acid is added gradually to a suspension of sodium perborate to produce, in one operation, a concentrated solution (above 10%) of hydrogen peroxide.—W. E. F. P.

*Magnesium salts; Process for manufacturing* —. *Process of making magnesium chloride*. H. A. Galt, Akron, Assignor to Columbia Chemical Co., Barberton, Ohio. U.S. Pats. (A) 1,262,938 and (B) 1,262,939, Apr. 16, 1918. Date of appl., Jan. 22, 1915.

(A) A MIXTURE of precipitated calcium and magnesium carbonates—obtained by causticising sodium carbonate with hydrated, calcined dolomite—is treated with water and carbon dioxide under pressure, to dissolve the magnesium carbonate. The filtered solution is then heated to re-precipitate magnesium carbonate, which is collected and digested with a solution of calcium chloride (obtained in the manufacture of alkali by the ammonia-soda process) to produce magnesium chloride and calcium carbonate. (B) The calcium chloride solution obtained in the manufacture of alkali by the ammonia-soda process, and the sludge produced by causticising sodium carbonate with magnesium hydroxide (hydrated, calcined magnesite), are heated together under pressure to form magnesium chloride.—W. E. F. P.

*Magnesium chloride liquors; Utilisation of waste* — of the potash industry. [Preparation of hydrochloric acid from alkali chlorides.] Chem. Fabr. Buckau, Magdeburg. Ger. Pat. 302,359, Mar. 28, 1914. Addition to Ger. Pat. 283,096.

ACCORDING to the chief patent hydrochloric acid was recovered from waste magnesium chloride liquors by heating them with sulphuric acid, whilst in previous additions to this patent claims were made to the use of diminished pressure (Ger. Pat. 284,177) and to the substitution of a mixture of sulphur dioxide and oxygen in presence of water for the sulphuric acid (Ger. Pat. 290,876; this J. 1916, 634). It has since been found that in presence of magnesium sulphate hydrochloric acid can also be liberated from alkali chlorides by this process. According to the present patent waste liquors containing magnesium chloride or magnesium sulphate, mixed with alkali chlorides, or double alkali-magnesium chlorides, such as carnallite, with or without addition of alkali chlorides, are heated with sulphuric acid, the hydrochloric acid is condensed, and magnesium sulphate and alkali sulphate are recovered either separately or as a double salt from the residual solution. Instead of distilling off the hydrochloric acid, the concentrated reaction mixture may be allowed to stand at a low temperature, whereby the sulphates crystallise out and a concentrated solution of hydrochloric acid is left.

*Percarbonates; Production of* —. O. Liebknecht, Frankfurt, Germany. U.S. Pat. 1,263,258, Apr. 16, 1918. Date of appl., Jan. 3, 1918.

SODIUM percarbonate is prepared by adding a large proportion of common salt to a dilute solution of hydrogen peroxide containing sodium carbonate.

—W. E. F. P.

*Tin chloride solution; Process for the regeneration of* —. Siemens und Halske A.-G., Siemensstadt. Ger. Pat. 302,040, July 19, 1914.

THE tin chloride solution flows along a trough at the bottom of which are horizontal cathodes. The anodes are suspended from a cover and separated from the cathodes by diaphragms through which the chlorine ions alone pass, the chlorine gas evolved being withdrawn through tubes.—L. A. C.

*Tin oxide; Preparation of very finely divided* — from stannate liquors. G. A. Krause, Munich. Ger. Pat. 302,155, May 3, 1916.

STANNATE liquor freed from impurities and in which the tin has been precipitated in the usual manner, is brought in contact in the form of a very fine spray, e.g., by means of nozzles, rotating plates, etc., with a counter-current of hot air or gas for the purpose of removing the moisture. The tin oxide produced thus is in a very finely divided form and suitable for enamel manufacture.—L. A. C.

*Tin oxide; Process for the preparation of* — from stannate liquors. G. A. Krause, Munich. Ger. Pat. 303,492, May 6, 1916.

THE liquor is sprayed, e.g., by means of blasts, rotating plates, etc., into a current of the warm gas, i.e., carbon dioxide, used for precipitating the tin. The tin oxide so obtained is of very low sp. gr.—L. A. C.

*Gas liquor; Preparation of concentrated ammonia* from —. C. Still, Recklinghausen. Ger. Pat. 302,195, Dec. 7, 1915.

THE liquor is passed down a column in which a portion of the carbon dioxide is removed; it is then divided into two portions, one of which enters another column in which the remainder of the carbon dioxide and a portion of the ammonia are removed; on leaving this column, the liquid unites with the untreated portion and together they enter a third column where they are treated with lime, whereby complete separation of the ammonia is effected. A regulated quantity, or if necessary the whole of the ammoniacal vapour from the third column, is led through the second column, where it becomes charged with the ammonia and carbon dioxide evolved therein. By regulating the two streams of liquid and the flow of vapour an ammonia solution of any required strength may be obtained.—L. A. C.

*Pyrophosphates; Manufacture of alkali and alkaline-earth acid* —. Chem. Fabr. Budenheim L. Utz m.b.H., Mainz. Ger. Pat. 392,672, July 5, 1916.

AMMONIUM monophosphate is heated with an equivalent amount of alkali or alkaline-earth hydroxide or carbonate.—L. A. C.

*Chromic liquors; Process for the electrolytic oxidation of acid* — to chromic acid. P. Goldberg, Berlin-Karlshorst. Ger. Pat. 303,165, Sept. 2, 1916.

CHROMIC liquor exhausted through oxidation, containing, e.g., 65 grms. of chromium oxide and 275 grms. of sulphuric acid per litre, and in which 110 grms. of copper oxide has been dissolved, is electrolysed, the electrodes being lead peroxide and copper plates. The chromium oxide is oxidised to chromic acid to the extent of about 80%, in accordance with the following equation:  $3\text{CuSO}_4 + \text{Cr}_2(\text{SO}_4)_3 + 6\text{H}_2\text{O} = 3\text{Cu} + 2\text{CrO}_3 + 6\text{H}_2\text{SO}_4$ . The very low copper content of the resulting solution is harmless for the oxidation of organic substances and for the solution of metals.—L. A. C.

*Nitrogen; Preparation of — from the air.* Elektrizitätswerk Lonza A.-G., Gampel, Switzerland. Ger. Pat. 302,671, June 20, 1916.

Air or any mixture of nitrogen and oxygen is passed into a solution of ammonium sulphite at at least 70° C., until removal of the oxygen is complete as far as possible. The best results are obtained between 70° and 75° C.; above 75° C. dissociation of the ammonium sulphite takes place, but the ammonia formed can be removed from the nitrogen by adding sulphur dioxide and passing through a further quantity of ammonium sulphite solution, or by bubbling the gas through water or sulphuric acid. Nitrogen sufficiently pure for the manufacture of calcium cyanamide, i.e., containing 0.1–0.2% of oxygen, can be obtained by this process.—L. A. C.

*Molybdenum compounds; Separation of — from substances.* E. H. Westling and C. Andersen. San Francisco, U.S.A. Eng. Pat. 115,274, May 3, 1917 (Appl. No. 6317 of 1917.)

SEE U.S. Pat. 1,250,063 of 1917; this J., 1918, 88 A.  
*Nitrogen; Apparatus for fixation of —.* J. A. Currie, Toronto, Canada. U.S. Pat. 1,263,533, Apr. 23, 1918. Date of appl., Apr. 8, 1916.

SEE Eng. Pat. 105,953 of 1916; this J., 1917, 656.

## VIII.—GLASS; CERAMICS.

*Silica products.—I. Raw materials.* A. Bigot. Ceram. Soc., Refractories Sect., May 14, 1918. [Advance proof.]

The raw materials used are quartzose rocks and sands; quartz and flints are the most free from impurities, quartzites usually containing a notable proportion of foreign matters. Quartzites which had been ground to pass a No. 200 sieve, then suspended in water, and the heaviest particles separated, showed 13.6% impurities as compared with 4.05% in the untreated material, showing that the latter contained about 7% of clay, which acts as an agglomerant in the manufacture of bricks. The material should be heated at 1710° C. for 1 hr. and all samples which remain neither compact nor tough after this heating should be regarded as useless. Many French quartzites expand from 9 to 15% when heated at 1710° C.; in Germany those with an expansion of 9.5–10% are preferred. Flint expands less and is less porous than quartzites and for this reason should be preferable for silica bricks, but practical experience does not confirm this.—A. B. S.

*Magnesite and dolomite; Economy of fuel and increased output in dead-burning of — and in burning of cement.* E. Steiger. Ceram. Soc., Refractories Sect., May 14, 1918. [Advance proof.]

The vertical shaft type of kiln gives a satisfactory product and costs little to instal, but the labour costs in working it are excessive. The rotary kiln is expensive to instal; labour costs are low, but the product is often irregular. The advantages of both types are secured by providing the shaft type of kiln with suitable charging and discharging devices. The latter consists of a vertical shaft in the bottom of the kiln, fitted with a rotary steel head which seals the kiln but, in turning, discharges a definite proportion of the burned material.—A. B. S.

*Corindite; A new refractory and abrasive material called —.* A. Bigot. Ceram. Soc., Refractories Sect., May 14, 1918. [Advance proof.]

BAUXITE when melted by mixing it with anthracite

and blowing air through the mixture (Lecesse, Fr. Pat. 471,513; this J., 1915, 356) yields a vesicular mass containing many small crystals of alumina. To this material the term "corindite" has been assigned. It has a higher melting point than the original bauxite owing to the reduction of the iron oxide to metal and spinel ( $\text{Al}_2\text{O}_3, \text{FeO}$ ) and the separation of some of the more fusible constituents. The product made from white (Var) bauxite is suitable for lining furnaces and other refractory purposes; the material made from red bauxite forms an excellent abrasive and substitute for emery or corundum.—A. B. S.

*Abrasives; Distinction between different — and the detection of carborundum in them.* R. Peters. Pharm. Zentralh., 1917, 58, 217–219, 231–234. Z. angew. Chem., 1918, 31, Ref., 9.

As compared with natural emery the artificial corundum abrasive produced in the electric furnace has a deeper, greyish blue colour. By chemical analysis alone it is difficult to distinguish between the two products, but the artificial material is characterised by the presence of microscopic inclusions of gas and is also less magnetic than natural emery. Carborundum can be detected by heating the previously ignited sample with 20 parts of lead chromate in a hard glass tube, when the mass becomes red hot with liberation of carbon; this test gives appreciable results even with mixtures containing only 5% of carborundum. For the determination of small amounts of carborundum in an abrasive it is necessary to treat the residue insoluble in fused bisulphate, repeatedly with hydrofluoric and sulphuric acids until a constant weight is obtained. With abrasives containing a small proportion of carborundum it is possible to estimate the carbon by heating the previously powdered and ignited sample with potassium chlorate and lead chromate in a current of air for about one hour, the carbon dioxide in the gaseous products being determined by collection in barium hydroxide solution after successive washings with silver nitrate and ferrous sulphate solutions.—D. F. T.

*Dissociation of salt.* Thompson. See VII.

### PATENTS.

[*Porcelain*] *insulating material and body composed thereof.* A. Champion and T. G. McDougal, Assignors to Champion Ignition Co., Flint, Mich. U.S. Pat. 1,262,305, Apr. 9, 1918. Date of appl., Aug. 22, 1917.

CALCINED clay is used as a non-plastic ingredient in the manufacture of insulating material for sparking plugs. A mixture of plastic clay 60%, feldspar 10%, calcined clay and flint 30% is specified.—A. B. S.

*Insulating material; Manufacture of acid-resistant refractory —.* H. Stegmeyer, Charlottenburg. Ger. Pat. 302,834, Mar. 5, 1915.

A MIXTURE of water-glass with one or more filling materials (porcelain dust, sand, graphite, etc.) is worked to a plastic mass and, after being formed into the desired shape, is hardened by heating under pressure in an atmosphere of carbon dioxide or other acid gases or vapours. The filling material may be previously mixed with a small quantity of a substance which forms or liberates an acid on heating, e.g., sulphur, ammonium acetate, sodium formate, etc., and in such case the hardening is effected at a temperature above the melting point of the admixed substance. The product resists the action of concentrated sulphuric acid better than the known kinds of chemical



stoneware, and is much less affected than fire-brick, etc., by variations of temperature.

*Dryer for bricks.* A. E. H. Beyer, Wengeln. Ger. Pat. 301,583, Jan 10, 1913.

A SERIES of chambers arranged side by side in two parallel rows is heated by the waste gases from a continuous kiln, the gases being delivered to the chambers through four separate flues, one above another, leading into downcast pipes in each chamber. The temperature is regulated by means of four sets of dampers. After passing through the desired chambers the gases are led to the chimney.—A. B. S.

*Briquetting coarse materials, which have later to be burned, with clay.* A. Olschewsky, Coswlg. Ger. Pat. 302,704, Dec. 25, 1913.

THE INTERSTICES between the coarser particles are filled with finely ground material of a combustible nature and a little clay is then thoroughly incorporated. The addition of the combustible matter greatly reduces the proportion of clay required to make the material workable.—A. B. S.

*Sintered magnesia; Production of* —. Harburger Chem. Werke Schön und Co., Harburg. Ger. Pat. 302,836, Mar. 1, 1916. (Addition to Ger. Pat. 291,913; this J., 1916, 843.)

A MAGNESIA slurry with the desired proportion of lime, iron oxide, alumina, and silica, prepared in any convenient manner, alone or mixed with slurry prepared from magnesium chloride as described in the chief patent, is heated until it sinters.—A. B. S.

*Unfired refractory products; Production of* —. Petuel'sche Terrain-Ges. A.-G., München-Riesefeld. Ger. Pat. 302,877, July 11, 1916.

REFRACTORY ware which has a high heat-resistance, does not warp or spall under sudden changes of temperature, resists the action of furnace gases and vapours, and has a high crushing strength, is made by mixing silicious material free from fluxes with a calcareous binder, and a small quantity of grog, and hardening the product by means of steam under pressure.—A. B. S.

*Glass; Manufacture of* —. J. A. Chambers, Pittsburg, Pa., U.S.A. Eng. Pat. 103,672, Jan. 26, 1917. (Appl. No. 1339 of 1917.) Under Int. Conv., Jan. 26, 1916.

SEE U.S. Pat. 1,186,076 of 1916; this J., 1916, 842. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 20,415 of 1891, 15,293 of 1906, and 14,417 of 1907; this J., 1893, 46; 1906, 1045; 1908, 628.)

*Glass; Method of and apparatus for drawing sheet* —. W. J. Mellersh-Jackson, London. From The Libbey-Owens Sheet Glass Co., Toledo, Ohio, U.S.A. Eng. Pat. 114,977, Oct. 4, 1917. (Appl. No. 14,372 of 1917.)

SEE U.S. Pat. 1,248,809 of 1917; this J., 1918, 58 A.

*Preparation of very finely divided tin oxide from stannate liquors.* Ger. Pat. 302,155. See VII.

*Preparation of tin oxide from stannate liquors.* Ger. Pat. 303,492. See VII.

## IX.—BUILDING MATERIALS.

*Cement and concrete; Influence of substances on* —. J. C. Witt. Philippine J. Sci., 1918, 13 A., 29—48.

A BIBLIOGRAPHY of the influence of electrolytes on the setting of cement is given. Generalisations from it are difficult to draw because the various experiments are not comparable owing to differences in the conditions, *i.e.*, chemical and physical character of the cements used for the experiments, and concentration and temperature of the solutions. Experiments on four cements have been made, using eleven electrolytes under comparable conditions—NaCl, ZnCl<sub>2</sub>, CuCl<sub>2</sub>, NaNO<sub>3</sub>, KNO<sub>3</sub>, NH<sub>4</sub>NO<sub>3</sub>, Na<sub>2</sub>SO<sub>4</sub>, ZnSO<sub>4</sub>, CuSO<sub>4</sub>, NaHCO<sub>3</sub>, KHCO<sub>3</sub>. The effect was generally to lower the tensile strength, and any increase, which was usually associated with sulphate, was only small. The cement with the highest calcium content seemed most sensitive. The sulphates and chlorides of zinc and copper caused a retardation of the set, which increased with the concentration. The other salts caused a retardation which rose to a maximum as the concentration increased and then diminished. The positive ion seems to have more influence than the negative, but there is no clear connection between this effect and the solubility of the calcium salt formed.—H. J. H.

*Silica and Portland cement; Swelling phenomena in connection with* —. V. Rodt. Chem.-Zeit., 1918, 42, 173—175.

FINELY ground cement or certain forms of silica, when treated with lime-water or certain other solutions such as alum, ammonium carbonate, etc., swell greatly and form a loose, flocculent material. Quartzite and other forms of free silica do not swell so much as silica liberated from certain silicates. The swelling is apparently due to the absorption of water in a manner not fully understood. Portland cement appears to be hydrolysed, lime entering into solution and free silica being separated; the latter absorbs water and swells. It is suggested that the cracking and destruction of concrete may be due to a similar swelling, resulting from the absorption of sulphates.

—A. B. S.

*Silica products. I. Raw materials.* Bigot. See VIII.

*Economy of fuel and increased output in dead-burning of magnesite and dolomite and in burning of cement.* Steiger. See VIII.

### PATENTS.

*Cement.* W. H. Allen, Detroit, Mich. U.S. Pat. 1,261,750, Apr. 9, 1918. Date of appl., Dec. 12, 1917.

A CEMENT is composed of powdered iron oxide (magnetite) and a solution of an acid phosphorus compound (*e.g.*, a 50% solution of phosphoric acid) in proportions to form a paste.—A. B. S.

*Hydraulic cement; Production of* —. F. M. Meyer, Saarbrücken. Ger. Pat. 303,177, Dec. 20, 1912. Under Int. Conv., Aug. 8, 1912.

A MIXTURE of 30 parts of dust from gas-filters used in the dry gas cleaning process and 70 parts of slaked lime produces a hydraulic cement which, when mixed with 3 parts of normal sand, has a crushing strength of 209 kilos. per sq. cm. after eight days and 224 kilos. per sq. cm. after twenty-eight days.—A. B. S.

*Plaster; Burning slow-setting* —. Maschinenfabr. Pötry und Hecking G.m.b.H., Dortmund. Ger. Pat. 301,932, Mar. 17, 1915.

The plaster is separated into particles of approximately uniform size, and each fraction is burnt separately, first in a strongly heated drum until dehydration begins, and then in a less strongly heated drum, which may be heated by the waste gases from the first drum, the temperature being moderated if necessary by introduction of air. In the second drum the heating gases move in the opposite direction to the plaster. As raw gypsum is less easy to grind than the burned material, it is desirable to crush the gypsum only so far as is necessary to obtain a good plaster when burned.

—A. B. S.

*Hollow blocks, closed on all sides; Production of — from cement and artificial stone, by means of ice.* S. Röhm, Munich. Ger. Pat. 302,137, Dec. 28, 1916.

The concrete or artificial stone is packed around a core of ice and allowed to harden, due provision being made for water from melting ice to escape through vents in the concrete or stone.—A. B. S.

*Lime for mortar; Production of hydraulic — from pure lime free from silica.* G. Pralle, Gr.-Hartmannsdorf. Ger. Pat. 302,319, Feb. 16, 1916.

AIR-SLAKED lime which has become hydrated and carbonated is mixed with freshly burned and slaked lime and then stored in a silo. The product gives a mortar which sets as rapidly and firmly as one prepared with natural hydraulic lime.—A. B. S.

*Artificial stones; Hardening calcareous — by gases containing carbon dioxide.* A. Rinne, Charlottenburg. Ger. Pat. 302,635, Nov. 29, 1913.

The absorption of carbon dioxide from the gases used in the hardening process is regulated, e.g., by varying the velocity of the gases or by addition of an inert gas, such as air, so that it does not exceed the following amounts when the temperature of the stone mass is as stated: 25° C., 0.5–2.0% by vol.; 30°, 1.0–3.5%; 35°, 2.0–5.0%; 40°, 3.0–7.0%; 50°, 4.5–10.0%; 60°, 8.0–17.0%; and 70° C., 15–24.0% by vol.—A. B. S.

*Artificial stones; Production of — from filling material and asphalt.* A. Olschewsky, Coswig. Ger. Pat. 302,705, June 6, 1915.

A MIXTURE containing pitch and asphaltic material is formed into the desired shape, dried, and heated above 120° C. in absence of air. The asphalt and the distillation products of the pitch then form a binding agent.—A. B. S.

*Artificial stone; Production of — for use in the construction of ships.* O. Herzfeld, Charlottenburg. Ger. Pat. 303,115, June 12, 1917. (Addition to Ger. Pat. 298,322.)

To concrete made in the usual manner, pumice, trass, and bitumen (as fillers) and jute, esparto grass, or nettle or ramie fibres (as binders) are added in order to produce a material intermediate in character between wood and concrete, with a high degree of elasticity, crushing strength, and tensile strength, and low permeability specially suitable for use in the construction of ships.

—A. B. S.

*Wood; Preservation of —.* H. Bardev, Bad Stuer. Ger. Pat. 303,064, Dec. 16, 1913.

Wood is immersed in boiling water until saturated and all air expelled, and is then immersed in molten

paraffin, heated to a temperature above 100° C., until all water is driven off as shown by the cessation of bubbling. The wood is allowed to cool *in situ* until the paraffin solidifies. After such treatment the wood is very resistant to water and bacteria.—F. C. T.

*Dense sandy product from slag; Production of a —.* Oberschlesische Eisenbahn-Bedarfs-A.-G., Friedenshütte. Ger. Pat. 303,447, Feb. 21, 1914.

SLAG is reduced to fine powder in a disintegrator and thrown a considerable distance so as to cause the particles to solidify externally before they come into contact with water. By this means the production of a light porous slag is avoided and a dense, sandy product is obtained.—A. B. S.

*Slag bricks; Manufacture of —.* The Middlesbrough Slag Co., Ltd., Stockton-on-Tees, and J. L. Major. Bull. Eng. Pat. 115,100, Apr. 30, 1917. (Appl. No. 6121 of 1917.)

*Roofing materials.* H. Abraham, New York. Eng. Pat. 113,778, Oct. 2, 1917. (Appl. No. 14,247 of 1917.) Under Int. Conv., Feb. 27, 1917.

SEE U.S. Pat. 1,226,738 of 1917; this J., 1917, 719.

*Production of sintered magnesia.* Ger. Pat. 302,836. See VIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Ironsand; Smelting of New Zealand —.* J. E. L. Cull. New Zealand J. Sci. Tech., 1918, 1, 43–48.

THE ordinary blast-furnace process is inapplicable to the black ironsand found on the west coast of South Island owing to the physical condition of the ore. Small-scale attempts were made to reduce the ore, after magnetic concentration, by means of a coal dust flame. They were not successful owing to the readiness with which the spongy metallic iron produced was re-oxidised. More success was achieved when the mixed ore and coal dust was fed gradually into a chamber heated by a coal dust flame. When reduction was judged complete the charge was dropped into an electric arc furnace, limestone and fluorspar were added, and the whole was brought to fusion to permit of separation of metal and slag.—H. J. H.

*Malleable cast iron; Influence of silicon and of the duration of annealing on the mechanical and physical properties of —.* E. Lenenberger. Stahl u. Eisen, 1917, 37, 513–521, 601–609. Z. angew. Chem., 1918, 31, Ref., 115.

THE effect of impurities on malleable cast iron is much more marked than on grey cast iron, and the composition can only be altered within narrow limits. The effect of such impurities may be very much greater upon the course of the conversion into malleable iron than upon the casting properties or strength of the material. The lower the silicon content (below 0.65%) the more complete is the decarburisation. Silicon has no effect on the tensile strength. With increasing silicon the hardness increases slightly, while the elongation, reduction of area, and impact values decrease, especially with the more drastic annealing. Increasing the duration of refining lowers the strength and hardness, and raises the ductility and impact test. The specific volume of the annealed material is raised about 0.01 by 1% Si, and further annealing produces little further change. Low-silicon iron shows a



diminution in volume as a result of conversion into malleable iron, while a high silicon content produces an increase of volume. As the duration of annealing increases the limit between the positive and negative volume changes rises from 0.55 to 0.70% Si. The specific electrical resistance of the malleable iron is raised about 20 microhms per cm. cube by 1% Si and that of the untreated iron by 32. Silicon has little effect on the annealing within the limits in which it is usually present, but an increase of silicon content and of the period of annealing produces a product of coarser grain. Although increase in the time of annealing lowers the strength of the malleable iron, yet a long period is advantageous since the toughness is appreciably increased, and for malleable iron of the greatest toughness and ductility a low silicon content and long refining are required.—F. C. TH.

*Zinc; Electrodeposition of* —. C. A. Hansen. Trans. Amer. Inst. Mining Eng., 1918. Met. and Chem. Eng., 1918, 18, 481–482.

THE reactions involved include the deposition of zinc at the cathode according to the usual electrochemical theory, and re-solution of this deposited zinc, depending on the surface exposed and the acidity of the solution, the difference between perfect efficiency and the actual being a measure of this corrosion. An increased efficiency is attained by removing the unsatisfactory solution, and by reducing the current and the concentration of the acid, as well as by the use of organic reagents such as glue or lanolin. High current densities are found to yield rougher deposits, especially with low acid concentrations. Of the impurities present in the electrolyte copper and cadmium are precipitated with the zinc, whilst arsenic and antimony remain in solution. Manganese by itself exerts no harmful effect. Of the cumulative impurities the alkali metals and magnesium are unimportant, but even the smallest traces of nickel, cobalt, vanadium, and uranium act injuriously. Pure solutions yield excellent deposits; pinholes are almost always found when impurities are present. No inorganic addition has been found appreciably to improve the cell.—F. C. TH.

*Copper-zinc alloys; Influence of cadmium on the properties of* —. L. Guillet. Comptes rend., 1918, 166, 735–737.

Two series of alloys containing respectively 60% and 70% Cu and from 0 to 4% Cd have been studied as to their mechanical properties and microstructure. Cadmium has no effect on the mechanical properties of brasses with 70 or 60% Cu unless the cadmium content exceeds 1%. Its influence is first noted in a clearly marked lowering of the resilience. The elongation, under traction, only diminishes when the cadmium content reaches 2%. The modifications of the mechanical properties coincide with the appearance of free cadmium as a structural constituent; this at first forms a marked film round the metallic grains, and then when the cadmium content reaches 2%, separates in round grains. The injurious rôle of cadmium is more marked in  $\alpha$ -brasses than in those composed of  $\alpha$  and  $\beta$  solid solutions.—W. G.

*Patina; An artificial* —. O. Grotian. Z. Elektrochem., 1918, 22, 83.

AN incrustation or patina on copper articles similar to that found on ancient bronzes can be produced by electrolysis of a solution of copper sulphate between copper electrodes for three minutes with a current density of 1 amp. per sq. dm.; the current is then interrupted and the cathode removed

from the liquid. The anode is allowed to remain undisturbed in the liquid for 24 hours. The process is then repeated, alternately passing the current for 3 minutes and leaving the anode for 24 hours. After several repetitions the anode is found to be covered with a beautiful bluish-green non-crystalline patina, which cannot be removed by washing. The film has the composition  $\text{CuO} \cdot 3\text{H}_2\text{O}$ . (See also J. Chem. Soc., June 1918.)—J. F. S.

*Flotation versus cyaniding [of gold and silver ores]*. G. H. Clevenger. Eng. and Min. J., 1918, 105, 743–746.

FLOTATION has not been so successful in the treatment of gold and silver as in that of base metal ores because, whilst mechanical concentration of the latter was imperfect, the cyanide process had reached a high degree of efficiency. Hence flotation increased the extraction of base metals without involving a change in smelting methods; on the other hand, flotation in the case of precious metal ores produces only a concentrate, not bullion, as does the cyanide process. Further, the extraction by cyanide in the majority of cases cannot be improved upon. As regards cost of treatment, flotation is generally cheaper than cyaniding, although the cost of marketing the concentrate is often high. With ores containing base and precious metals, flotation may either remove metals interfering with cyaniding or, if gold and silver accompany the other values into the froth, render cyaniding superfluous. Flotation has been introduced at Cobalt, and in California for the treatment of amalgamation tailings from the Mother Lode; it was tried and abandoned at Tonopah and Goldfield (Nevada), and at Cripple Creek (Colorado).—W. R. S.

*Flotation in relation to gangue minerals*. J. M. McClave. Eng. and Min. J., 1918, 105, 738–739.

CASES of failure have occurred which prove the necessity of carefully studying the gangue minerals of an ore before designing a flotation plant. Ore from different parts of the same mine, although containing the same metalliferous minerals, may require different flotation treatment if the nature of the gangue is different; this has also been proved on synthetic ore mixtures. Presence or absence of calcite, or variation in its quantity, has been known to change the course of flotation. In another instance a small amount of rhodochrosite appearing in the gangue of a silver-lead ore made flotation with the usual oil mixture impossible. Failure to produce a good froth with a zinc-lead-copper ore after successful small-scale tests was traced to the presence of epidote.—W. R. S.

*Flotation; Troubles in* —. O. C. Ralston. Eng. and Min. J., 1918, 105, 735–737. (See also this J., 1918, 12 A.)

ACCIDENTAL over-oiling and addition of too much electrolyte where such is used are among the most common causes of irregular working in ore flotation processes. At the Ohio Copper Co.'s mill, where mine water containing iron and copper sulphate is used, satisfactory extraction seemed impossible until a very small amount of cyanide was added to the pulp; the beneficial effect must be ascribed to the formation of complex cyanides, as free potassium cyanide is known to be detrimental to flotation. Lubricating oil dripping off bearings, organic matter in swampy water, glue, variability in different shipments of oil or in the water supply (e.g., after heavy rain), fluctuation in the feed, soluble matter from recently-set cement, have all been found to disturb the work of flotation plants. Sizing the ore previous to flotation is harmful, and an unsized mixture almost invariably gives the best results.—W. R. S.

## PATENTS.

*Spiegel; Manufacture of* —. A. L. Cromlish, Sharon, Pa. U.S. Pat. 1,231,907, Apr. 9, 1918. Date of appl., Jan. 27, 1917.

Is a process for the recovery of manganese from "flush clinder," a mixture of the latter with coke and tap clinder is heated in a blast furnace to produce a spiegel of low manganese and high phosphorus content.—W. E. F. P.

*Zirconium ores; Process of purifying* —. W. R. Loveman, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,231,948, Apr. 9, 1918. Date of appl., Aug. 18, 1915.

The ore is crushed, fused "with sodium carbonate in a ratio greater than 1 to 6," and the soluble salts washed out from the fused mass. The residue is treated with dilute hydrochloric acid and then calcined.—T. H. B.

*Aluminium-alloy articles; Method of making* —. A. Wilm, Schlachtensee-Berlin, Germany. U.S. Pat. 1,261,987, Apr. 9, 1918. Date of appl., May 24, 1912. Renewed Aug. 23, 1917.

The articles are formed from an alloy produced by the addition of about 0.5% of magnesium to a molten mixture of aluminium, copper, and manganese, containing from 3 to 6% of copper, and manganese equivalent to one-seventh of the copper. They are then heated to somewhat below the congealing point of the alloy (above 420° C.) "until uniform proportional distribution of the metals is attained throughout the mass," and finally cooled at such a rate as to ensure fixation of this condition.—W. E. F. P.

*Aluminium skimmings and analogous material; Method of utilizing* —. *Process of treating aluminium skimmings, screenings, etc.* J. W. Lawrie, Assignor to W. F. Jobbins, Inc., Aurora, Ill. U.S. Pats. (A) 1,262,062 and (B) 1,262,063, Apr. 9, 1918. Dates of appl., Apr. 8, 1913, and Aug. 1, 1914.

(A) SKIMMINGS, slags, dross, or similar waste aluminous materials are treated with a solution of caustic alkali, and after separating zinc, copper, iron, etc., as sulphides, pure alumina is precipitated and separated from the aluminate solution. (B) After removal of larger pieces of free aluminium or alloys the material is boiled with water under pressure to decompose nitrides and carbides, and oxidise the remaining free metal. The residual solids are treated with dilute acid to remove the bulk of the iron, the residue is treated with strong acid to produce a solution of an aluminium salt, from which any copper is precipitated, and the purified aluminium salt solution evaporated.—T. H. B.

*Shaft for roasting furnaces.* D. Balrd, Berkeley, Cal., Assignor to General Chemical Co., New York. U.S. Pat. 1,262,432, Apr. 9, 1918. Date of appl., July 3, 1915.

A hollow shaft, divided into two parallel and continuous chambers of unequal cross-section by a longitudinal partition, and having a number of sets of rabble-arm inlet and outlet ports spaced longitudinally. The ports of a given set are angularly disposed in the same horizontal plane, and the ports of the different sets are separated by opposite edges of the partition.—W. E. F. P.

*Volatilising ores; Process of* —. S. I. Clawson, Salt Lake City, Utah. U.S. Pats. (A) 1,262,452 and (B) 1,262,453, Apr. 9, 1918. Dates of appl., Aug. 4 and 11, 1909.

(A) The ore is preheated by direct treatment with fire-box gases to a temperature below the volatilisa-

tion point of metal chlorides, and fumes and gases are allowed to escape. While the ore is still hot it is treated with a halogen reagent, and the halogen compounds volatilised into a fume arrester. (B) The pulverised ore is heated and treated with chlorine, and the fumes containing chlorides of the metals are passed into an aqueous acid solution, from which the chlorine is subsequently separated by electrolysis to be used again in the process.

—T. H. B.

*Magnesium; Apparatus for the electrolytic preparation of* —. Allgem. deutsches Metallwerk, G.m.b.H., Berlin-Oberschönewalde. Ger. Pat. 302,024, Mar. 3, 1915.

The cover of the electrolytic vessel is provided with a removable bell dipping below the surface of the molten electrolyte. The cathode passes through the bell and can be lowered, raised, or removed at will. The molten magnesium as it forms around the cathode rises into the bell, in which it solidifies, and can be removed with it, dove-tail shaped grooves in the underside of the top of the bell assisting in holding the solidified metal in place.

—B. V. S.

*Pickling iron and steel plates and the like; Machines for* —. J. Paton, Pontypool. Eng. Pat. 115,111. (Appl. Nos. 6765, May 12, and 13,647, Sept. 22, 1917.)

*Screening plant for dealing with metalliferous ores and other substances.* R. F. and H. S. Pochin, Leicester. Eng. Pat. 115,065, Apr. 20, 1917. (Appl. No. 5549 of 1917.)

*Metal briquettes; Apparatus for use in the production of* —. Campbells and Hunter, Ltd., and C. Korte, Leeds. Eng. Pat. 115,258, Apr. 23, 1917. (Appl. No. 6011 of 1917.)

*Buddles for concentrating tin and other ores.* A. Richards, Redruth. Eng. Pat. 115,377, Nov. 15, 1917. (Appl. No. 16,761 of 1917.)

*Blast furnaces and the like; Charging apparatus for* —. A. Sahlin, London. Eng. Pat. 115,405, Mar. 20, 1918. (Appl. No. 4953 of 1918.)

*Process for treating sulphurous fumes.* U.S. Pat. 1,262,295. See VII.

## XI.—ELECTRO-CHEMISTRY.

## PATENTS.

*Voltaic cell.* M. E. Conrad, Atlantic, Iowa, Assignor to C. B. Schoenmehl, Waterbury, Conn. U.S. Pats. (A) 1,262,019 and (B) 1,262,020, Apr. 9, 1918. Date of appl., Feb. 7, 1917.

A NEGATIVE electrode is composed of (A) oxy-arsenide or (B) oxy-phosphide of iron.—B. N.

*Separator; Storage battery* —. W. Morrison, Chicago, Ill. U.S. Pat. 1,262,228, Apr. 9, 1918. Date of appl., Feb. 20, 1918.

The separator is composed of porous non-ligneous material, such as asbestos, impregnated with barium sulphate.—B. N.

*Electrolytic apparatus.* J. B. Herreshoff, jun., Richmond Hill, N.Y. U.S. Pat. 1,262,045, Apr. 9, 1918. Date of appl., Dec. 4, 1915.

SEVERAL bi-polar electrodes are supported in an electrolytic cell by means of lugs projecting later-



ally at the top of each electrode, and off-set in relation to the centre of the electrode. Suitable supports are arranged for each lug, whereby when the electrodes are assembled in the cell or tank a vertical line will pass through the supports and the centre of gravity of the electrode. The electrodes are thus supported in an inclined position at such an angle that impurities or slime will not adhere to either the cathode or anode surfaces, and succeeding electrodes are substantially parallel.—B. N.

[*Electric*] induction furnaces. The Ajax Metal Co., Assignees of J. R. Wyatt, Philadelphia, U.S.A. Eng. Pat. 103,645, Dec. 22, 1916. (Appl. No. 18,388 of 1916.) Under Int. Conv., Jan. 24, 1916.

SEE U.S. Pat. 1,201,671 of 1916; this J., 1916, 1236.

*Electrical apparatus for heating stills, boilers, and the like.* U.S. Pat. 1,262,072. See 11b.

*Process for the regeneration of tin chloride solution.* Ger. Pat. 302,040. See VII.

*Process for the electrolytic oxidation of acid chromic liquors to chromic acid.* Ger. Pat. 303,165. See VII.

## XII.—FATS; OILS; WAXES.

*Determination of fatty acids in butter fat.* Holland and Buckley. See XIXa.

### PATENTS.

*Hydrogenating resistant [acid] fatty material for edible purposes.* C. Ellis, Montclair, N.J. U.S. Pat. 1,261,911, Apr. 9, 1918. Date of appl., Jan. 9, 1915.

STRONGLY acid fatty material is heated with a compound of high boiling point containing an esterifying hydroxyl group, e.g., glycerin, at a temperature above 250° C. in presence of hydrogen, until the acid value of the material is reduced to a relatively low point, a hydrogenating catalyst is added, the temperature reduced, and the contact with hydrogen continued until the material is hydrogenated to the desired degree.—A. de W.

*Fat; Process for the recovery of — from raw materials and by-products.* P. Lindner, Charlottenburg. Ger. Pat. 302,692, Feb. 19, 1914.

LIVING organisms are added to suitable decaying and waste material which is thereby converted in part into living fatty matter, and after a time the organisms are killed and the fat extracted by a suitable process.—L. A. C.

*Glycerin; Process for the purification of crude —.* Vereinigte Chem. Werke A.-G., Charlottenburg. Ger. Pat. 302,826, Aug. 19, 1915.

The glycerin is combined, in the absence of water, and if necessary in the presence of a condensing agent, with an organic acid of which the glyceryl ester is insoluble in water. The ester is purified, hydrolysed, and the glycerin solution concentrated or distilled, if necessary in a vacuum.—L. A. C.

*Apparatus for cleansing and filtering oil and similar liquids.* Eng. Pat. 115,140. See I.

*Manufacture of foodstuffs [from oil-cakes].* U.S. Pat. 1,260,656. See XIXa.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

### PATENTS.

*Lithopone; Method of rendering — light-proof. Light-proof lithopone and process of making same.* G. Rigg, Palmerton, Pa., Assignor to the New Jersey Zinc Co., New York. U.S. Pats. (A) 1,260,811 and (B) 1,260,812, Mar. 26, 1918. Date of appl., Apr. 28, 1916.

(A) LITHOPONE is rendered light-resistant by decomposing the zinc sulphate therein into products unaffected by light, e.g., by grinding the lithopone with barium nitrate so as to convert the zinc sulphate into zinc nitrate and barium sulphate. (B) After removal of soluble constituents the calcined lithopone is treated with barium sulphide to convert the remaining zinc sulphate into zinc sulphide.—C. A. M.

*Resins; Method of separating — from impurities.* J. S. MacLaurin, Wellington, New Zealand. U.S. Pat. 1,260,984, Mar. 26, 1918. Date of appl., Dec. 19, 1917.

RESIN containing heavier impurities is agitated for 15 to 60 mins., in a partially exhausted vessel, with an aqueous solution of salt, the sp. gr. of which is greater than that of the resin, but less than that of the impurities. Air is then admitted and the agitation continued for 2 to 3 mins., after which a vacuum is again created, and the agitation renewed. After settling, the resin is withdrawn from the upper part of the mass and washed free from salt.—C. A. M.

*Oil varnish and process of making same.* O. Boecking, Hamburg, Germany. U.S. Pat. 1,262,302, Apr. 9, 1918. Date of appl., July 6, 1914.

AN oil varnish is prepared by heating together copal resin, fatty oil, and sulphur to form a homogeneous mixture, and then heating at an increased temperature to form a varnish.—A. de W.

*Benzol varnishes.* S. Langguth, Dortmund. Ger. Pat. 302,741, Sept. 26, 1916.

So-called naphthol-pitch, i.e., the condensation product formed in the preparation of  $\alpha$ - and  $\beta$ -naphthol, is extracted with benzol or alcohol, whereby a resin is extracted which is insoluble in soda, soluble in benzol and alcohol, and gives waterproof coatings.—A. de W.

*Shellac substitute [; Naphthol-resin as —].* S. Langguth, Dortmund. Ger. Pat. 302,742, Apr. 7, 1916.

THE resinous condensation products formed in the manufacture of  $\alpha$ - and  $\beta$ -naphthols are extracted with alcohol, and the residue from the evaporation of the same is utilised as a shellac substitute.—A. de W.

*Varnish; Process for the preparation of —.* J. Fluss and J. Tanne, Vienna. Ger. Pat. 302,825, Nov. 25, 1916.

RESINS of the Olibanum species are subjected to dry distillation to free them from their accompanying ethereal oils. The residue is extracted with solvents of low boiling-point, e.g., dichloroethylene, whereby the resinous substances only are removed. The residue may be further extracted with a solvent of higher boiling-point. The resinous constituents are in this way freed from injurious impurities, and yield a hard, quick-drying varnish of a high

degree of gloss, transparency, and resistance to weather and water.—A. de W.

*Litharge cement; Preparation of a* —. Th. Goldschmidt A.-G., Essen. Ger. Pat. 302,852, Apr. 15, 1917.

An improvement on the ordinary litharge-glycerol cement is obtained by using glycol in place of glycerol. A mixture of 75% of litharge and 25% of glycol yields a product which hardens in 1–2 hours, whilst a mixture of 90% of litharge and 10% of glycol sets still more quickly.—A. de W.

*Process of drying varnished surfaces.* U.S. Pat. 1,202,977. See XV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Chemical and mechanical-technical examination of* —. J. G. Fol. Gummi-Zeit., 1917, 31, 761. Z. angew. Chem., 1918, 31, Ref., 45.

In order to form a correct judgment on the quality of rubber it is necessary, in addition to making chemical analysis and vulcanisation tests, to ascertain whether the various original constituents of the rubber and any substances which have been subsequently incorporated in it, remain in the same relative proportions and can be rediscovered by chemical analysis. Preliminary experiments have led to the following conclusions:—The resin content remains practically constant throughout the various operations, although a slight decrease may sometimes be observed after vulcanisation. The proportion of insoluble constituents is decreased by mechanical working. The protein matter is not quantitatively precipitated by the petroleum method applied for the separation of insoluble substances. When free sulphur is present to the extent of several units per cent., it cannot be oxidised quantitatively by nitric acid. The estimation of fillers by boiling with petroleum yielded with the mixture investigated (rubber 50, zinc oxide 62, magnesia 13, sulphur 5) low results, as also did the determination of the ash. The viscosity of the rubber is diminished by mechanical working on the washing rolls, and still more by working on hot rolls; this is attributed to a reduction in the molecular complexity. The indirect method for the estimation of rubber in a vulcanised product gives results which deviate several units per cent. from the quantity of rubber actually used.—D. F. T.

*Rubber; Estimation of the content of unsaponifiable resins in various kinds of* —. P. Dekker. Gummi-Zeit., 1917, 31, 824. Z. angew. Chem., 1917, 31, Ref., 46.

When the mineral oil in a rubber mixing is estimated by the measurement of the unsaponifiable portion of the acetone extract which is soluble in petroleum ether, the accuracy of the result is affected by the fact that a part of the rubber resins resists saponification and is included with the mineral oil. The oxidation products of rubber are completely saponifiable, and the ordinary method of analysis includes them with the saponifiable resins.—D. F. T.

*Balata and gutta percha; Estimation of resin and impurity in* —. P. Dekker. Gummi-Zeit., 1917, 31, 824. Z. angew. Chem., 1917, 31, Ref., 46.

ACETONE is the most trustworthy solvent for the extraction of balata and gutta percha. Ether dissolves a part of the gutta or balata and so gives

high results, whilst with alcohol the gutta becomes plastic on account of the higher boiling point of the solvent, and the extraction is thereby rendered incomplete. When balata is extracted with alcohol a portion of the material insoluble in xylene passes into solution and is included with the resinous matter.—D. F. T.

*Rubber; Depolymerisation of raw* —. A. van Rossem. Gummi-Zeit., 1917, 31, 851. Z. angew. Chem., 1918, 31, Ref., 46.

When rubber is heated for 6 hours at 130° C. in a sealed glass tube containing air, it melts to a brown syrup, whereas this change is not observed in a vacuum or in hydrogen, nitrogen, or carbon dioxide. With 1% rubber solutions in xylene, oxidation does not set in until after several hours, commencing first when a certain limit of relative viscosity has been passed. Depolymerisation occurs much more rapidly in the presence of oxygen than in other gases, oxygen appearing to exert a catalytic effect; the rate of depolymerisation depends also on the nature of the solution. (See also this J., 1917, 1242.)—D. F. T.

#### PATENT.

*Vulcanised rubber (soft rubber, ebonite, etc.); Regeneration of* —. B. J. F. Varenhorst, The Hague, and J. G. Fol, Dell, Netherland Indies. Ger. Pat. 302,995, Mar. 19, 1914.

VULCANISED rubber is heated to a high temperature, without melting, in a vacuum or in an inert gas, and the harmful effect of the air on heated rubber is avoided by rapid cooling, e.g., by treatment with cold water, or solutions of sodium carbonate or alkali.—D. F. T.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Skins; Conservation of* — by desiccation. E. Nihoul. J. Soc. Leather Trades' Chem., 1918, 2, 2–16.

SKINS should be dried whilst raised well above the ground so as to expose both sides to draughts of air, and ground covered with vegetation should be avoided, as it is necessarily cool and damp. If skins are thus dried immediately after flaying, putrefaction should not occur. The sun's light may cause some chemical decomposition, and certainly inhibits bacterial activity. The heat of the sun's rays is not only fatal to many bacteria, but causes coagulation and hardening of the tissues, especially on the surface, and thus further limits bacterial activity. Surface hardening prevents the rapid escape of moisture from the interior. The author, however, does not think that the moist interior will gelatinise at the temperatures reached if ammonia is absent.—F. C. T.

*Tannin-colloids; Fermentation of vegetable* —. W. Moeller. Collegium, 1917, 49–55. Z. angew. Chem., 1918, 31, Ref., 7.

TANNINS, like polyhydric phenols, are converted by certain enzymes (polyphenoloxidases) into humic acids, a change involving simultaneous oxidation and condensation with considerable loss of tanning power. As such fermentation may occur at any time during the storing or use of extracts, tanners should ascertain to what extent formation of humous matter has taken place, and should reject extracts in which the fermentation has been considerable. Phenol, thymol, toluol, and other substances inhibit or entirely prevent the process.

—F. C. T.



*Tannins; Detection of — by the formaldehyde precipitation method.* R. Lauffmann. *Collegium*, 1917, 322.

THE precipitates obtained by the Stiasny formaldehyde test (this J., 1906, 275) for tannins were weighed in an attempt to obtain characteristic figures for different tannins. The results are given in the following table:—

	% of total soluble matter precipitated.	% of tannin precipitated.
Pine bark .. ..	59-65	92-99
"   extract .. ..	40-49	66-80
Oak bark .. ..	61-80	82-101
"   extract .. ..	42-62	64-89
Oak wood .. ..	20-30	26-36
"   extract .. ..	12-19	17-26
Quebracho extract .. ..	94-96	105-107
4 parts oak bark ext. .. ..	62 (calc. 55)	82 (calc. 74)
1 part queb. ext. .. ..		

The variations found were disappointingly large.  
—F. C. T.

*Tanning materials, extracts, chrome liquors, and leather; Critical examination of —.* R. Lauffmann. *Collegium*, 1917, 377 and 401. J. Soc. Leather Trades' Chem., 1918, 2, 58 and 150-151.

THE value of tannins does not depend simply on the percentage of tannin, but on the ratio of tannins to non-tannins, and on the degree of clarification and decolorisation. Badly clarified extracts show a high content of insoluble matter on analysis, and liquors made from them should be cleared by settling before use, or slow tanning and an inferior leather will result. The best practical test for insoluble matter is that of Paessler and Kelt, in which 20 c.c. of extract is diluted with 80 c.c. of water and allowed to stand overnight. The volume of the settled precipitate should be at most a few cubic centimetres and the supernatant liquor should be clear. Sulphites in extracts should be determined by expelling sulphur dioxide and determining sulphates in the residue, and then oxidising the sulphites in the extract and redetermining sulphates. The difference between the two estimations gives sulphites as sulphuric acid. The physical properties of leather deserve consideration. A brittle grain may be due to too great excess of tannin or filling materials, damage to fibres by heat, use of too strong alkalis in liming, or to mineral acid. Paessler's method of detecting free sulphuric acid consists in dialysing 10 grms. of the leather before and after heating at a low temperature *in vacuo* to expel volatile acid. The outer water of the dialyser should be coloured with Congo-red which will indicate diffused acid. If no sulphates are present in the dialysate, free sulphuric acid is absent. If the second dialysate is acid but free from sulphates, non-volatile organic acids (lactic and oxalic) should be sought. There is no means of detecting free sulphuric acid in the presence of non-volatile organic acids. The permeability of leather to water should be tested.

—F. C. T.

*Salt stains [on leather]; Theory of the cause of —.* W. Moeller. *Collegium*, 1917, 7-14, 55-59, 105-124, and 153-161. *Z. angew. Chem.*, 1918, 31, Ref., 8.

A "PHYSIOLOGICAL" tanning process in either the living or dead organism is regarded as the cause of salt stains. Various substances may act as tanning agents, *e.g.*, iron or sulphur, melanines, or sulphur and iron bacteria. The stains are due to very small quantities of the active substances. The most important preventive is the use of some

alkali as a preserving or curing agent. The tanning cannot take place in the presence of alkali.

—F. C. T.

*Leather; Analysis of vegetable sole —.* J. Soc. Leather Trades' Chem., 1918, 2, 51-54 and 82-87.

THE following methods have been agreed upon by the leading English leather trade chemists. Moisture is determined before and after drying, and all results in the analysis are calculated on a standard basis of 14% of moisture. In determining total ash, the charred mass is washed with water if soluble salts are present which hinder the incineration. The residue is ignited at a low red heat, and the solution added and evaporated and the whole gently ignited. Water-soluble matter is extracted by continuous percolation in a Procter extractor. The rate of flow should be about 5.5 c.c. per minute. Only potassium (or sodium) sulphate is to be added to the sulphuric acid in the digestion for the determination of nitrogen. 20 c.c. of sulphuric acid should be used for not more than 1 grm. of leather. A blank test of the reagents should be made with sugar. In the determination of glucose, a little sodium carbonate (about 0.1 grm.) should be used to remove the last trace of lead after the bulk has been removed by sodium sulphate. After heating the solution with hydrochloric acid, reducing sugars are determined by the official method of the American Leather Chemists' Association.—F. C. T.

*Sodium sulphate in leather.* T. Mann. J. Soc. Leather Trades' Chem., 1918, 2, 19-23.

THE usual method of determining sodium sulphate in leather by estimating the total SO<sub>4</sub> in the leather ash, deducting the amount corresponding to the magnesium present, and calculating the remainder as sodium sulphate, leads to quite erroneous results, as the whole of the sulphur present in any form in the leather is returned as magnesium or sodium sulphate. It is preferable to determine the soda present in the leather, since any sodium salt is just as objectionable as the sulphate. 5.5 grms. of leather is incinerated in a platinum dish at a low red heat and the residue weighed. 11 grms. may be used if the ash is very low. 1 c.c. of N/1 sulphuric acid is added for every centigram of ash, the mixture digested over a low flame till the residue is almost dissolved, the water evaporated as far as possible, and the sulphuric acid then fumed off. The residue is ignited and weighed, and then dissolved in 10 c.c. of strong hydrochloric acid by gentle boiling. The solution is filtered into a 110 c.c. flask and made up to the mark. The residue on the filter is silica, and, if barium is present, barium sulphate. 50 c.c. of the filtrate is measured into each of two beakers. The solution in one is made neutral to methyl orange with strong ammonia, then made faintly acid, and a further 15 drops of concentrated hydrochloric acid added. The solution is boiled, 10 c.c. of 7.62% barium chloride solution added, and the whole allowed to digest on the hot plate for half an hour. The precipitate of barium sulphate is filtered off and weighed next day. A quarter of a 7 cm. filter is torn to pieces, shaken to pulp with 5 c.c. of strong hydrochloric acid, and added to the second 50 c.c. of original solution. Strong ammonia is then added until there is a distinct smell of the reagent, the solution boiled and filtered, and the precipitate weighed as ferric oxide and alumina, etc. To the filtrate, 5 c.c. of 2.5% ammonium oxalate solution is added, the mixture boiled, allowed to stand on the hot plate, and filtered. The precipitate is ignited and weighed as calcium oxide. Magnesium is precipitated from the solution by slightly acidifying, adding 10 c.c. or more

of 8.95% sodium phosphate solution, cooling, and making just alkaline with ammonia. After the mixture has stood for half an hour, 25 c.c. of strong ammonia is added and the solution left overnight. The precipitate is washed with dilute ammonia and weighed as magnesium pyrophosphate. From the above determinations the amount of soda is calculated. The percentages of silica, ferric oxide, lime, magnesia, and  $\text{SO}_2$  are added, and the total deducted from the percentage of sulphated ash. The difference is sodium oxide ( $\text{Na}_2\text{O}$ ) and is calculated as sodium sulphate. The procedure is slightly modified if barium is present. It is not satisfactory to precipitate silica, iron, and lime together.—F. C. T.

*Water-soluble matter in leather; Extractor for* —. L. Balderston and W. K. Alsop. J. Amer. Leather Chem. Assoc., 1918, 13, 142–144.

AN extractor is described and illustrated which allows of the maintenance of a constant temperature, the covering of the sample with water throughout the extraction, easy regulation of the flow of water, independent water supply for each sample, easy washing of all parts, and convenient weighing of sample after extraction. The leather is extracted in an alundum thimble, which fits loosely in a copper cylinder passing through and soldered to a copper vessel in which water is maintained at a constant temperature. The water for percolation flows into the copper cylinder from an inverted copper flask, and the rate of flow is regulated by a screw-cock at the bottom of the cylinder.—F. C. T.

*Fat; Determination of* — in leather. J. A. Wilson and E. J. Kern. J. Amer. Leather Chem. Assoc., 1918, 13, 138–141.

A Mixture of equal parts of ether and carbon tetrachloride is the best solvent for fat extraction. It dissolves the fats more completely than petroleum spirit, is not readily inflammable like pure ether, and is not liable to attack the leather by reason of a high boiling point, as is pure carbon tetrachloride. Levi and Orthmann's method (J. Amer. Leather Chem. Assoc., 1915, 10, 445), which consists in shaking the finely divided leather with petroleum spirit, allowing to stand for 24–72 hours, and subsequently evaporating an aliquot part of the solution, gives in general too low results.—F. C. T.

*Leather; [Determination of] moisture in* —. C. D. Wilkinson. J. Soc. Leather Trades' Chem., 1918, 2, 132–133.

Two hours' drying of powdered leather in a vacuum of 60 mm. of mercury at  $98^\circ$ – $100^\circ\text{C}$ . is not sufficient. Results are much worse at about 250 mm.—F. C. T.

*Chromium; Estimation of* — in chrome salts, liquors, leather ashes, and residues. K. Schorlemmer. Collegium, 1917, 345 and 371. J. Soc. Leather Trades' Chem., 1918, 2, 57–58.

Chromium determinations should always be made volumetrically, the gravimetric method being invariably inaccurate owing to the occlusion of alkali salts by the chromium hydroxide, and subsequent production of chromate on ignition. Chrome salts are best oxidised by means of alkali and hydrogen peroxide. The chromate produced is most accurately and rapidly determined by the potassium iodide method. Chromium in leather ashes is oxidised by fusion with magnesia and sodium carbonate. Chrome liquors containing organic matter cannot be oxidised by peroxide solutions, but require strong alkaline permanganate,

which is added at intervals until the colour persists on boiling. The excess is removed by adding alcohol, drop by drop, whilst boiling. If iron is present in chromate solutions, it will be determined as chromium by the potassium iodide method. It is best to free the solution from iron and re-determine the chromium. The titration of the chromate solution with ferrous ammonium sulphate should give a correct result, but apparently does not always do so.—F. C. T.

*Lactic acid; Economy of* — [in leather manufacture]. H. R. Procter. J. Soc. Leather Trades' Chem., 1918, 2, 121–122.

Formic and oxalic acids are efficient substitutes for lactic acid. Boric and sulphurous acids are sufficiently weak to be used with safety, and the latter is very cheap when made by burning sulphur. If organic acids are used in delining, they should be regenerated in the used liquor by the addition of sulphuric acid. If the calculated quantity of organic acid is first used, afterwards  $\frac{1}{3}$  of that amount may be added together with  $\frac{2}{3}$  of the equivalent of sulphuric acid. This may be repeated until the liquor is too foul to use. Formic or oxalic acid is better than mineral acid for adjusting the acidity of tan liquors.—F. C. T.

*Lactic acid; Determination of lactic anhydride in* —. F. C. Thompson and K. Suzuki. J. Soc. Leather Trades' Chem., 1918, 2, 115–121.

PURE lactide was prepared and found to react completely with alkali (including calcium hydroxide) in the cold in ten minutes, if the alkali used was twice the equivalent of the lactide present. Consequently heating with excess of alkali in the analysis of lactic acid is unnecessary, and should a method involving heating show a higher content of lactide than a method using the reaction in the cold, the excess is due to compounds other than lactide which are capable of reacting with alkali. Lactide will effectively remove lime from limed skins. Water slowly converts lactide into lactic acid, the greatest change occurring with the greatest dilution. Consequently determinations of lactide are valueless when lactic acid is to be subsequently used in dilute solution.—F. C. T.

*Lactic acid; Estimation of iron in* —. A. Harvey. J. Soc. Leather Trades' Chem., 1918, 2, 37–38.

Iron in lactic acid can be estimated with great accuracy by a colorimetric method. Potassium ferrocyanide is used as indicator, and the colour developed by a definite quantity of dilute lactic acid is matched by the colour due to a standard iron solution. Potassium thiocyanate is quite useless as indicator.—F. C. T.

#### PATENTS.

*Varnished surfaces [patent leather]; Process of drying* —. G. W. Priest, Newton, Mass. U.S. Pat. 1,262,977, Apr. 16, 1918. Date of appl., June 10, 1911.

The varnished surface of patent leather is finished by exposure to light rich in ultra-violet rays, and may be simultaneously exposed to the action of a heated atmosphere.—F. C. T.

*Leather substitute.* Deutsche Gasglühlicht A.-G. (Auerger.), Berlin. Ger. Pat. 302,329, Jan. 14, 1914. Addition to Ger. Pat. 256,407 (this J., 1913, 438).

Especially good results are obtainable by submitting the skins repeatedly to successive treatments



in two baths, one containing a solution of a mineral salt such as lead acetate, barium chloride, zinc sulphate, or aluminium sulphate, and the other containing sulphuric acid or a solution of sodium sulphate or carbonate. An additional advantage is that the tanned skins need less glycerin to render them pliable.—D. F. T.

*Leather; Production of sheet — from leather waste.* M. Rigell, Buda Pest. Ger. Pat. 302,330, July 16, 1916.

The selected waste after treatment with an aluminium salt and vegetable tannin is dried and submitted to the action of a solution of soap; the dried material may then be soaked in a 10–14% solution of barium chloride before being passed between grooved rolls into a warm solution of an adhesive, after which it is lightly pressed and the immersion repeated in a weaker solution of adhesive, followed by greater pressure; passage through a tanning solution completes the chemical treatment, the material being finally dried.

—D. F. T.

*Tanning of hides and skins.* E. Hupertz, Rodenkirchen. Ger. Pat. 302,992, Apr. 29, 1916.

The tanning of skins with phenols or formaldehyde derivatives of these is effected in the presence of quinoline, or of compounds of quinoline with thiocyanic acid or with an alkali thiocyanate; formates of magnesium, aluminium, potassium, or sodium may also be present, and further additions may be made of vegetable tanning extracts and of sulphite-cellulose waste liquor. With these modifications the ordinary process of tanning is considerably accelerated, and the characteristics of the product may resemble those of material obtained with vegetable tannin.—D. F. T.

*Tanning hides and skins; Process of —.* E. Hupertz, Rodenkirchen. Ger. Pat. 303,601, Jan. 31, 1917. Addition to Ger. Pat. 302,992 (see preceding abstract).

PYROLIGNEOUS acid or one of its salts is added to the tanning liquor described in the chief patent. On account of its content of wood tar, acetone, and methyl alcohol, pyroligneous acid is much more suitable for tannery purposes than is pure acetic acid, having a strong antiseptic action and an accelerating effect on tannage.—F. C. T.

[*Tanning agents.*] *Condensation products of  $\alpha$ - or  $\beta$ -naphthol-monosulphonic acids and formaldehyde; Process for preparing —.* Deutsche Koloniale Gerb- und Farbstoff-Ges., Karlsruhe-Rheinhafen. Ger. Pat. 303,640, Aug. 10, 1915.

THE products obtained by condensing 1 mol. of formaldehyde with 2 mols. of  $\alpha$ - or  $\beta$ -naphthol-monosulphonic acid are further condensed with 1 mol. of formaldehyde or with the equivalent of a formaldehyde-yielding substance. New products are obtained which precipitate gelatin from solution and possess tanning properties like the original condensation products, but which are in general more easily soluble in water.—F. C. T.

*Leather substitute suitable for the reproduction of models; Preparation of a —.* F. Krohmer, Pforzheim-Brötzingen, and A. Schätzle, Pforzheim. Ger. Pat. 302,194, May 21, 1916.

LEATHER dust is mixed to a paste with a glue adhesive and an oil varnish, and the warm mixture used to impregnate bandages or strips of wide-meshed fabric. Before use the strips are softened in warm water and then wrapped to the desired thickness around a suitable model, e.g., for the

production of an artificial limb. When dry the hollow shape is removed from the model and may be polished.—D. F. T.

*Leather substitute; Process for the manufacture of —.* C. Jung-Reinhart, Aggsbach-Markt, Austria. Ger. Pat. 304,096, Dec. 3, 1916.

FIBROUS material, saturated with sulphurised oils, is mixed to a homogeneous mass with alkali caseinate, sulphur, shellac, and balata by means of mixing and rolling machinery. The product, finally obtained in sheets or plates, is of the character of leather and is suitable for soles when containing so little as 5% of balata. The quality and toughness of the material improve with increasing percentages of balata up to a maximum of 15%.

—F. C. T.

*Plastic non-inflammable and odourless material [from gelatin]; Process of manufacture of a —.* R. Pioget, Assignor to Pioget et Cie., Paris. U.S. Pat. 1,263,284, Apr. 16, 1918. Date of appl., Feb. 9, 1917.

SEE Eng. Pat. 106,448 of 1917; this J., 1917, 728.

## XVI.—SOILS; FERTILISERS.

*Nitrifying bacteria; Effect of — on the solubility\* of tricalcium phosphate.* W. P. Kelley. J. Agric. Res., 1918, 12, 671–683.

EXPERIMENTS were made with a light sandy loam soil containing 27 parts of soluble calcium and 17 parts of soluble phosphoric acid per million. Addition of tricalcium phosphate increased both values to a small extent, whilst addition of ammonium sulphate raised the proportion of soluble calcium at once to 90 parts per million. Added ammonium sulphate underwent fairly rapid nitrification, associated with an increase in the proportion of soluble calcium to more than 200 parts per million. Nitrification of ammonium sulphate, in presence of added calcium carbonate, did not increase the solubility of the soil phosphates or added tricalcium phosphate, but in absence of calcium carbonate 0.156 lb. of phosphorus was rendered soluble for every pound of nitrogen oxidised. This proportion is much less than that observed by Hopkins and Whiting in solution cultures of nitrifying bacteria containing ammonium sulphate and tricalcium phosphate; they found that in consequence of the acidity produced by nitrification 2 lb. of phosphorus was dissolved per lb. of nitrogen oxidised to nitrite. In soils the acid formed seems to decompose calcium carbonate in preference to tricalcium phosphate. From these and other experiments the author concludes that the nitrification of organic forms of nitrogen (e.g., dried blood) does not increase the solubility of rock phosphate under field conditions that are favourable to crop growth. It is possible, however, for the nitrification of ammonium sulphate to result in the solution of small amounts of tricalcium phosphate in soil of low carbonate-content.—J. H. L.

*Fertiliser; Comparison of ammonium nitrate with ammonium sulphate as a —.* T. Schloesing, fils. Comptes rend., 1918, 166, 714–718.

POr trials were carried out comparing ammonium nitrate with ammonium sulphate as a nitrogenous fertiliser, maize being the crop grown. The results of the somewhat limited trial indicate that a given weight of ammonium nitrate produces the same

increase in crop as a weight of ammonium sulphate containing the same amount of nitrogen. In a discussion on field trials the author advocates the use of plots of such a size that any given operation can be performed by one person in one day on the whole series of plots.—W. G.

*Animal urine; Conservation and transformation of nitrogen in —.* A. Pickarski. *Z. Spiritus-Ind.*, 1917, 40, 351. *Z. angew. Chem.*, 1918, 31, Ref., 58.

Loss of nitrogen increases as water evaporates from the urine, but the loss is negligible if the liquid is kept in closed containers. Up to 90% of the urea nitrogen may be converted into ammonia; dilution with water decreases the loss of nitrogen, but conversion into ammonia takes place even in the presence of added preservatives. Solid excrements mixed with water and allowed to rot give a fertiliser having properties equal to that of a mixture of solid excrement and urine. The organism which effects fermentation of animal urine is *Micrococcus urea* Cohn.—W. P. S.

*Precipitation of phosphoric acid in the form of ammonium phosphomolybdate.* Clarens. See XXIII.

*Estimation of phosphoric anhydride as ammonium phosphomolybdate.* Clarens. See XXIII.

#### PATENTS.

*Fertiliser for ponds; Production of a —.* M. Engerls, Berlin. Ger. Pat. 302,255, Jan. 11, 1916. Addition to Ger. Pat. 301,550.

REEDS and other water weeds are treated with a mixture of nitric and sulphuric acids (e.g., spent nitrating acid), and the product is neutralised.

—W. P. S.

*Fertiliser; Preparation of a phosphatic water-soluble —.* A.-G. der Chem. Produkten-Fabr. Pommerensdorf, Stettin, and R. Siegler, Pommerensdorf. Ger. Pat. 302,357, Apr. 6, 1916.

RAW phosphate, during or after treatment with sulphuric acid, is treated with sodium bisulphate or sodium sulphate and a quantity of water sufficient to dissolve the phosphoric acid and sodium phosphate; the solution is separated from calcium sulphate, ammonia is added to precipitate iron and aluminium, the solution is then evaporated, and mixed with potassium sulphate or ammonium sulphate or both.—W. P. S.

*Calcium cyanamide; Process for improving the physical character of crude —.* F. Steinmig, Hanover. Ger. Pat. 303,679, Aug. 1, 1915.

CRUDE calcium cyanamide is mixed with a dry, hydrated neutral salt, such as sodium sulphate, free from chlorine, or with an anhydrous salt and subsequently with a quantity of water sufficient to hydrate the salt, and the mixture is stirred until a non-dusty, friable crystalline meal is obtained.

#### XVII.—SUGARS; STARCHES; GUMS.

*Beetroot molasses; Neutral and acid polarisation of vinasses from —.* H. Pellet. *Bull. Assoc. Chim. Sucr.*, 1917, 35, 40—43.

THE polarimetric reading of vinasses from beet molasses is practically the same in presence of hydrochloric acid as in presence of excess of sulphurous acid. This confirms the validity of the use of the direct polarisation of beet molasses taken in presence of sulphurous acid, for comparison

with the inversion polarisation in presence of hydrochloric acid, in calculating the sucrose-content of the molasses. (Cp. Ogilvie, this J., 1912, 1195.)

—J. H. L.

*Furfuroids (furfurosans); Determination of — in products of beet-sugar factories.* I. R. Gillet. *Bull. Assoc. Chim. Sucr.*, 1917, 35, 53—62.

THE pectic substances of beets, according to Herzfeld, yield a certain amount of furfural when distilled with hydrochloric acid by Tollens' method. With the object of adapting this method to the determination of pectic substances in beet juices, etc., the author first investigated the behaviour of sucrose when distilled with hydrochloric acid by the Tollens-Couder procedure. According to Chalmot, sucrose yields not more than 0.2% of furfural, but the author, in spite of precautions to ensure uniformity of procedure, obtained results fluctuating between 0.38 and 0.76%. In operating on materials containing a large proportion of sucrose or hexoses, therefore, it is not possible to make a satisfactory correction for the influence of the sugar, but it appears practicable to eliminate the latter by fermentation before the distillation with acid. (See also *J. Chem. Soc.*, July, 1918.)

—J. H. L.

#### XVIII.—FERMENTATION INDUSTRIES.

*Mucor Boulard; Acids produced by —.* Bettinger and Delavalle. *Bull. Assoc. Chim. Sucr.*, 1917, 35, 13—15. (See also this J., 1918, 219 A.)

DATA respecting the production of acids by *Mucor Boulard* in sugared extract of malt-combs and also in liquefied grain mash, are given, confirming the general statements previously made (*loc. cit.*). In the liquefied grain mash, the initial acidity of which corresponded with 1.85 grms. of sulphuric acid per litre, the organism was cultivated at 30° and at 37° C. and the mycelium was immersed at frequent intervals; in 4 days the acidity had increased by 1.7—2 grms. per litre at the lower and by 0.5—0.6 grm. per litre at the higher temperature. After the fourth day the acidity diminished in both cases. In sugared extract of malt-combs considerably higher acidities were produced under favourable conditions, as regards aeration, temperature, and sugar-content of the medium. About 21% of the dry substance of the plants grown in this medium was soluble in ether.—J. H. L.

*Cognacs and wine distillates; Composition of —.* A. Jonscher. *Z. öfientl. Chem.*, 1918, 24, 25—36.

ANALYSES of French and German wine distillates show that the former now more nearly resemble the latter in composition, particularly as regards the amount of higher alcohols. Whilst the French products used to contain from 150 to 300 mgrms. of higher alcohols per 100 c.c. of absolute alcohol, the quantity now rarely exceeds 80 mgrms. Of twenty-six German cognacs examined, only five contained more than 100 mgrms. of higher alcohols per 100 c.c. of alcohol; in the remainder of the samples the quantity varied from 20 to 79.8 mgrms. Three out of five samples of French cognac contained less than 100 mgrms. of higher alcohols.—W. P. S.

*Fermentation of vegetable tannin-colloids.* Moeller. See XV.

*Neutral and acid polarisation of vinasses from beet molasses.* Pellet. See XVII.



## PATENTS.

*Beverage; Non-alcoholic carbonated* —. W. J. Eisenbeiss, Chicago, Ill. U.S. Pat. 1,262,472, Apr. 9, 1918. Date of appl., Nov. 4, 1916.

BRAN is steeped in a saline liquid to extract the albumin, water and sweetening matter are added, and the whole is boiled and hopped. Colouring matter may be added, and when cooled the liquid may be acidulated and carbonated, or treated with vinegar and slightly carbonated and then filtered and again carbonated. Special claim is made to the extraction of albumin, in a soluble form, from vegetable matters, by steeping them in a saline liquid at 38°–50° C., whereby peptase is set free.

—J. H. L.

*Beer rich in protein; Production of* —. Nathan Institut A.-G., Zürich. Ger. Pat. 301,830, Sept. 12, 1915. Under Int. Conv., Sept. 9, 1915.

THE primary fermentation is carried out, with a sufficient quantity of yeast, at a low temperature, e.g., 0°–6° C., so as to effect the maximum decomposition of sugar and proteins whilst minimising the assimilation of nitrogenous matters by the yeast. To accelerate fermentation the amount of yeast employed may be increased, even to 3 litres or more per 100 litres of wort.—J. H. L.

*Preparation of a lipid nitrogenous compound from yeast*. Ger. Pat. 302,220. See XX.

## XIXA.—FOODS.

*Butter fat; Determination of fatty acids in* —. E. B. Holland and J. P. Buckley. J. Agric. Res., 1918, 12, 719–732.

THE method described consists in the direct esterification of the fat and subsequent fractionation of the resulting esters. For the esterification, 150 grms. of the fat is boiled for 24 hrs. with 400 c.c. of absolute alcohol containing 8 grms. of hydrogen chloride or 4 c.c. of concentrated sulphuric acid; after cooling, 50 c.c. of ether and 150 grms. of magnesium chloride are added, the mixture is shaken gently, the lower layer is then drawn off, and the esters are shaken two or three times with 50 c.c. of ether and 50 c.c. of alcoholic magnesium chloride solution (250 grms. of dry magnesium chloride in 500 c.c. of absolute alcohol). The esters are filtered (the filter is afterwards washed with ether and the ethereal solution added to the alcoholic solution and a further small quantity of esters separated from the latter) and distilled; the condenser is filled with water at the outset, but no circulation of water is permitted during the distillation. After the alcohol and ether have been separated, the distillate between 85° and 365° C. is collected. The combined distillates from two portions of 300 grms. of butter fat are then redistilled and fractions are collected separately at the following temperatures:—125°–180° (ethyl butyrate, caproate, and oleate); 180°–225° (ethyl caproate, caprylate, and oleate); 225°–270° (ethyl caprylate, caprate, and oleate); 270°–300° (ethyl caprate, laurate, and oleate); 300°–325° (ethyl laurate, myristate, and oleate); 325°–365° (ethyl myristate, palmitate, and oleate). The proportions of the various fatty acids are then calculated from the saponification and iodine values of the fractions. The percentage quantities of fatty acids in butter fat were found to be: Caproic acid, 1.36; caprylic acid, 0.97; capric acid, 1.83; lauric acid, 6.89; myristic acid, 22.62. Butyric acid (3.15%) and palmitic acid (19.23%) were determined by difference, stearic acid (11.38) by crystallisation, and oleic acid (27.34%) was calculated from the iodine value of the insoluble acids.—W. P. S.

*Fat in milk; Influence of paraffin on the Gerber method for the determination of* —. F. Reiss. Milchwirtsch. Zentr., 1918, 47, 52–54.

PARAFFIN wax is sometimes used on the stoppers of sulphuric acid carboys, and it may happen that some of the paraffin finds its way into the acid. When such acid is used in the Gerber method, the paraffin separates together with the butter fat and increases the quantity of the latter proportionally; each 0.01 c.c. or 0.0079 grm. of paraffin increases the amount of butter fat by 0.10%.—W. P. S.

*Kafir kernel; Physical and chemical study of* —. G. L. Bidwell. U.S. Dept. Agric. Bull. No. 634, Apr. 4, 1918. [6 pages.]

THE composition of kafir kernel (dawn kafir or dwarf blackhull) and its various parts is as follows, the figures being calculated on the dry substance:—

	% of whole kafir	Ash.	Ether extract.	Crude protein.	Crude fibre.	Pentosans.	Starch.
Whole kafir ..	100	1.8	4.1	12.7	1.8	3.3	61.9
Pure endo-sperm ..	83.9	0.3	0.7	12.7	0.8	1.9	69.3
Pure germ ..	10.0	13.2	31.5	19.3	3.8	6.1	—
Pure bran ..	6.1	2.0	6.8	4.8	16.2	18.4	—
Starchy endo-sperm ..	35.0	0.3	0.8	10.1	0.8	1.6	70.4
Horny endo-sperm ..	48.9	0.3	0.7	13.7	0.7	1.6	68.8

These figures show that kafir kernel has a composition similar to that of maize.—W. P. S.

*Salicylic acid in foods; Estimation of* —. H. D. Steenberg. Chem. Weekblad, 1917, 14, 914–921. Z. angew. Chem., 1918, 31, Ref., 106.

A ROUND-BOTTOMED flask containing 66 c.c. of benzene and a few pieces of tile is connected with an extraction apparatus containing 50 c.c. of benzene to which an amount of the material under examination equivalent to about 5 mgrms. of salicylic acid is added, together with 2 c.c. of 4 N sulphuric acid, a little alcohol, and just sufficient water to cause the benzene to run over. The whole is connected with a condenser and the extraction carried on for six hours. After cooling, a few c.c. of benzene from the extractor are shaken with water and a drop of ferric chloride solution (1 in 1000). If this gives no salicylic acid reaction the whole of the benzene is shaken in a separating funnel with two separate 5 c.c. of N/10 potassium hydroxide and then washed with water. The resulting united aqueous extracts are evaporated to dryness and the residue transferred by means of 10 c.c. of N/10 hydrochloric acid and 20 c.c. of water to a long-necked stoppered flask. To this are added 1 c.c. of hydrochloric acid (sp. gr. 1.13) and 10 c.c. of a solution containing 1.67 grms. of potassium bromate and 6 grms. of potassium bromide per litre, a blank test being performed at the same time. After a quarter of an hour 6 c.c. of N/2 potassium iodide and 1 c.c. of chloroform are added to each, and the liberated iodine titrated with N/80 thiosulphate.

—W. G.

*Straw; Treatment of — for preparation of fodder*. B. Tollens. J. Landwirts., 1917, 65, 219–232. Z. angew. Chem., 1918, 31, Ref., 105–106.

ACCORDING to Lehmann the digestibility of the organic matter of winter-sown cereal straw can be taken as 42%, and the different processes of treatment aim at increasing this value. Lehmann's pressure method using a minimum of soda (2% of

the straw) increases the digestibility to 60–62%, and the product can be fed without danger. By employing more soda (10% of the weight of the straw) the digestibility can be increased to 74%, but the product must be washed with water, thus entailing a loss of 30% of the original straw. The increased value of these products balances this loss, but it is an open question whether the process justifies the increased expenditure on soda. Lehmann's process without pressure, but using more soda (10%), also requires that the material be washed, and again there is a loss of organic matter. This method increases the digestibility to over 70%, but requires a longer time than the pressure method. Similar results were obtained by Colmann in experiments carried out at Dahlem, using closed boilers without pressure and with excess of soda. The treatment without pressure or shaking yields a material ("Kraftstroh") which is still of the nature of straw. By treatment under pressure a uniform mass is obtained which in the wet state can be fed satisfactorily; in the dry state, however, it is similar to cardboard and is not satisfactory to feed. If the wet "Kraftstroh" is mixed with molasses, potatoes, or with products rich in nitrogen and dried, a mixture is obtained which keeps well, can be transported, and is readily consumed. The treated straw has a digestibility exceeding that of meadow hay (60%) and equal to that of clover (70%). It is rich in carbohydrates but poor in nitrogen, and would require supplementing in this respect as a food.—W. G.

*Straw; Method of distinguishing treated from raw — and the determination of digestible fibre.*  
F. Mach and P. Lederle. Landw. Versuchstationen, 1917, 90, 269–289. Z. angew. Chem., 1918, 31, 1918.

A METHOD for distinguishing between raw straw and that which has been treated with alkali consists in determining the soluble portion of the crude cellulose by treating the material with ammoniacal cupric hydroxide and precipitating the dissolved cellulose, etc., with alcohol and acetic acid; the insoluble portion is then used for the determination of the crude fibre.—W. P. S.

*Feeding stuffs; Influence of natural and artificial drying on the digestibility and assimilation of —.* Völitz. Chem.-Zeit., 1918, 42, 147.

NATURALLY dried fodders, such as hay, are but slightly less digestible and assimilable than freshly mown grass, but artificial drying produces considerable changes; loss of digestibility in this case is due principally to the temperature which the substance attains during the process, and is less dependent on the temperature of the drying apparatus. The proteins are most affected, and 36% of the digestible protein may be lost; carbohydrates are less affected, and fats least of all.

—W. P. S.

#### PATENTS.

*Vegetable-ivory meal and method of making same.*  
R. M. Chapman, Hammond, Ind., Assignor to R. W. Chapin, Chicago, Ill., and G. M. Chapin, Hammond. U.S. Pat. 1,260,327, Mar. 26, 1918. Date of appl., Dec. 29, 1917.

VEGETABLE-IVORY material is heated at about 280°–320° F. (138°–160° C.) in a bath of 1 to 2% sulphuric acid or other disintegrating liquid until it is friable and is then reduced to a porous meal, which contains mannose, dextrose, and a fairly high proportion of fat.—C. A. M.

*Foodstuffs; Manufacture of — [from oil-cakes].*  
H. Bollmann, Hamburg, Germany. U.S. Pat. 1,260,656, Mar. 26, 1918. Date of appl., Jan. 3, 1918.

EXPRESSED oil-cake or other foodstuff is leached in a heated condition with a solvent composed of alcohol and a volatile liquid hydrocarbon (benzene) which will dissolve fat, so as to extract the oil and bitter substances and leave a residue suitable for food.—C. A. M.

*Steritisation of milk and other albuminous liquids.*  
H. Laessig, Berlin. Ger. Pat. 301,966, Sept. 5, 1915.

THE liquid is heated almost to the necessary temperature by being caused to flow over a corrugated surface maintained at a temperature insufficient to cause coagulation of the albumin, and the flowing liquid is raised to the degree needful for rapid sterilisation by radiation from a heated surface above.—D. F. T.

*Cattle feeding stuff; Manufacture of —.* Futtermittel- u. Getreidehandel-A.-G., Halle. Ger. Pat. 302,342, June 30, 1914.

IN the preparation of compressed cakes of feeding stuffs by atomising the material in contact with a spray of drying oil to act as binding material, the absorption of the oil by the feeding stuffs, as far as possible, restricted to the surface layer of the latter by mixing with the feeding stuff an inert material, such as clay, bolus, or the like, which has a greater absorptive power for the oil, or by dissolving in the oil a substance, such as rosin, which increases its viscosity, or by using an aqueous solution or emulsion of the oil.

*Baking powder.* L. Elkan Erben, G.m.b.H., Charlottenburg. Ger. Pat. 303,058, Feb. 11, 1916.

A BAKING powder, produced by mixing carbonates with protein acids such as casein or nucleic acid, possesses the advantage that the carbon dioxide is liberated only slowly, and that the resulting protein salts possess agglutinating properties.—D. F. T.

*Hydrogenating resistant [acid] fatty material for edible purposes.* U.S. Pat. 1,262,911. See XII.

#### XIXB.—WATER PURIFICATION; SANITATION.

*Water analysis in the field.* G. W. Heise and A. S. Behrman. Philippine J. Sci., 1918, 13, A, 1–17.

THE methods are based on those of Leighton (U.S. Geol. Survey, Water Supply Paper (1905) No. 151). Iron is determined colorimetrically with potassium thiocyanate. Sulphates after precipitation as barium sulphate and suspended matter are determined turbidimetrically by measuring the depth of turbid liquid in a tube required to extinguish the illumination at one end due to a flash lamp. Titrations of alkalinity, acidity, etc., are made against standard pellets of reagent in a glazed porcelain mortar, the water being measured in a 100 c.c. graduated cylinder. Accuracy to within 5% is claimed. Pellets of sodium bisulphate bound with glucose are used with "butter yellow" as indicator for the determination of alkali, and with phenolphthalein to measure normal carbonates. Sodium carbonate pellets bound with kaolin are used for titrations of acidity, different indicators being used to distinguish between mineral acids and carbonic acid. For chlorides, pellets of silver nitrate made with a kaolin binder are used, and



potassium chromate indicator. For total hardness determination see following abstract. Bacteriological examinations are made in apparatus sterilised in the laboratory and carried in sealed tins or paper envelopes. The whole equipment is portable and weighs less than 20 kilos.—H. J. H.

*Total hardness of water; Field methods for determination of —.* A. S. Behrman. Philippine J. Sci., 1918, 13, A, 21—27.

BLACHER'S method of titration with potassium palmitate (this J., 1912, 553; 1913, 158) has been adapted to field conditions. Standard pellets are made by neutralising a known weight of palmitic acid with potash in alcoholic solution. The residue after evaporation to dryness is incorporated with glucose and made into pills. These lose strength on storing, and require frequent checking by titration with calcium sulphate using phenolphthalein as indicator. Water to be titrated is rendered just acid with sulphuric acid, and carbon dioxide is removed by bubbling air through the liquid, which is then neutralised with alcoholic potash. Pellets are now added until a deep red coloration is produced. An alternative method is to crush a number of pellets in a mortar and add the neutralised water from a graduated cylinder until the end point is reached. The result may be checked by adding one or more pellets and again titrating with water to the end point. An accuracy of about 5% is claimed.—H. J. H.

*Activated sludge and treatment of packing-house wastes.* L. Pearse. J. Amer. Leather Chem. Assoc., 1918, 13, 145—159.

THE activated sludge process offers the best promise for the treatment of packing-house wastes. Extensive experiments are to be carried out in Chicago. Results obtained up to the present (compare this J., 1916, 904) indicate that 4—6 cub. ft. of air per second per gallon of sewage is sufficient to produce a stable effluent. The larger quantity is needed in winter. Full details of the experiments are given.—F. C. T.

*Dust in mine air; Estimation of injurious — by the Kotzé konimeter.* J. Innes. J. Chem. Met. Min. Soc. S. Africa, 1918, 18, 199—206.

THE gravimetric results obtained by the sugar-tube method hitherto employed are misleading in the absence of efficient means for eliminating non-injurious dust, i.e., particles of greater diameter than 5 microns, since the weight of the deposit is largely that of the coarser constituents. In the konimeter method a known quantity of the air is caused to impinge at a high velocity (30—120 metres per second) from a fine nozzle (0.6—1.0 mm. in diameter) on to the surface of a glass slide which is thinly coated with vaseline, and in the dust spot so obtained separate counts are made of the particles of larger and smaller diameter than 5 microns, the latter constituting "injurious dust" from the miners' plithis standpoint. The apparatus consists of a small suction pump the piston of which is actuated by a spiral spring and contained in a cylinder of such dimensions that, on release of the spring by means of a trigger, the piston displaces exactly 5 c.c. This quantity of air is drawn rapidly through the nozzle and impinges on the vaselined plate before passing into the cylinder. The concordance of the results obtainable with the konimeter, and the misleading character of the indications afforded by the gravimetric method, are shown in micrographic and other data relating to a series of 36 tests.

—W. M. F. P.

*"Perocide" liquor as a substitute for copperlime liquor.* O. Appel. Apoth.-Zeit., 1917, 32, 275. Z. angew. Chem., 1918, 31, Ref., 21.

A MIXTURE of sulphates of the rare earths, consisting mainly of cerium and didymium sulphates, obtained as a by-product in the manufacture of incandescence gas mantles, is sold under the name of "perocide." It contains at least 45% of cerium oxide, and when mixed with milk-of-lime may be used for destroying mildew on vines, scab on stone-fruit trees, etc. Its insecticidal action is about one-half of that of mixtures prepared from copper sulphate and lime.—W. P. S.

#### PATENTS.

*Disinfectant.* Tetralin G.m.b.H., Berlin. Ger. Pat. 302,003, May 27, 1916.

THE *ar*-tetrahydronaphthols (this J., 1890, 603) and their derivatives substituted in the aromatic nucleus by nitro groups, mercury salt residues, alkyl groups, or halogens, possess marked disinfectant power, greatly in excess of that exhibited by the parent naphthols; they may be applied in the free condition or in the form of salts, either alone or dissolved in sulphuric acid or in aqueous solutions of alkali salts of the fatty or sulphonated fatty acids, with or without the additional presence of formaldehyde.—D. F. T.

*Insecticide; Production of an —.* O. Hinsberg, Nackenheim. Ger. Pat. 302,238, Apr. 24, 1917.

TOBACCO waste, quassia wood, capsicum, valerian, or similar material is subjected to dry distillation, and the distillate is collected in a suitable solvent. The solution forms a basis for sprays.—W. P. S.

*Disinfectant dressing; Production of a —.* Deutsche Desinfektionsmittelfabrik Lipschitz u. Co., Berlin-Wilmersdorf. Ger. Pat. 303,737, Aug. 26, 1915.

Dry powdered peat is carefully treated with caustic alkali solution, and the mixture is then saturated with chlorine at a temperature below 90° C. The dry powdery product retains most of its chlorine even after long storage, and is capable of absorbing up to ten times its own weight of liquids; it is therefore of value as a dressing, particularly for suppurating wounds.—D. F. T.

*Disinfectants containing halogenated phenols or phenolic salts; Production of —.* Carholsäure-Fabrik G.m.b.H., Ladenburg. Ger. Pat. 303,738, July 13, 1915.

SALTS of phenol with the alkali or alkaline-earth metals are mixed with hypohalogenites of the alkali or alkaline-earth metals in the absence of a solvent. Calcium phenoxide and bleaching powder in this way yield the calcium salt of mono-, di-, or trichlorophenol, and the product possesses the advantage that it can be sprinkled.—D. F. T.

*Nitro-compounds of phenols; Non-explosive disinfectants and preservatives containing —.* Chem. Fabr. Flörsheim H. Noerdlinger. Ger. Pat. 304,127, Feb. 20, 1914.

THE disinfecting and preserving properties of the alkali salts obtained by dissolving in alkali solution the product obtained by passing air, oxygen, or ozonised air through heated wood-tar (Ger. Pat. 163,446 and Eng. Pat. 8590a of 1905; this J., 1906, 367, 529), are increased by admixture of nitro-compounds of phenols. The concentrated mixtures so obtained may be reduced to a fine dry powder by heating on a water-bath, and, in some cases, at reduced pressure.—B. V. S.

*Preparation of a derivative of hexamethylenetetramine.* Ger. Pat. 393,450. See XX.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Peppermint oil; Japanese* —. H. Walbaum. J. prakt. Chem., 1917, 96, 245-250.

THE fraction of Japanese peppermint oil of b.pt. 250°-310° C., sp. gr. at 15° C. 0.9490, consists largely of *n*-hexen-1-yl phenylacetate

$\text{CH}_2(\text{C}_6\text{H}_5)\text{CO}_2\text{CH}_2\text{CH}_2\text{CH}:\text{CH}-\text{CH}_2-\text{CH}_3$ , which when prepared in a pure condition is a liquid of onion-like odour, b.pt. 135°-136° C. at 4 mm., 290° C. at 760 mm.; sp. gr. at 15° C. 1.000;  $n_D^{25}$  = 1.49810. Several other synthetic esters of the alcohol are described. The isomeric  $\beta$ -alcohol has already been detected together with its corresponding aldehyde in the green leaves of numerous plants (Curtius and Franzen, this J., 1912, 706, 958; 1914, 1026). (See also J. Chem. Soc., June, 1918.)

—D. F. T.

*Determination of lactic anhydride in lactic acid.* Thompson and Suzuki. See XV.

*Estimation of iron in lactic acid.* Harvey. See XV.

### PATENTS.

*Salicylic acid; Manufacture of* —. M. Theodor. London. Eng. Pat. 115,080, Apr. 24, 1917. (Appl. No. 5755 of 1917.)

CRUDE salicylic acid is dried, preferably under vacuum, and heated to about 170° C. The melted acid is kept in thorough agitation by means of a stirrer, and a current of dry air, or other gas which does not act on the acid, previously heated to about the same temperature, is blown through the mass. The gas containing the volatilised salicylic acid is then passed through a condenser, or series of condensers, maintained at suitable temperatures, e.g., a series of three may be used, the first at 120° C., the second at 95° C., and the third at 40° C. On leaving the last of these, the gas is passed through a trap or filter to retain any crystals not previously deposited. Any impurities present in the crude acid which may be volatilised, e.g., phenol, etc., remain in the gas as vapour after passing through the apparatus.—L. A. C.

*Carbon tetrachloride; Process of separating — from other compounds. Process of making carbon tetrachloride from carbon bisulphide and chlorine.* G. Baillio, Assignor to Iseo Chemical Co., Niagara Falls, N.Y. U.S. Pats. (A) 1,260,621 and (B) 1,260,622, Mar. 26, 1918. Date of appl., Apr. 30, 1917.

(A) A MIXTURE of carbon tetrachloride, sulphur monochloride, and carbon bisulphide is treated with sufficient tin to decompose all the sulphur monochloride into free sulphur and stannic chloride. The sulphur is separated, and sufficient water added to hydrate the stannic chloride. The carbon tetrachloride is then separated from the mixture by distillation. (B) Carbon bisulphide containing free sulphur is treated with an excess of chlorine in the presence of a catalyst, carbon tetrachloride and sulphur dichloride being formed. On the addition of a further quantity of carbon bisulphide to the mixture, this is converted into carbon tetrachloride, and sulphur monochloride is produced. The constituents of the mixture are then separated as described above, the sulphur monochloride converted into sulphur dichloride by treatment with chlorine, and this used for the production of more carbon tetrachloride.—L. A. C.

*Organic condensation products; Process for the synthesis of* —. H. Hilbert, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,261,811, Apr. 9, 1918. Date of appl., Nov. 28, 1913.

AN organic compound containing a carbon atom and an oxygen atom united by a double bond (e.g., an aldehyde) is treated with an alkali metal (e.g., sodium) in the presence of an anhydrous liquid organic compound capable of forming a salt with the alkali metal.—L. A. C.

*Chlorinated hydrocarbons; Manufacture of — [from natural gas].* J. H. Garner and H. D. Clayton, Pittsburgh, Pa., Assignors to Metals Research Co., New York. U.S. Pat. 1,262,769, Apr. 16, 1918. Date of appl., June 23, 1915.

HYDROCARBONS (natural gas) and chlorine are mixed in a body of charcoal at a low temperature, and then passed through a body of charcoal heated to about 300° C. The chlorinated hydrocarbons are condensed, and the hydrogen chloride absorbed in water.—L. A. C.

*p-Aminophenol; Therapeutically valuable derivatives of* —. M. Hartmann, Assignor to Society of Chemical Industry in Basle, Switzerland. U.S. Pat. 1,263,238, Apr. 16, 1918. Date of appl., June 29, 1917.

ACYL derivatives of *p*-aminophenol allyl ether, e.g., *p*-acetaminophenyl allyl ether (leadlets, m. pt. 94° C.), which are crystalline substances, fairly soluble in water and readily soluble in alcohol, ether, and other organic solvents, possess narcotic, sedative, and anti-neuralgic properties.—D. F. T.

*Mercury succinimide; Production of derivatives of — easily soluble in water.* A. Bernard Nachfolger-Einhornapotheke, Berlin. Ger. Pat. 302,156, Sep. 16, 1916.

MOLECULAR proportions of an alkali salt of monomethylarsinic acid and mercury succinimide are mixed in aqueous solution with the formation of a double compound which may be isolated by crystallisation or evaporation preferably under reduced pressure. The compounds obtained in this way will yield 8% solutions in water, and 3-4% solutions, which contain more than 0.01 gm. of mercury per c.c., can be injected subcutaneously without causing the irritation commonly observed with mercury salts, but yet exerting a rapid therapeutic action. The double salts in question are insoluble in the common organic solvents and are decomposed by mineral acids.—D. F. T.

*Lipoid, nitrogenous compound; Production of a — from yeast.* Ges. für Chem. Ind. in Basel. Ger. Pat. 302,229, Jan. 25, 1916. Under Int. Conv., Dec. 19, 1914.

AFTER removing the solvent from the alcoholic extract of yeast by adding water and distilling under reduced pressure, the resulting emulsion is shaken with ether; the ethereal solution is then treated with acetone, when the fats, cholesterol, and ethereal oils remain in solution, whilst a substance is precipitated capable of coagulating blood; the precipitate is again extracted with ether and the ethereal solution evaporated to dryness under reduced pressure.—D. F. T.

*Digestive preparation enriched in crepsin; Production of a* —. F. W. Hopmann, Cologne. Ger. Pat. 302,291, Mar. 27, 1915.

CRUDE crepsin prepared in the ordinary way by precipitation with calcium salts is dried and



powdered, and then treated repeatedly with extracts of erepsin from the intestinal membrane. The final product is a stable white powder which yields erepsin to dilute hydrochloric acid; this solution has the power of effecting the rapid and complete hydrolysis of peptone in neutral or feebly alkaline solution.—D. F. T.

*Tropic acid; Preparation of* —. Chem. Werke Grenzach A.-G., Grenzach. Ger. Pat. 302,737, May 18, 1916.

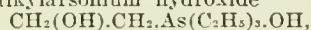
OPTICALLY inactive tropic acid, identical with the product obtained from atropine and suitable for the manufacture of pharmaceutical preparations, is prepared by hydrolysis of its ester, which is formed by the reduction of ethyl oxymethylenephénylacetate.—D. F. T.

*Proteins; Preparation of compounds of* — with the heavy metals. K. Kottmann, Berne, Switzerland. Ger. Pat. 302,911, June 9, 1916. Addition to Ger. Pat. 300,513.

PROTEIN substances from animal organs and tissues which have been freed from soluble degradation products so that they no longer give the ninhydrin reaction, are treated with compounds of the heavy metals or with the heavy metals in a colloidal form. The products are especially suited to therapeutic application and do not produce undesirable side-effects as do similar substances prepared from protein which has not been purified. The metal is present in them in the non-ionised condition, but can be liberated by the action of enzymes capable of hydrolysing the protein matter.—D. F. T.

*Ethanoltrialkylarsonium hydroxides and their salts; Preparation of* —. Chem. Werke Grenzach A.-G., Grenzach. Ger. Pat. 303,032, Jan. 25, 1916.

WHEREAS the hydrolysis of  $\omega$ -bromoethyltriethylarsonium bromide  $\text{CH}_2\text{Br} \cdot \text{CH}_2 \cdot \text{As}(\text{C}_2\text{H}_5)_3\text{Br}$  by silver oxide is known to cause concurrent elimination of hydrogen bromide with formation of vinyltriethylarsonium hydroxide, the same salt on treatment with water at higher temperatures yields ethanoltrialkylarsonium hydroxide



possessing valuable therapeutic properties.

—D. F. T.

*Ricinostearolic acid; Preparation of a crystalline ester of the di-iodide of* —. J. D. Riedel Akt.-Ges., Berlin-Britz. Ger. Pat. 303,052, Mar. 14, 1914.

THE di-iodide of ricinostearolic acid may be converted into its ethyl ester, or ethyl ricinostearolate may be caused to form the additive compound with iodine, the resulting product, as compared with the methyl ester, being unexpectedly crystalline.—D. F. T.

*Hexamethylenetetramine; Preparation of a derivative of* —. L. Egger, Buda Pesth. Ger. Pat. 303,450, Jan. 23, 1915. Under Int. Conv., Jan. 31, 1914.

HEXAMETHYLENETETRAMINE and a molecular proportion of acetylsalicylic acid are ground with a quantity of alcohol insufficient to dissolve the former by itself, until complete dissolution is effected, and the hexamethylene-tetramine acetylsalicylate is allowed to crystallise. The product shows a greater bactericidal power than the corresponding non-acetylated compound.—D. F. T.

*Camphor; Preparation of emulsions of* —. Saccharin-Fabrik A.-G. vorm. Fahlberg, Llist & Co., Magdeburg. Ger. Pat. 303,537, June 29, 1913.

By dissolving camphor in a small quantity of ether or methyl alcohol and treating the solution with water in the presence of lipoids such as blood alcohols, lecithin, or oxysterol, permanent emulsions can be obtained containing 5% of camphor, which are without harmful action on the blood and can therefore be injected intravenously.—D. F. T.

*Lithium acetylsalicylate; Preparation of compact, friable tablets of* —. J. A. Wülfing, Berlin. Ger. Pat. 304,574, Jan. 12, 1917.

THE tablets consist of a mixture of lithium acetylsalicylate with an alkali carbonate, particularly sodium bicarbonate. The addition of 5% of the alkali carbonate is sufficient to form tablets which do not harden and which even after keeping for a month remain readily soluble in water.—L. A. C.

[*Dicresyl carbonate.*] Substitute for lubricating oil and glycerin. Ger. Pat. 302,361. See IIA.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Development of the image in a dry plate negative; Mechanism of* —. F. W. T. Krohn. Phot. J., 1918, 58, 179–194.

A DESCRIPTION is given of the appearance of cross-sections of developed film over a very wide range of exposure. If a film is exposed until a visible image is formed, so that reversal would show on development, and is then fixed without development, a red image is left which has the appearance under the microscope of shells of light-reduced silver. It is suggested that these shells, probably along with surrounding films of gelatin halide, act as protective coverings for the silver halide grains and so retard development. The inertia of a plate is found to vary according to the development it receives, being a maximum at about  $\gamma = 0.8$ . In an example given the inertia was 0.22 at  $\gamma = 0.295$ , time of development =  $\frac{1}{3}$  min., 0.19 at  $\gamma = 0.8$ , time = 2 mins., and 0.36 at  $\gamma = 2.7$ , time = 12 mins.; taking  $\gamma = 0.8$  to  $\gamma = 1.4$  as the range of normally useful development factors, the variation of the inertia value from the value at  $\gamma = 1$  is on the average –13% on one side and +26% on the other. Constancy of inertia over a fairly wide range of  $\gamma$  values was found generally only with an easy tendency to fog. The author has observed that low densities develop to their maximum more quickly than high densities, and it is suggested that the change of inertia may be due to this. The variation of plate curves with strength of developer was also examined and a developer half the strength of that originally recommended by Hurter and Driffield (this J., 1890, 455; 1896, 100) was found to give a longer straight line portion in the curve and a lower inertia than either stronger or weaker developers. The addition of increasing quantities of bromide to the developer was found to give an increasingly lower rate of development, higher inertia, and somewhat longer range.—B. V. S.

[*Colour-*] sensitising [photographic plates]; A new method of —. E. König. Phot. Rundsch., 1917, 54, 257–258. Z. angew. Chem., 1918, 31, Ref., 102.

PLATES are not sensitised by bathing in a pure alcoholic dye-solution, but may be sensitised if the alcoholic dye-bath is followed by a water bath.—B. V. S.

*Colour-sensitive bathed photographic plates.*  
Lüppo-Cramer. Phot. Ind., 1917, 657. Z. angew.  
Chem., 1918, 31, Ref., 102.

COLOUR sensitising of photographic plates by bathing as compared with the introduction of dyes into the emulsion confers greater sensitiveness with, however, loss of keeping quality. The latter may be increased at the expense of the former by introducing some potassium bromide into the dye-bath.

—B. V. S.

#### PATENTS.

*Photographic pictures on silver mirrors; Process for the production of —.* J. Riedler, Berlin-Steglitz. Ger. Pat. 301,929, Sept. 12, 1916.

A SILVERED glass plate is coated with a light-sensitive film such as caoutchouc and asphalt, exposed under a line or half-tone negative, developed in acetone or the like, etched through to the glass with any suitable solvent of the silver film and then coated with a coloured backing as desired.

—B. V. S.

*Positives; Process for the photographic preparation of — from positives.* R. Kögel, Beuron. Ger. Pat. 302,786, Nov. 10, 1916.

THE printing film contains a light-sensitive diazo compound which can be developed by amines or phenols to a stable azo dye, but loses this property on exposure to light. Where the effect of exposure is not visible a print-out image may be obtained by the addition of a suitable bleach-out dye. If a suitable de-sensitiser or fixing bath be employed, this dye helps to strengthen the picture.

—B. V. S.

*[Palladium] toning of silver chloride emulsion papers; Process for —.* Kraft und Stendel Fabr. phot. Papiere, G.m.b.H., Dresden. Ger. Pat. 302,817, Oct. 3, 1916.

THE toning bath contains at least 0.3% of concentrated hydrochloric acid and 1% of alum, e.g., potassium chloro-palladite, 1 grm., hydrochloric acid 1 : 5, 15 c.c., alum, 15 grms., and water up to 1500 c.c. The hydrochloric acid prevents the formation of yellow stains in the gelatin film and the paper by precipitation of palladium hydroxide, and the formation of an opalescent fog especially in the shadows; the alum prevents softening of the gelatin, and, not being completely removed by washing, is effective also in the fixing bath. There must be at least 10 mins. washing between the toning bath and the fixing bath, which latter is a solution of ammonia, e.g., about 30 c.c. of concentrated ammonia per litre.—B. V. S.

*Development films free from stick-marks; Preparation of —.* Chem. Fabr. auf Aktien vorm. E. Schering. Ger. Pat. 303,144, Nov. 29, 1913. Addition to Ger. Pat. 295,502 (this J., 1917, 307).

To prevent stick-marks on development papers, etc., with baryta-coated supports, albumin is added to the baryta mixture.—B. V. S.

*Photographic prints in colours; Paper for producing —.* J. E. Thornton, London, Assignor to J. O. O'Brien, Manchester. U.S. Pat. 1,263,962, Apr. 23, 1918. Date of appl. May 21, 1915.

SEE Eng. Pat. 13,711 of 1914; this J., 1915, 818.

*Photometry and apparatus therefor.* Eng. Pat. 112,614. See XXIII.

#### XXIII.—ANALYSIS.

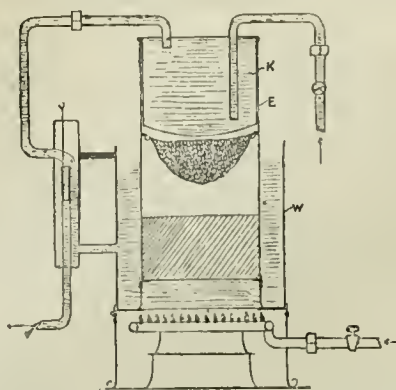
*Thermocouples; Standardisation of base-metal —.*  
P. D. Foote, T. R. Harrison, and C. O. Fairchild.  
Met. and Chem. Eng., 1918, 18, 403—408.

THE rapid method of calibrating base-metal couples by placing the hot junctions of the couples, together with the hot junction of a standard couple, close together in a suitable furnace and comparing the e.m.f.s. developed, though suitable for many industrial purposes, is liable to inaccuracy owing to the difficulty of obtaining an even distribution of temperature in the furnace. A more satisfactory method consists of immersing the hot junctions, suitably insulated, in a bath of molten tin, and comparing the readings at various temperatures. With this type of bath a range of 300° C. to 1000° C. may be obtained. The galvanometer resistance should be at least 300 ohms, otherwise change in the resistance of the circuit will have an appreciable effect upon the accuracy of the results. Should a low-resistance galvanometer have to be used, the resistance of the remainder of the circuit, i.e., the couple and connections, should be frequently checked and changes allowed for. In reading furnace temperatures it is essential that the depth of immersion of the couple should be sufficient to render negligible any error caused by the conduction of heat from the region of the hot junction by the protecting sheath of the couple. If the temperature gradient of the interior of the sheath is found to be small at the closed end this error may be neglected. It is essential that the cold junction temperature should be kept constant, which may be done by means of a water jacket or other device. Hot junctions may be made by fusing the ends of the two wires together, using borax as a flux. Afterwards the joint, whilst still hot, is dipped in water to remove borax.

—H. J. H.

*Extraction apparatus; Laboratory —.* C. G. Schwalbe and W. Schulz. Chem.-Zeit., 1918, 42, 194.

A MODIFICATION of the Besson extractor (Chem.-Zeit., 1915, 40, 860) in which no corks are employed and larger quantities (4–6 litres) of material may be extracted. The apparatus consists of a cylinder of tinfoil, E, in the upper part of which is fitted a wire ring which carries a fine cotton bag holding the material to be extracted. Above this bag is



fitted a cylindrical condenser, K, through which a stream of water flows. The apparatus is heated in a water-bath, W. All gas- and water-connections are made of metal so as to ensure the safety of the apparatus. (See also J. Chem. Soc., July, 1918.)

—A. B. S.



*Potassium; Light filters for the detection of —.*  
A. Herzog. Chem.-Zeit., 1918, 42, 145.

GLASS plates coated with gelatin coloured by certain dyes may be used in the flame test for potassium. The most suitable screen is one having a coating containing 3.9 grms. of Patent Blue and 2.5 grms. of Tartrazine per sq. metre of glass. Viewed through this screen the potassium flame appears bright red fringed with yellowish-green. The rubidium flame is like the potassium flame, but this is the only element which cannot be distinguished from potassium by means of the screen. Violet and green dyes may be used in making the screen, but the combination mentioned above will be found to be the most useful.—W. P. S.

*Gravimetric analysis; Time as a factor in —.*  
*Precipitation of barium sulphate.* Z. Karaglanow. Z. anal. Chem., 1918, 57, 77—98.

THE rate at which barium chloride solution is added has a considerable effect on the precipitation of barium sulphate; to obtain correct results the addition should be made gradually over a period of not less than 1.5 min. The precipitation is also affected by the concentration of the solution, but not by temperature. (See also J. Chem. Soc., July, 1918.)—W. P. S.

*Phosphoric acid; Precipitation of — in the form of ammonium phosphomolybdate.* J. Clarens. Bull. Soc. Chim., 1918, 33, 146—159.

A MORE detailed discussion of work already published. (Compare J. Chem. Soc., April, 1918.)—W. G.

*Phosphoric anhydride; Estimation of — as ammonium phosphomolybdate.* J. Clarens. Bull. Soc. Chim., 1918, 33, 159—163.

THE phosphoric anhydride is precipitated as ammonium phosphomolybdate. 100 c.c. of ammonium molybdate solution containing 15–20 grms. of ammonium nitrate, being used for a quantity of solution containing 0.1 gm.  $P_2O_5$ . The precipitate is washed with distilled water and the nitrogen in it liberated by sodium hypobromite and measured in a nitrometer. Under the above conditions the nitrogen and  $P_2O_5$  are present in the precipitate in the proportions requisite to form tri-ammonium phosphate. The method is not satisfactory for the estimation of phosphoric acid in the presence of sulphuric acid.—W. G.

*Silver nitrate; Iodine titration of — with palladous nitrate as indicator.* L. Scheider. J. Amer. Chem. Soc., 1918, 40, 583—593.

THE Volhard method for the volumetric estimation of silver is interfered with by the presence of certain metals and loses its sensitiveness for very dilute solutions. Silver solutions may, however, be titrated with iodide in the presence of such metals, and in very dilute solution, by the use of palladous nitrate as indicator. The indicator solution is prepared by dissolving 0.06% of palladous nitrate in 16% nitric acid; it is advisable to use a protective colloid along with the indicator to prevent the occlusion of potassium iodide or silver nitrate. For this purpose a 5% solution of gum arabic is most advantageous. The indicator is converted by the first excess of iodide into palladous iodide which gives the solution a reddish colour; this action is sensitive to 1 part in 500,000. The method is further to be recommended on account of the accuracy and easy reproducibility with both N/10 and N/1000 solutions. The author discusses at some length the possible errors of the method, and gives full details

of the procedure to be adopted for N/10 and N/1000 solutions.—J. F. S.

*Lead; Volumetric determination of — by ammonium molybdate.* Lindt. Z. anal. Chem., 1918, 57, 71—76.

AN excess of ammonium acetate should be avoided in dissolving the lead sulphate in this method (see Sacher, this J., 1910, 47). The ammonium acetate solution used may be prepared by adding 50% acetic acid to 25% (by weight) ammonia until the mixture is slightly acid to litmus paper. Correct results are obtained when just sufficient of this solution is employed to dissolve the lead sulphate; otherwise, the results are too high. (See also J. Chem. Soc., July, 1918.)—W. P. S.

*Thorium and zirconium; Colour reaction of — with pyrogallol-aldehyde.* H. Kaserer. Chem.-Zeit., 1918, 42, 170—171.

ON the addition of pyrogallol-aldehyde to solutions of thorium and zirconium compounds a yellow coloration is produced, and, on standing, or immediately on boiling or on addition of hydrogen peroxide, a dirty yellow precipitate and colourless solution. With cerous compounds a brownish-yellow colour is produced but no precipitate, even on boiling. On the addition of nitric, sulphuric, or hydrochloric acid the colour is discharged. The reagent is prepared by condensing a solution of 38 grms. of pyrogallol and 36.3 grms. of formamide in anhydrous ether with 15.2 grms. of phosphorus oxychloride and filtering after 12 hours. The residue is recrystallised from alcohol, to which sodium chloride is added, and the crystals are suspended in hot caustic soda through which a current of hydrogen is passed. The liquid is acidulated, the aldehyde extracted with ether, and purified by means of its bisulphite compound. (See also J. Chem. Soc., July, 1918.)—A. B. S.

*Distinction between different abrasives and detection of carborundum in them.* Peters. See VIII.

*Chemical and mechanical-technical examination of rubber.* Fol. See XIV.

*Estimation of the content of unsaponifiable resins in various kinds of rubber.* Dekker. See XIV.

*Estimation of resin and impurity in balata and gutta-percha.* Dekker. See XIV.

*Detection of tannins by the formaldehyde precipitation method.* Lauffmann. See XV.

*Critical examination of tanning materials, extracts, chrome liquors, and leather.* Lauffmann. See XV.

*Estimation of chromium in chrome salts, liquors, leather ashes, and residues.* Schorlemmer. See XV.

*Analysis of vegetable sole leather.* See XV.

*Sodium sulphate in leather.* Mann. See XV.

*Extractor for water-soluble matter in leather.* Balderston and Alsop. See XV.

*Determination of fat in leather.* Willson and Kern. See XV.

[*Determination of*] *moisture in leather.* Wilkinson.  
See XV.

*Determination of lactic anhydride in lactic acid.*  
Thompson and Suzuki. See XV.

*Estimation of iron in lactic acid.* Harvey. See XV.

*Neutral and acid polarisation of rhuasses from  
beet molasses.* Pellet. See XVII.

*Determination of furfuroids (furfurosans) in pro-  
ducts of beet sugar factories.* Gillet. See XVII.

*Influence of paraffin on the Gerber method for the  
determination of fat in milk.* Reiss. See XIXA.

*Determination of fatty acids in butter fat.* Holland  
and Buckley. See XIXA.

*Estimation of salicylic acid in foods.* Steenberg.  
See XIXA.

*Method of distinguishing treated from raw straw  
and determination of digestible fibre.* Mach and  
Lederle. See XIXA.

*Water analysis in the field.* Heise and Behrman.  
See XIXB.

*Field methods for determination of total hardness  
in water.* Behrman. See XIXB.

*Estimation of injurious dust in mine air by the  
Kotzé konimeter.* Innes. See XIXa.

#### PATENT.

*Photometry and apparatus therefor.* Siemens und  
Halske Akt.-Ges., Siemensstadt, Germany. Eng.  
Pat. 112,614, Sept. 29, 1917. (Appl. No. 14,096  
of 1917.) Under Int. Conv., Jan. 12, 1917.

The apparatus is designed to allow alteration of colour of the standard light source without any alteration in its brilliancy, so that the determination of the luminosity of coloured light sources may be easily carried out. In the more general form the standard light is enclosed in a box with a rectangular opening containing three adjacent colour screens, red, green, and blue, of equal size and luminosity; in front of this opening are two adjustable shutters, each equal in width to that of one colour-screen, so that two thirds of the light passed by the composite screen is cut off, but the light passed may contain any desired proportions of the three colours. In another form the three colour screens are arranged on the sides of a triangle, and a single shutter of special shape and adjustable in two directions is used. For some special purposes, especially where a number of lights differing only slightly in colour are to be compared, a simpler arrangement may be used employing only two colour-filters and one shutter; also the opening in the box may be only partly filled by the colour-filters, a certain amount of unfiltered light being transmitted. The light passes from the opening along a tube to a diffusing screen.—B. V. S.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Bellwood and Downs. 9639. See XII.

Brownlow. Centrifugal separators. 9555. June 11.

Burns, Bush, and Huntington & Heberlein. Means for separating suspended particles from gaseous bodies. 9195. June 4.

Elektrochem. Werke. Furnace for carrying out endothermic gas reactions. 9301. June 5. (Ger., Mar. 23, 1917.)

Green, and Levinstein, Ltd. Separating constituents of mixtures of volatile liquids. 9689. June 12.

Green, Levinstein, and Levinstein, Ltd. Preparation of a catalyst. 9764. June 13.

Green, Levinstein, and Levinstein, Ltd. Apparatus for carrying out catalytic reactions. 9765. June 13.

Harries. Separation of matter in suspension from liquids. 9095. June 3.

Hoffmann. Induction and distribution of liquids and gases. 9319. June 6.

King and Rawson. Calcination. 9748. June 13.

Malcolm, and Salt Union. Evaporating or drying apparatus. 9298. June 5.

Marks (Driver-Harris Co.). Melting-pots or crucibles. 9210. June 4.

Matousek. Centrifugal separators. 9444. June 8.

Morton. Evaporator coil. 9228. June 4. (New Zealand, Dec. 21, 1917.)

Richards. Filters. 9760. June 13. (U.S., June 4, 1917.)

Roudy, and Tréfilerles et Laminiers du Havre. Furnaces. 9534. June 10. (Fr., Sept. 29, 1917.)

Sinclair and Thorold. Apparatus for dissolving chemicals. 9209. June 4.

Techno-Chemical Laboratories, Ltd., and Testrup. Conducting high-temperature reactions. 9655. June 12.

Wilson. Filter-presses. 9160. June 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

7939 (1917). Roy, and Morson & Son. Means for carrying out catalytic or contact reactions between gases and/or vapours. (116,151.) June 12.

16,268 (1917). Wehner. See XI.

588 (1918). Rowley and Merton. Roasting furnaces. (116,235.) June 12.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Aktiebolaget Ingenjörssfirma Egnell. 9509. See XXIII.

Alexander and others. 9641. See XII.

Anderson. 9888. See VII.

Atkinson, Clark, and Powdered Fuel Plant Co. Preparation or treatment of fuel. 9335. June 5.

Dinning and Richards. Gas-generators. 9153. June 3.

Dunstan. Refining liquid hydrocarbon. 9114. June 3.



Francart. Gas generators or producers. 9473. June 8.  
 Harger. Consuming smoke. 9180. June 4.  
 Harger and Helps. Manufacturing fuel gases. 9186. June 4.  
 Kean. Electro-dynamical devices for purifying waste lubricating oil. 9620. June 12.  
 Pease. Manufacture of gas. 9353. June 6.  
 Wake. Treatment of anthracite. 9687. June 12.  
 Yeadon. Vertical retort. 9623. June 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3192 (1917). Dutt and Dutt. Synthetic production of methane. (116,302.) June 19.  
 3368 (1917). Nelson. Process and apparatus for cracking hydrocarbon oils and the like. (116,304.) June 19.  
 5290 (1917). Marks. *See XII.*  
 7079 (1917). Jenkins. Process and apparatus for cracking heavy oils. (116,119.) June 12.  
 7674 (1917). Spencer. Washer scrubbers for extracting ammonia, naphtha, etc., from coal gas. (116,319.) June 19.  
 10,368 (1917). Peel and Shevels. Coke ovens. (116,293.) June 19.  
 10,544 (1917). Bartlett. Gas producers, cupola furnaces, etc. (116,185.) June 12.  
 11,238 (1917). Rose. Manufacture of gaseous fuel. (116,406.) June 19.  
 12,672 (1917). Fairweather (Dunham). *See XX.*  
 16,268 (1917). Welner. *See XI.*  
 16,951 (1917). Walker and Bates. Vertical retorts for distilling, carbonising, or roasting coal, etc. (116,458.) June 19.  
 6925 (1918). Edmonds. Compounds for treating fuel. (116,252.) June 12.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Abbey and Calvert. Manufacture of nitrophenols. 9161. June 4.  
 Abbey and Calvert. Production of amidobenzene-disulphonic acid. 9164. June 4.  
 Dunstan. 9114. *See II.*  
 Morris, and Morris & Co. Sulphonation, nitration, and chlorination of organic substances. 9256. June 5.  
 Morris, and Morris & Co. Manufacture of salts of amido and other salt-forming organic substances. 9257. June 5.  
 Morris and others. 9258. *See XXII.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

205 (1917). Oman. *See V.*  
 5290 (1917). Marks. *See XII.*  
 7674 (1917). Spencer. *See II.*

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATION.

Vegas and Vegas. Colouring-product and tinctorial mordant. 9823. June 14.

#### COMPLETE SPECIFICATION ACCEPTED.

8303 (1917). Imperial Trust for the Encouragement of Scientific and Industrial Research, and Hepburn. Production of insoluble azo colours on the fibre. (116,360.) June 19.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

British Cellulose and Chemical Manufacturing Co., and Cumbers. Covering and producing tautness of aeroplane wings, etc. 9488. June 10.

Dunlop Rubber Co., and Twiss. Treatment of aluminium-coated fabrics for air and gas containers. 9617. June 12.

Dydynski (Gaszynski and Oshourkoff). Apparatus for treating fibrous material. 9126. June 3.

Goulding and Hickman. Treatment of fabric for use in preserving fruit, vegetables, etc. 9092. June 3.

Miyake. Raw material for woven fabric. 9422. June 7.

Napier & Son, and Tyndall. Doping, etc., fabrics for aircraft, etc. 9276. June 5.

Nobel's Explosives Co., and others. 9849. *See XXII.*

Partington and Robertson. Fibre substance for paper making, etc. 9091. June 3.

Wade (Barrett Co.). Manufacture of felt, paper, etc. 9693. June 12.

Zdanowich. Nitrocellulose solutions. 9559 and 9560. June 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

205 (1917). Oman. Preparation of nitroso compounds from lignon sulphonie acid or its salts. (103,654.) June 12.

6793 (1917). Oman. Treatment of sulphite cellulose waste lye. (103,493.) June 12.

8418 (1917). Cross, and Viscose Development Co. Manufacture of coverings or masses of structureless cellulose. (116,366.) June 19.

12,744 (1917). Strain. Rippling or boiling of flax. (116,421.) June 19.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

McCardell and Moseley. Bleaching. 9385. June 7.

Newton and Woodhouse. Treating textile fabrics. 9636. June 12.

Vegas and Vegas. 9823. *See IV.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

7522 (1917). Wrigley and others. *See XV.*

7603 and 17,253 (1917). Walker. Apparatus for scouring, dyeing, or otherwise treating with liquor, wool, yarn, etc. (116,129.) June 12.

8303 (1917). Imperial Trust. *See IV.*

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Anderson. Recovery of cyanogen compounds and/or sulphur from spent oxide. 9888. June 15.

Bailey, Denny, and Jefferis. Separation of potassium nitrate from sodium nitrate. 9283. June 5.

Dehn (Henshaw). Obtaining alkaline-earth fluorides and soluble potassium salts. 9672. June 12.

Linder. Manufacture of sulphate of ammonia. 9128, 9129, and 9130. June 3.

Metals Extraction Corporation, Peterson, and Williams. Purification of zinc solutions. 9692. June 12.

Wyd. Preparation of dry gaseous ammonia. 9094. June 3.

#### COMPLETE SPECIFICATIONS ACCEPTED.

6602 (1917). Hone. Decomposing siliceous-bearing earths, rocks, minerals, and compounds. (116,118.) June 12.

7674 (1917). Spencer. *See II.*

7888 (1917). Fujiyama. Manufacture of ammonium sulphate. (116,321.) June 19.

7715 (1917). Halvorsen. Recovery of soluble compounds of potassium and aluminium from silicates. (107,012.) June 12.

8401 (1917). Calvert. Manufacture of cyanide. (116,365.) June 19.

9803 (1917). Hyatt and Fellowes. Utilisation of nitre cake. (116,181.) June 12.

14,968 (1917). Dutt and Dutt. Manufacture of potassium chloride. (116,438.) June 19.

2019 (1918). Goldschmidt. Production of aluminium compounds. (113,276.) June 19.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Brownlow. Manufacture of pottery by casting from slip. 9630. June 12.

Wade (Carborundum Co.). Manufacture of refractory articles. 9297. June 5.

##### COMPLETE SPECIFICATIONS ACCEPTED.

8319 (1917). Wurga. Production of articles channelled with vitreous enamel. (116,361.) June 19.

2439 (1918). Peeters. Glass furnaces. (116,477.) June 19.

#### X.—METALS, METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Ashcroft. Manufacture of magnesium or its alloys. 9644 and 9645. June 12.

British Thomson-Houston Co. (General Electric Co.). Production of steel alloys, and constituents thereof. 9267. June 5.

British Thomson-Houston Co. (General Electric Co.). Manufacture of steel. 9268. June 5.

Davidson and Mannel. Process for case-hardening steel and tempering. 9543. June 12.

Hadfield. Remelting manganese steel scrap. 9254. June 6.

Hadfield. Manufacture of iron-silicon alloys. 9577. June 11.

Hazard and Morris. Electrodes for electric smelting, etc. 9226. June 4.

Henderson. Recovery of tin from tinned metals. 9424. June 7.

Marks (Driver-Harris Co.). 9210. *See* I.

Morgan Crucible Co., and Speirs. 9469. *See* XI.

Naish. Aluminium alloys. 9413. June 7.

Wake. Treatment of iron ore. 9686. June 12.

##### COMPLETE SPECIFICATIONS ACCEPTED.

5177 (1917). Jones. Electrically fusing and depositing metal. (116,308.) June 19.

6602 (1917). Hone. *See* VII.

7742 (1917). Holloway. Refining nickel-copper matte. (116,139.) June 12.

8234 (1917). Pozzo and Colonnetti. Testing of iron materials. (116,162.) June 12.

10,541 (1917). Bartlett. *See* II.

588 (1918). Rowley and Merton. *See* I.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

Breeden and Lucas. Electric accumulators. 9898. June 15.

Elektrochem Werke. 9301. *See* I.

Forster. Electric furnace. 9901. June 15.

Hazard and Morris. 9226. *See* X.

Kilburn (Norske Aktieselskab for Elektrokemisk Industri). Manufacture of carbon electrodes. 9285. June 5.

Morgan Crucible Co., and Speirs. Electrically-heated crucibles, melting-pots, etc. 9469. June 8.

Morgan Crucible Co., and Speirs. Crucibles for electric furnaces. 9822. June 14.

Pitt. Electric furnaces. 9249. June 5.

Rogers and Walter. Electrolytic apparatus. 9622. June 12.

##### COMPLETE SPECIFICATIONS ACCEPTED.

5177 (1917). Jones. *See* X.

16,268 (1917). Wehner. Ovens or furnaces for use in baking carbon electrodes and for other purposes. (116,455.) June 19.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Alexander, Imbery, and Vint. Heating oils, fats, etc., by electricity for distillation, etc. 9641. June 12.

Bellwood and Downs. Evaporating or recovering solvent from solution containing oil, fat, wax, etc. 9639. June 12.

Chapman. Drying-oil. 9526. June 10.

Christensen (Christoffersen). Producing emulsion. 9601. June 11.

Orth. Treating soya bean to recover oil and produce synthetic milk and flour. 9471. June 8.

Huxley. Castor oil. 9795. June 14.

Imray (Boyd). Making cakes of soap. 9470. June 8.

Levinstein, Robertshaw, and Levinstein, Ltd. Production of compounds of oils, fats, greases, waxes, fatty acids, etc. 9448. June 8.

Martin. Deodorising and decolorising cocoa-butter. 9732. June 13.

Soc. Franç. des Glycerines. Distilling glycerin. 9294 and 9432. June 5 and 7. (Fr., Feb. 14 and Apr. 16.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

5290 (1917). Marks (Soc. Franco-Belge de Fours à Coke). Apparatus for distilling oils, fats, etc. (116,369.) June 19.

8179 (1917). Reed, and Eastern Flour Co. Treatment of soya beans. (116,158.) June 12.

9591 (1917). Dehn (National Sanitary Service Co.). Apparatus and method for recovering fats. (116,384.) June 19.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

##### APPLICATIONS.

Chapman. 9526. *See* XII.

Nederlandsche Maatschappij. *See* XIV.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### APPLICATIONS.

Dagnall and Sumner. Mineral rubber composition. 9705. June 13.

La Rosée. Manufacture of caoutchouc. 9613. June 11. (Ger., Feb. 14.)

N. V. Nederlandsche Maatschappij tot Exploitatie van Optimietfabrieken. Manufacture of a substitute for ebonite, bakelite, etc. 9746. June 13. (Holland, Aug. 14, 1917.)

##### COMPLETE SPECIFICATIONS ACCEPTED.

7885 (1917). Jackson (General Rubber Co.). Treatment of unvulcanised rubber, rubber-like and similar vulcanisable material. (116,322.) June 19.



7887 (1917). Jackson (General Rubber Co.). Rubber coagulum, etc., and preparation of the same for vulcanisation. (116,323.) June 19.

7899 (1917). Jackson (General Rubber Co.). Drying coagulated and uncoagulated latex. (116,324.) June 19.

7905 (1917). Jackson (General Rubber Co.). Rubber coagula or similar materials, and preparation of the same from latex for vulcanisation. (116,326.) June 19.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### APPLICATION.

Tada. Preparation of gelatin. 9144. June 3.

##### COMPLETE SPECIFICATIONS ACCEPTED.

7522 (1917). Wrigley, Spence, and Spence and Sons. Dyeing or staining and mordanting leather. (116,123.) June 12.

7979 (1917). Groth. Treatment of hides and skins prior to and during tannage. (116,334.) June 19.

148 (1918). Heyl. Drying japanned or patent leather. (112,460.) June 19.

#### XVI.—SOILS; FERTILISERS.

##### APPLICATION.

Allibon and Goulding. Apparatus for manufacture of superphosphates or like artificial fertilisers. 9855. June 15.

#### XVIII.—FERMENTATION INDUSTRIES.

##### APPLICATIONS.

Anderschou. Treatment of waste brewers' yeast to render it suitable for baking. 9449. June 8.

Spiers and Weizmann. Fermentations. 9133. June 3.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

Abrahamsen. Antiseptics, disinfectants, etc. 9207. June 4. (Norway, June 28, 1917.)

Anderschou. 9449. *See* XVIII.

Barnabé. Preserving organic substances. 9614. June 11.

Calvert. Manufacture of vegetable extracts. 9230. June 4.

Cotterill and Croft. Manufacture of vegetable butter. 9369. June 7.

Crossley. Treatment of powdered and other dried eggs to render them emulsifiable. 9736. June 13.

Firth. 9471. *See* XII.

Goulding and Hickman. 9092. *See* V.

Grousseau and Vicoigne. Treatment of cocoa and waste products of chocolate manufacture. 9224. June 4. (Fr., Apr. 10.)

Johnston. Apparatus for drying vegetables, fruit, fish, meat, etc. 9859. June 15.

Linden. Treatment of sewage, etc. 9744. June 13.

Martin. 9732. *See* XII.

Morton. Apparatus for drying vegetables, etc. 9429. June 7.

Palmer. Manufacture of potato flake, flour, and meal. 9489. June 10.

Swithenbank and Tailby. Insecticides, sheep dips, vermifuges, cattle dressings, etc. 9420. June 7.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Bainbridge and Weizmann. Production of glycols. 9131. June 3.

Boake, Roberts and Co., and Durrans. Preparation of organic acid anhydrides and chlorides. 9119. June 3.

Buratti. Combinations of chlorine with hexamethylenetetramine and manufacture of derived salts. 9702. June 13. (Ital., June 11, 1917.)

Harding. Manufacture of chlorinated hydrocarbons. 9603. June 11.

Lapworth, and Levinstein, Ltd. Manufacture of N-monoalkyl derivatives of aromatic compounds. 9710. June 13.

Matthews. Chlorination of acetic acid. 9574. June 11.

Matthews. Bromination of acetic acid. 9575. June 11.

Morris, and Morris and Co. 9256 and 9257. *See* III.

Nobel's Explosives Co., Rintoul, and Thomas. Preparation of amines. 9894 and 9895. June 15.

Pickard, Rankin, and Pickard, Coe, and Rankin. Aspirin tablets. 9684. June 12.

Spiers and Weizmann. Production of butyric acid. 9132. June 3.

##### COMPLETE SPECIFICATIONS ACCEPTED.

3192 (1917). Dutt and Dutt. *See* II.

8152 (1917). Weston Chemical Co., and Savage. Manufacture of benzoic acid. (116,348.) June 19.

12,672 (1917). Fairweather (Dunham). Making paraffinum liquidum. (116,418.) June 19.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

##### APPLICATIONS.

Hess-Ives Corporation. Colour photograph or film, and method of producing same. 9436. June 7. (U.S., Oct. 9, 1917.)

Middleton. Pigment or carbon printing by silver. 9293. June 5.

#### XXII.—EXPLOSIVES; MATCHES.

##### APPLICATIONS.

Gardner. Explosives. 9134. June 3.

Marin. Preparation of combustible salt for making explosives. 9753. June 13. (Belg., June 13, 1917.)

Nobel's Explosives Co., Nolan, and Rintoul. Gelatinisation of cellulose esters, and explosives and materials therefrom. 9849. June 14.

Tisell. Explosives. 9137 and 9832. June 3 and 14.

Zdanowich. 9559 and 9560. *See* V.

#### XXIII.—ANALYSIS.

##### APPLICATIONS.

Aktiebolaget Ingeniörsfirma F. Egnell. Gas-analysing apparatus. 9509. June 10. (Ger., June 9, 1917.)

Rideal and Taylor. Indicating and/or recording quantities of carbon monoxide in hydrogen, etc. 9205. June 4.

##### COMPLETE SPECIFICATION ACCEPTED.

8234 (1917). Pozzo and Colonnetti. *See* X.

## I.—GENERAL ; PLANT ; MACHINERY.

## PATENTS.

*Drying apparatus.* S. H. Meihulzen, Veendam, Holland. Eng. Pat. 114,620, Feb. 20, 1918. (Appl. No. 3035 of 1918.) Under Int. Conv., Mar. 31, 1917.

The material to be dried is placed in small cups which are arranged one above the other on platforms of slightly greater diameter than the cups. Baffle-plates perforated in the centre are arranged over the cups. The whole framework may be lowered into a vertical heating tube closed at the bottom, which is surrounded by a larger concentric tube containing a heating liquid. Air previously dried by passing it through sulphuric acid is supplied to the bottom of the inner tube, and is made to pass over the material in the cups by the baffles. A suitable heating burner is placed below the double tube and a third tube surrounds the heating tube for a portion of its height to maintain the hot combustion gases in contact with the walls of the heating tube. The two inner tubes are extended vertically for some distance above the material to be dried, the upper annular space being filled with suitable material to facilitate condensation of the vapour and its return to the heater. The top of the annular chamber may be connected to a vertical tube dipping into mercury to any desired depth, so that the pressure at which the liquid boils may be regulated. A large air vessel is connected to the system to receive air which is displaced by the vapour generated, and a mercury manometer is also provided. The apparatus is intended for drying amylum or similar materials, for the purpose of determining their moisture content for example.—W. F. F.

*Drying installations.* J. Schlatter and R. Frorath, Zurich. Eng. Pat. 115,193, Nov. 5, 1917. (Appl. No. 16,135 of 1917.)

In a drying apparatus for fruit, vegetables, cosmetics, chemical products, dyes, etc., hot gases and cold air are passed into the passages, 6 and

7 respectively, shown in cross-section in the figure, and a number of drying chambers, of which one, X, is shown, are arranged on both sides of the passages. The partition, 8, separating the passages, 6 and 7, terminates opposite a mixing chamber, 10, opening into the drying chamber, and the gases are finally discharged from the drying chamber into the conduit, 13. A balanced valve plate, 16, is arranged so that hot gas alone may be supplied by the openings, 20, 21. On lowering the plate, the opening, 20, may be cut off, and mixed hot gas and cold air supplied to the chamber, 10, in any desired proportions, or the hot gas may be completely cut off and cold air only supplied from the passage, 7. The

hot gas is obtained from a furnace through which air and combustion gases are drawn in regulable quantities by a fan. The furnace is surrounded by a cylindrical casing through which air can be drawn to mix with the hot

gases, and the mixture is discharged through a spiral conduit by a fan placed eccentrically in the casing. The passages, 6 and 7, may alternatively be supplied by a single blower, and a furnace may be arranged in the conduit leading to the passage, 6.—W. F. F.

*Drying drum; Internally heated* —. P. Frank, Charlottenburg, A. Frank, Berlin-Halensee, and M. Lebram, Frankfurt on the Oder. Ger. Pat. 302,708, May 30, 1915.

The heating element consists of a drum through which hot gases flow. Inside the drum is fixed concentrically a second cylinder which causes the gases to flow over the inner surface of the outer one. The inner cylinder is filled with material capable of acting as a heat-accumulator and thus more uniform heating is attained.—H. J. 11.

*High temperature reactions; Methods of and furnaces or apparatus for conducting* —. N. Testrup, and Techno-Chemical Laboratories, London. Eng. Pat. 115,087, Apr. 25, 1917. (Appl. No. 5829 of 1917.)

In apparatus for conducting high temperature reactions continuously in which the material is passed in a continuous layer in front of the source of heat, the material is fed on to a travelling conveyor in a greater depth than that subsequently treated, so that when the conveyor travels below and close to the heating device, a recessed path is swept out in the material, leaving a deeper layer outside the walls of the heating device to seal the heated zone against cooler air. The heating apparatus consists of a horizontal chamber of refractory material, heated internally by gas burners, and having its floor composed of a slab of magnesia or the like to transmit heat to the material below. The side walls extend slightly below the magnesia slab into the material, leaving a narrow space between the slab and the material. After passing the heater the layer of material is given a downward inclination, and a cover plate attached to the heater extends parallel to and just above the layer. The clearance space is thus filled with highly heated gas which prevents the passage of cooler gas to the heating zone. An adjustable dividing plate is provided just beyond the cover plate to remove the upper treated layer from the untreated material, and a toothed roller is arranged just above the dividing plate to break up the material. The treated substance falls into a trough, from which it is removed by a screw conveyor, and the untreated substance falls into another trough from which it is transferred by a conveyor to a receptacle and then fed again to the travelling conveyor for re-heating. To prevent any reaction between the furnace walls and the hot material, the magnesia slab is made thicker at the edges so that it transmits less heat to the material at the part in contact with the walls.—W. F. F.

*Generator for mixed steam and combustion products.* A. Cornet and L. Humblet, Quaregnon, Belgium. Eng. Pat. 115,211, Jan. 8, 1918. (Appl. No. 472 of 1918.)

A hollow cylindrical body is provided with a curved extension at one end, and a mixture of liquid or gaseous hydrocarbon and air under pressure is fed to an axial burner at the other end. The flame strikes the curved walls and produces an intensely heated zone, and water is injected into this zone from a number of nozzles arranged round the cylindrical body. The hydrocarbon fuel is fed to the burner through a jacket surrounding it whereby



the fuel is heated or vaporised, and the water is passed to the nozzles through a jacket surrounding the combustion chamber. Any water which is not vaporised on leaving the nozzle flows through the curved extension of the combustion chamber, and is vaporised by the hot gases passing through this extension. A mixture of steam and combustion products of low temperature and high pressure, suitable for operating a steam engine or turbine, is obtained.—W. F. F.

*Oil-gas and other gas furnaces.* Burdons, Ltd., W. M. and M. M. Burdon, Bellshill, Lanark. Eng. Pat. 115,214, Jan. 17, 1918. (Appl. No. 966 of 1918.)

IN a furnace heated by oil-gas or other gas under pressure, two perforated pipes are provided at the base of the furnace door from which cold air blasts are emitted to cool the escaping hot gases when the door is opened. A damper is also provided at the top of the furnace, which is connected by levers with the door-lifting mechanism, so that both are lifted simultaneously, thus reducing the gas pressure within the furnace. The valves for controlling the air blast are connected by suitable mechanism with the operating gear of the damper, so that both are operated simultaneously when the furnace door is opened.—W. F. F.

*Crushing, granulating, or reducing to powder, stone, ore, and like materials; Machinery or apparatus for —.* W. H. Baxter, Ltd., and W. H. Baxter, Leeds. Eng. Pat. 115,218, Feb. 7, 1918. (Appl. No. 2223 of 1918.)

A BED or frame is provided with a pair of fixed bearings supporting a crushing roller with concave periphery, and with a pair of bearings movable horizontally in slideways supporting the co-operating roller which has a convex periphery. Each of the movable bearings carries a horizontal rod which passes through a fixed vertical part of the bedplate. A helical spring supported between the vertical member and a collar on the rod presses the movable bearing towards the corresponding fixed bearing, whilst two weaker helical springs supported between the opposite side of the vertical member and a bridge-piece on the rod similarly press in the opposite direction to the first spring. A more uniform pressure of the rolls is thus obtained. The tension of the springs may be varied by adjusting the position of the abutments carried by the rod.—W. F. F.

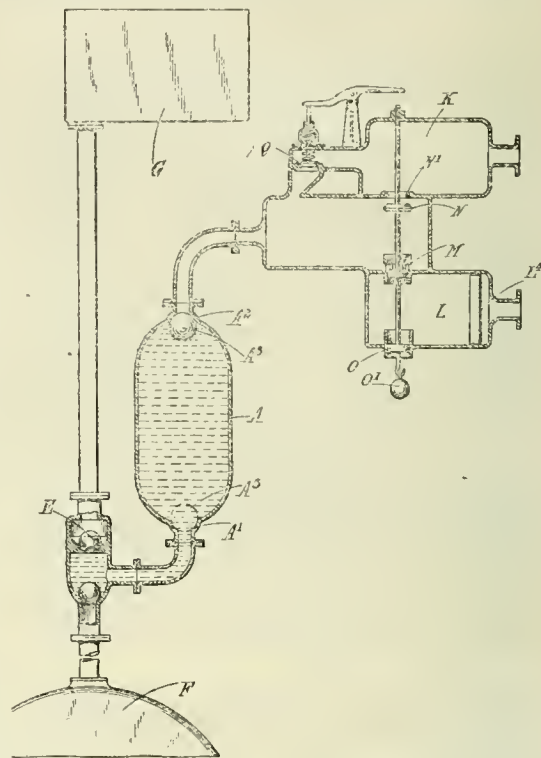
*Roller mill.* T. Robinson & Son, Ltd., and C. J. Robinson, Rochdale, and J. W. Collins, Roker, Durham. Eng. Pat. 115,689, May 15, 1917. (Appl. No. 6975 of 1917.)

IN a roller mill having rolls with spiral grooves for grinding grain or granular material, the grooves are spaced apart so as to leave a smooth part of width equal to that of a groove. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 1891 of 1882.)—W. F. F.

*Pumping liquids; Apparatus for —.* A. H. Human, Gerrards Cross, Bucks, and Kestner Evaporator and Engineering Co., Ltd., London. Eng. Pat. 115,254. (Appl. Nos. 4538, Mar. 29, and 6096, Apr. 30, 1917.)

IN a pulsometer pump for raising corrosive liquids, the chamber, K, is connected to a reservoir of low-pressure air or a vacuum pump, and with the loaded valve, N, open, liquid is raised from the container, F, to the vessel, A, till the float valve, A<sup>2</sup>, reaches its seating, A<sup>3</sup>. The pressure in K then

falls and the external air pressure acting on the piston, O, causes the valve, N, to close the opening, N<sup>1</sup>, the piston valve, M, being simultaneously opened. High-pressure air entering the chamber, L, through a contracted opening in L<sup>1</sup> then acts on the liquid in the vessel, A, and forces it past the valve, E, into the reservoir, G. When the valve, A<sup>2</sup>, reaches its seating, A<sup>1</sup>, the air pressure above



it increases till the valve, Q, is lifted and the resulting air pressure in K opens the valve, N, when the cycle is repeated. In a modification, the balance piston, O, may be replaced by a valve opening outwards. In another modification, the valves, M and N, are combined into a single two-way valve which is operated at the proper intervals by a piston working in a cylinder and actuated by the high-pressure air. The admission and discharge of air are regulated by a relay device consisting of two connected piston valves working in a casing, one of them being subjected to the pressure in the chamber, A. The movements of the piston valves are controlled by a suitable wire-drawing or a dash-pot device. The valves periodically place one or other side of the piston in communication with the high-pressure air, and the other side with the exhaust.—W. F. F.

*Power system and pump.* J. Wilkinson, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,263,981, Apr. 23, 1918. Date of appl., Jan. 14, 1914.

IN an internal combustion pump of the type in which the exploded charge acts directly on an oscillating column of liquid, the usual openings for the admission of fuel, air, and combustion gases are provided, and also for the admission and discharge of liquid. An air pressure accumulator is provided at the delivery end of the moving column to diminish pulsations in the flow. The admission and discharge valves are operated by a constant-speed electric motor, the circuit of which is opened

and closed by means controlled by the pressure of the air in the accumulator, and thus responsive to the demand on the pump. An auxiliary electrical device is also provided, acting on the valve-controlling device, by which it is ensured that the pump is stopped only at a definite predetermined point in its cycle of operations.—W. F. F.

*Gaseous products of chemical reactions; Purifying and absorbing* — C. S. Roy, Chingford, and T. Morson & Son, Ltd., London. Eng. Pat. 115,311, June 2, 1917. (Appl. No. 7940 of 1917.)

The gases flow upwards through a tower in which they are washed and purified, and then pass from the upper end of the tower to an absorber having an upper and a lower series of baffle plates arranged alternately. The upper plates are inclined to the vertical and the lower ones are preferably vertical. Solvent liquor from a compartment in the upper part of the absorber flows down over the upper plates as a film, collects in the lower part of the absorber to a level below the lower ends of the upper plates, and is thence returned to the compartment in the upper part of the absorber. The lower part of the tower is also connected with the absorber by a passage provided with a gate-valve, so that when desired the gases may pass directly to the absorber without ascending the tower.—J. H. P.

*Separating water and other matter from compressed air and other gases; Apparatus for* — C. E. Jeffcock and W. H. Yardley, Sheffield. Eng. Pat. 115,325, June 22, 1917. (Appl. No. 9015 of 1917.)

A CASING of decreasing cross-section from its inlet to its outlet end is divided into compartments by transverse partitions. Each partition has a central opening, and through these openings extends a metal strip twisted in spiral form to guide and impart a rotary motion to the air passing through the casing. The air inlet is of small sectional area compared with the sectional area of the adjacent part of the casing. Liquid separated from the air passes through holes in the lower part of the partitions and flows to a discharge opening.—J. H. P.

*[Fluid] meter; Calorimetric* — A. Guillemin, and Comp. des Forges d'Audincourt et Dependances, Audincourt, France. Eng. Pat. 115,354, Aug. 23, 1917. (Appl. No. 12,102 of 1917.) Under Int. Conv., June 30, 1917.

In apparatus for the measurement of hot fluids, particularly hot water or steam, the fluid passes upwards through a tube fixed at the bottom but free to expand at the top. The upper end carries a projection which bears against a substantially horizontal lever pivoted at its centre, the projection being slightly on one side of the centre. The tube opens at the top into a flat horizontal chamber having two openings in its upper face controlled by slotted piston-valves operated by the two ends of the pivoted lever, so that when one is open the other is closed. The valve-operating device is so constructed that the sum of the two openings remains constant. The valves are fully open at 100° C. and 0° C. respectively. Each valve controls the admission into a conduit containing a volume meter, and the two conduits are combined into a single conduit beyond the meters. The total volume of flow is obtained by adding the readings of the two meters. If  $V_1$  and  $V_2$  are the readings of the high and low temperature meters in c.c., then the mean temperature over a period is given by  $\frac{100 V_1}{V_1 + V_2}$  in the case of water and by  $100 + \frac{100 V_1}{V_1 + V_2}$  for steam between 100° and 200° C. The number of

calories passing through is given by the product of the total quantity of water passing and the mean temperature, or more simply by 100V. The instantaneous temperature reading may be obtained by using the proportional expansion of the tube to operate an indicator through a toothed rack at one end of the lever. The mean rate of flow is calculated from the total volume and the time.

—W. F. F.

*Wet fillers, water coolers, humidifying apparatus and the like.* A. B. Cleworth, Hollnwood, Lancs. Eng. Pat. 115,521, June 8, 1917. (Appl. No. 8219 of 1917.)

In apparatus for heating, cooling, moistening, cleaning, or otherwise treating air or gas with liquid, or for cooling water, oil, or other liquid with air, or for condensing vapour, of the type in which the gas is passed over rotating discs having their lower portions immersed in liquid, the discs are formed of expanded metal and are arranged in groups on the same or on different shafts which may rotate at the same or different speeds. The discs may be made in sections if desired. The discs are enclosed in a cylindrical casing, and the liquid for each group of discs may be in a separate compartment. Baffles may be provided so that the air or gas which is driven axially through the casing is made to pass through and not around the discs.—W. F. F.

*Air and gas; Apparatus for treating and cooling* — Heenan and Froude, Ltd., and G. H. Walker, Manchester. Eng. Pat. 115,516, July 28, 1917. (Appl. No. 10,860 of 1917.)

In apparatus of the type in which air is passed through one or more drums formed of convolutions of cloth or other suitable material wound spirally or concentrically around a horizontal shaft, with distance pieces to separate the coils, the liquid is distributed to each drum by perforated pipes, weir plates, gutters, or the like arranged at the edges of the convolutions of the drum. The perforated pipes may be arranged radially from a central annular supply chamber mounted eccentrically on the shaft, or the liquid may be supplied at the outer ends of the radial pipes which are connected to a central eccentric discharge chamber. The pipes are thus moved over the edges of the convolutions during rotation. A rotary extractor may be provided to eliminate moisture from the treated air or gas.—W. F. F.

*Air-filtering or washing apparatus.* P. H. H. Jantzen, London. Eng. Pat. 115,609, Jan. 25, 1918. (Appl. No. 1449 of 1918.)

In an air-filtering or washing apparatus in which air is passed over wet plates, the plates are arranged vertically in groups, the plates of adjacent groups being oppositely inclined to the flow of the air. Grid-bars or distance strips are arranged between the top edges of the plates, and are slightly recessed over the greater part of their length so as to leave a narrow slit between them and the plates. The tops of each group of plates thus form the bottom of a reservoir, and the water passes through the slits and flows down in thin films over the plates. The plates are supported in slotted racks so that they may be easily lifted out.

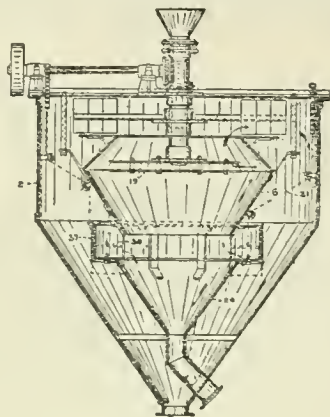
—W. F. F.

*Air-separator.* G. S. Emerick, Philadelphia, Pa. U.S. Pat. 1,263,394, Apr. 23, 1918. Date of appl., Jan. 27, 1917.

The material to be separated is fed over the deflecting plate, 19, into the double conical vessel,



6, and passes downwards over the sloping side of that vessel to the inverted cone, 24. The two conical vessels are connected by an annular vessel, 37, communicating through its lower horizontal face with the outer casing, 2. Air is admitted to



this casing and passes upwards against the flow of material through the annular vessel, 37, and conical vessel, 6, to the outer vessel, 2, as shown by the arrows. The current of air may be regulated by an annular valve plate, 30, movable vertically by cables, 31, operated from outside by bars extending through the top of the outer casing so as to vary the air inlet.—W. F. F.

*Separator [for oils]; Magnetic* —. A. L. Hadley, Fort Wayne, Ind., Assignor to General Electric Co. U.S. Pat. 1,263,406, Apr. 23, 1918. Date of appl., Oct. 7, 1916.

An iron or steel tank is provided with a central vertical partition extending from the top nearly to the bottom, and carrying at its lower end a horizontal plate of smaller diameter than the tank. A similar plate is arranged parallel to the first and below it, so that any liquid passing from one compartment to the other must pass between the plates. A flat solenoid is arranged in the bottom of the tank below the lower plate so that a magnetic field is maintained between the plates. Oil to be cleaned is admitted to one of the compartments and withdrawn from the other.—W. F. F.

*Kiln.* H. R. Straight, Adel, Iowa. U.S. Pat. 1,263,620, Apr. 23, 1918. Date of appl., Sept. 25, 1916.

Gas is supplied from a drum through several small pipes which pass through the crown of the kiln. Each of these gas-pipes is surrounded by a larger one through which the air for the combustion of the gas is supplied.—A. B. S.

*Evaporating liquids; Method and apparatus for* —. W. T. Hoofnagle, Glen Ridge, N.J., Assignor to Electro-Chemical Products Co., New York. U.S. Pat. 1,263,893, Apr. 23, 1918. Date of appl., Oct. 12, 1916.

In a method of evaporating liquids under vacuum, the system is constantly exhausted, and a regulated quantity of ionisable gas is admitted and subjected to an ionising influence. The ionising chamber comprises an outer metal casing, within which is a dielectric vessel containing a conducting liquid. A conduit passing through the dielectric vessel connects the evaporating chamber and the casing.

—J. H. P.

*Extraction of solids; Continuous* —. H. Eberhardt, Wolfenbüttel. Ger. Pat. 302,641, Feb. 8, 1916.

The material descends one or more cylinders containing screw conveyors, meeting a rising stream of solvent. The material collecting in the chamber below is raised by another screw conveyor through an inclined cylinder against a stream of fresh solvent and ejected above the surface. The whole apparatus is enclosed, so that volatile liquids can be handled—H. J. H.

*Centrifuge drum with a surrounding annular chamber.* H. Stegmeyer, Charlottenburg. Ger. Pat. 302,876, Apr. 12, 1916.

The apparatus consists of a casting in the form of a truncated cone divided by a perforated diaphragm in the form of an inverted truncated cone. The liquid separated from the material treated collects in the annular lower chamber and can be returned to the upper chamber.—H. J. H.

*Cooler for salt solutions and other liquids.* Maschinenfabr. Thyssen und Co., Mülheim. Ger. Pat. 302,887, Feb. 12, 1916.

SOLUTIONS are cooled preparatory to crystallisation by being allowed to fall through air in a spray from nozzles which are heated, e.g., by a steam jacket—to prevent premature crystallisation of salts and stoppage of the plant.—H. J. H.

*Oils, resins, waxes, etc., and hygroscopic substances; Conversion of — into dry products.* R. Marcus, Frankfurt. Ger. Pat. 303,122, Feb. 20, 1914.

ARTIFICIALLY prepared magnesium silicate has a higher capacity for adsorption than silicic acid. Alone or with the latter in admixture with liquids and hygroscopic solids it gives dry powders.—H. J. H.

*Refrigerating machines; Method of working compression* —. G. Döderlein. Ger. Pat. 304,077, Dec. 4, 1914.

DRY and moist vapours are separately withdrawn from the compressor and passed through an oil separator before entering the condenser. The compression may also be effected in stages, an oil separator being placed between each compressor and the condenser.—C. A. M.

*Cooling water; Process for increasing the cooling action of* —. W. Billig, Berlin-Friedenau. Ger. Pat. 304,078, Aug. 15, 1915.

GASES are removed from the cooling water, e.g., by filtration through a charcoal filter, and the loss of heat conductivity due to the formation of gas bubbles on the walls of the cooling apparatus is thus prevented.—C. A. M.

*Crushing and pulverising machines.* A. D. Furse and A. J. Neill, London. Eng. Pat. 115,542, July 13, 1917. (Appl. No. 10,150 of 1917.)

*Ammonia compressor.* G. Ferguson, Assignor to Apeldoornsche Maschinenfabriek en Metaalgietterij voorheen L. Landaal, Apeldoorn, Netherlands. U.S. Pat. 1,264,043, Apr. 23, 1918. Date of appl., Nov. 10, 1917.

SEE Eng. Pat. 111,101 of 1917; this J., 1918, 1 A.

*Method of disposing of waste gases from chemical operations.* Eng. Pat. 115,350. See V.

*Method of treating finely-subdivided material.* U.S. Pat. 1,263,082. See XIII.

## II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Combustion; Calculation of the excess of air used in —.* V. Hassfeldt. Metall u. Erz, 1918, 15, 139–143.

When a volume,  $G$ , of producer gas is burned, the volume of air,  $L$ , required is not  $R-G$  as commonly stated, but  $R-G+c-d$ , where  $R$  is the volume of products of combustion, and  $c$  the contraction and  $d$  the expansion due to combustion. For 1 vol. of each of the following gases, when dry, the respective values of  $c$  are:— $C_2H_4$  or  $C_2H_2$ , 2.5;  $CH_4$  or  $C_2H_6$ , 2.0;  $H_2$  or  $C_2H_2$ , 1.5;  $CO$ , 0.5. When the volume of the moist products of combustion is taken, neither expansion nor contraction occurs in the combustion of methane and ethylene; hydrogen and acetylene contract 0.5 vol., and with ethane and benzene an expansion ( $d=0.5$ ) occurs. Again, if  $L_o$  = the volume of air theoretically necessary and  $L$  the excess of air admitted,  $L_o = R-L-G+c-d$ . This equation is applicable to moist flue gases if the volume of the water-vapour in them is taken into consideration. On the customary

assumption that  $L=R-G$ , the ratio  $\frac{L}{L_o}$  may have only half its proper value. If  $E$  = the percentage of air in excess of that theoretically necessary,  $x$  = the excess of oxygen in the products of combustion,  $X$  = the volume of oxygen theoretically required for the combustion, and  $R$  = the volume of products of combustion,  $E = \frac{100 R x}{X}$ . Fischer's ratio,

$\frac{21}{21-79 \frac{o}{n}}$  : 1 (where  $o$  = % oxygen and  $n$  = % nitrogen in the products of combustion), is applicable to coal, coke, and illuminating gas, but not to producer gas and other fuels containing a considerable percentage of nitrogen. Further, it is not satisfactory to use the percentage of nitrogen in the products of combustion as a basis for calculating the excess of air present, as a variation of 10% in the latter causes a variation of only 0.1% in the nitrogen, but a variation of 8% in the free oxygen.—A. B. S.

*Wood; Carbonisation of — in inclined retorts at Värta gasworks, Stockholm.* A. Molin. J. Gasbeleucht., 1918, 61, 50–53.

NEARLY one-half of the gas supply of Stockholm is now manufactured from wood carbonised in inclined retorts. To reduce the excessive proportion of carbon dioxide in the gas from wood, it is arranged that the lower half of the retort should always contain hot charcoal over which the gas passes on its way to the exit. A portion of the carbon dioxide is thereby converted into monoxide, but owing to the violent evolution of the gas in the early stages, much carbon dioxide passes unchanged. On discharging the retort one-half of the charge is left in the lower end. Fresh wood is piled above that and on top coal equal to one-tenth of the weight of wood. Coal is included to furnish hydrocarbons which on decomposition deposit carbon and so keep the retorts gas-tight, and also so that the ammonia liberated shall neutralise any acetic acid which may pass the hot charcoal undecomposed. The aqueous distillate is alkaline. The make of gas is at least 600 cub.

metres per ton, of calorific value 3000–3300 kilocalories per cubic metre. 50 kilos. of wood is charged every 2 hours, and the output of gas is approximately the same as that obtained when coal is carbonised, although the calorific value and the amount of useful coke produced are lower. The coke and charcoal mixture has been successfully used as fuel in gas producers.—H. J. II.

*Olefines; Action of concentrated sulphuric acid on — with particular reference to the refining of petroleum distillates.* B. T. Brooks and I. Humphrey. J. Amer. Chem. Soc., 1918, 40, 822–856.

The principal results of the action of sulphuric acid on olefines are polymerisation and the formation of secondary and tertiary alcohols, and of alkyl hydrogen and dialkyl sulphates; no simple mono-olefine appears to yield "tar" with sulphuric acid at below 15° C. With olefines of higher molecular weight the tendency is mainly to polymerisation with formation of hydrocarbons containing at least one double bond, which are relatively resistant towards sulphuric acid. During the refining of petroleum oils by treatment with sulphuric acid it is therefore probable that the treated oil contains the viscous polymerides and that these do not pass into the acid; contrary to the usually accepted view that the refined viscous petroleum oils of the lubricant type consist exclusively of naphthenes and polynaphthenes, it is consequently likely that they contain a considerable proportion of unsaturated hydrocarbons formed by the polymerisation of simpler olefines, and this view receives confirmation from the appreciable iodine value of such refined oils and from the fact that polymerides prepared by the action of sulphuric acid on definite olefines such as duodecene are very closely similar to lubricating oils of the same molecular weight. In the refining of petroleum distillates, tars are apparently not formed from the mono-ethylenic hydrocarbons but from diolefines, the presence of which is due to the high temperature of the previous distillation. The saturated paraffin hydrocarbons are not attacked by ordinary sulphuric acid although their sulphonation can be effected by fuming sulphuric acid. When gasoline or other hydrocarbon fractions have been refined by treatment with sulphuric acid, they may contain a small percentage of dissolved normal alkyl sulphates and a considerable quantity of polymerised olefines, so that the "keeping" and "drying" qualities may be impaired and re-distillation is necessary to remove these impurities which are introduced during the refining. The unpleasant odour of "cracked" gasolines is due to sulphur compounds and nitrogen bases, whilst the resinification observed on prolonged storage is probably due to diolefines; in these cases "sweet" and stable gasoline can be obtained by treatment with less than 6% by weight of 85–90% sulphuric acid followed by washing with alkali and re-distillation. In the refining of crude benzols the distillation after the sulphuric acid treatment is accompanied by the liberation of sulphur dioxide and the formation of a residue of high-boiling viscous oils which becomes converted into pitch or resin on longer heating; the sulphur dioxide arises from the decomposition of dissolved normal alkyl sulphates whilst the pitch or resin, often erroneously described as "coumarone resin," probably consists of or arises from the highly polymerised products derived from the diolefinic constituents of the crude liquid. As an outcome of the results obtained in this and previous investigations, the conclusion is drawn that at present there is no method for the quantitative determination of the volume percentage of unsaturated hydrocarbons in petroleum oils. (See also J. Chem. Soc., 1918, 1, 286.)—D. F. T.



*Paraffin wax; Oxidation products of* — M. Bergmann. Z. angew. Chem., 1918, 31, 69—70.

REFINED white paraffin wax, m. pt. 52° C., of Galician origin was oxidised by blowing through it a rapid current of air at 130°—135° C. for 15—18 days. The product was a brown, unctuous mass, with acid value 132, forming soaps with alkalis. The acid products were separated as barium salts and fractionated by distillation under reduced pressure after liberation by hydrochloric acid. The fraction distilling from 270° to 310° C. under 100 mm. pressure was further purified and identified by its m. pt., 80.1° C., and by that of its ethyl ester, 55° C., as lignoceric acid, which occurs in arachis oil and beech-wood paraffin. From the fraction distilling between 230° and 270° C. were isolated a neutral body, m. pt. 48° C., crystallising in leaflets, an acid,  $C_{11}H_{22}O_2$ , m. pt. 53.7° C., and another acid, m. pt. 38.4° C., which might be an isopalmitic acid,  $C_{16}H_{32}O_2$ . (See also J. Chem. Soc., 1918, I., 285.)—J. F. B.

*Effect of load on refractoriness of firebricks, etc.* Mellor and Emery. Report of Refractory Materials Research Committee of Inst. Gas Engineers. See VIII.

#### PATENTS.

*Coking ovens; Horizontal* — Soc. Franco-Belge de Fours à Coke, Brussels. Eng. Pat. 113,779, Nov. 9, 1917. (Appl. No. 16,469 of 1917.) Under Int. Conv., Feb. 26, 1917.

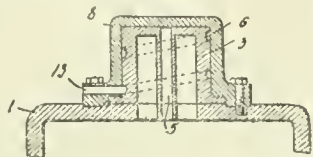
THE oven is divided transversely into two substantially symmetrical parts heated independently, and the space below each half is divided by a partition into two similar independent recuperators. Producer gas is passed through one of these recuperators from bottom to top and thence to the burner at the base of the combustion chamber, and air is similarly passed through the other recuperator to the burner. Combustion products pass from the top of the vertical heating flues into a horizontal passage and thence downwards through vertical conduits to the two recuperators through which they pass in zig-zag paths to pre-heat the gas and air. No reversal of the gas, air, and combustion products is necessary.

—W. F. F.

*Gas generators; Method for operating* — [with recovery of ammonium chloride]. A. Riedel, Kössern, Saxony. Eng. Pat. 101,220, Aug. 16, 1916. (Appl. No. 11,575 of 1916.) Under Int. Conv., Aug. 16, 1915.

IN the normal working of a gas producer, chlorides such as common salt, calcium or manganese chlorides or the like, are added, together with water, the amount of chlorine in the salt being equivalent to the nitrogen content of the coal. The whole of the nitrogen is thereby converted into ammonium chloride, which is recovered by condensation.—W. F. F.

*Gasifying retort.* H. R. Wemple, Elizabeth, N.J. U.S. Pat. 1,262,886, Apr. 16, 1918. Date of appl., Apr. 6, 1917.



A CUP-SHAPED member, 3, has its open end mounted on the intake, 1, of an engine and is provided with

a nozzle, 5, fixed at the top as shown. A cap, 8, fits over the member, 3, and fuel to be gasified passes through the opening, 13, and helical groove, 6, to the top of the nozzle, 5.—W. F. F.

*Gas producer.* F. Siemens, Berlin. Ger. Pats. (a) 303,062, Apr. 24, 1917, and (b) 303,562, May 10, 1917.

(A) IN addition to the usual gas outlet the producer is provided with two openings in the lower part, through one of which a highly heated gas is introduced to decompose the fuel, whilst through the other a portion of the gas produced is withdrawn for the preliminary heating of the gas used for the decomposition. These openings are connected with regenerators, one of which is used for heating the gas used for the decomposition, whilst the other is heated by the combustion of the gas withdrawn from the producer. The regenerators are so arranged that heat can be conducted to the fuel in the producer through one of the walls of the regenerators or of the flues leading thereto. The gas withdrawn for heating purposes is preferably taken from a place in the producer where the temperature is high, but where tarry compounds are not formed. (B) By far the greater portion of the producer gas is passed to the regenerator as described under (A), only a small portion, sufficient to ensure decomposition of the fuel in the producer, being withdrawn through the usual gas outlet.

—C. A. M.

*Methane; Preparation of* — Badische Anilin u. Soda Fabrik. Ger. Pat. 303,718, Sep. 12, 1914. Addition to Ger. Pat. 292,615.

NICKEL catalysts as claimed in the original patent (see Fr. Pat. 463,114 of 1913; this J., 1914, 313) and also as prepared by impregnating substrata with relatively small amounts of nickel salt solutions free from halogens and sulphur (Ger. Pat. 297,258; this J., 1917, 873) are used for the preparation of methane at temperatures not materially exceeding 450° C. and avoiding a large excess of steam. This enables water-gas or other gaseous mixture containing carbon monoxide to be used in place of carbon monoxide.—C. A. M.

*Tarry substances such as pitch or steel works tar and tar oils; Process for the fractional separation of* — from superheated crude gases H., G., and E. Feld, Linz. Ger. Pat. 303,195, Oct. 12, 1913.

HOT water at or above the temperature of the "dew point" of the gas for steam is sprayed into the gas in such limited quantity that the temperature is reduced only to the point necessary for the separation of the desired tarry product. The receivers into which the gases pass from the retort may also be washed with water at or above the temperature of the "dew point" for steam. A thermostat may be used in which the gases and the water rise concentrically about the entrance tube, where they are again heated before leaving the thermostat. By repeatedly passing the washing agents used for washing the tar constituents of high boiling point through the gas-washing apparatus tar fractions of definite uniform composition may be obtained.—C. A. M.

*Liquid fuel.* H. Shannon, Richmond, Surrey, Assignee of W. P. Bending, San Francisco, Cal., U.S.A. Eng. Pat. 114,411, Sep. 7, 1917. (Appl. No. 12,856 of 1917.) Under Int. Conv., Mar. 27, 1917.

A SUITABLE quantity of animal fat or grease is added to a mixture of approximately equal volumes of

hydrocarbon oil and water, previously heated to a temperature sufficient to melt the added fat, and the whole agitated to form an emulsion. The mixture is suitable for use as a liquid fuel.

—T. St.

*Distillate of shale free from sulphur; Process for obtaining a —.* G. E. Heyl, London. Eng. Pat. 115,452, Sep. 14, 1917. (Appl. No. 4249 of 1917.)

The shale is finely ground with sufficient ferrous or ferric chloride to combine with all the sulphur contained in the shale, and the mixture distilled. It is claimed that practically all the sulphur is retained as iron sulphide.—T. St.

*Motor fuels and light paraffin oils; Method of obtaining — from shale; and benzene, toluene and solvent naphtha from coal.* P. M. Justice, London. From S. G. Pirani, Melbourne, Australia. Eng. Pat. 115,573, Sep. 19, 1917. (Appl. No. 13,485 of 1917.)

Shale or coal in the form of coarse powder is mixed with a finely ground material, such as limestone, which under the action of heat will give off carbon dioxide, and with small iron scrap, or its chemical equivalent, and the mixture is slowly heated in a vertical retort to temperatures ranging from 175° to 800° C. according to the oils required. Shale may also be treated in the form of small lumps, when it is desired to obtain a coke ready for market without further treatment. The carbon dioxide given off during the distillation acts as a carrier for the hydrocarbon vapours evolved from the shale or coal, and prevents them from becoming permanently fixed gases, while the action of the iron scrap increases the volume of hydrocarbon vapours given off in the retort. The proportions of the ingredients vary according to the amount of oxygen contained in the coal or shale. With an oxygen content of 6 to 13% a suitable amount of limestone is 3 to 8%, and of iron scrap 1½ to 4%. When no more vapours are evolved the temperature is raised beyond 800° C. and water in the form of a fine mist or jet is injected into the lower part of the retort. The vapours formed in the two operations are passed through a condenser, and any uncondensed vapours are then passed through an absorbing tower containing coke which has been saturated with a suitable non-volatile oil. The condensed oils are separated by fractional distillation. The residual oils from the various distillations may be mixed with fresh quantities of limestone and iron scrap and subjected again to distillation. The vapours formed in this operation are however passed through a second retort exactly like the first, and at the same temperature, but charged to about half its capacity with manganese dioxide and iron scrap, preferably in the proportions of 1 part of the former to 2 parts of the latter. The effect of these last-mentioned materials is to enhance the production of condensable vapours.—T. St.

*Oil; Recovery of — from oil-sands.* F. Squires, Marletta, Assignor to W. Squires, Jamesville, Ohio. U.S. Pat. 1,263,618, Apr. 23, 1918. Date of appl., Jan. 26, 1918.

A GASEOUS medium is caused to pass through the oil-sand towards the pumping well, where the oil vapour carried by the gas is condensed. The oil is pumped from the well and the gaseous medium allowed to escape upwards through the well, a counter-current of oil supplied from the top of the well absorbing any remaining uncondensed vapour.

—L. A. C.

*Sludge acid [from mineral oil refining]; Concentration and purification of —.* W. A. Slater, Fort Worth, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,263,950, Apr. 23, 1918. Date of appl., May 18, 1917.

Acid sludge is treated with dilute acid and the weak acid is separated and concentrated by causing it to flow against a counter-current of heated air which has been blown through a body of previously concentrated acid in order to concentrate it further and to oxidise impurities therein. The air is finally scrubbed with water to furnish the dilute acid for the initial treatment of the sludge.

—L. A. C.

*[Gas] retorts; Construction of vertical —.* H. J. Toogood, and R. Dempster and Sons, Ltd., Elland. Eng. Pat. 115,738, July 16, 1917. (Appl. No. 10,282 of 1917.)

*Oil-gas and other gas furnaces.* Eng. Pat. 115,214. See I.

*Magnetic separator [for oils].* U.S. Pat. 1,263,406. See I.

*Method and plant for the distillation of tar, crude petroleum, resin, and similar materials.* Ger. Pat. 302,323. See III.

*Oil-tester [for determining the flash-point].* U.S. Pat. 1,263,145. See XXIII.

## IIB.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Carbonisation and distillation of peat, sawdust, sweepings and other waste organic matter.* C. Galaine and C. Houlbert. Comptes rend., 1918, 166, 854—856.

Six cylindrical rotating retorts are mounted on separate shafts on a frame which enables the retorts to be heated in turn in a fixed semi-circular furnace. Three retorts are heated simultaneously, whilst one is cooling, one being emptied, and one being filled at the same time. With retorts each containing 1 ton of peat and rotating 10 times per min., the distillation requires about 40 min. The gas passes away through a hollow shaft in each retort to a central collecting flue in the main shaft of the frame. The by-products are treated in the customary manner for the recovery of ammonia, etc. By the rotation of the retorts, combined with the large surface exposed to the heat, rapid transmission of heat through them is attained, and hence the distillation can be carried out at a lower furnace temperature than in other apparatus. It is suggested that this arrangement should be used for the destructive distillation of household refuse.

—A. B. S.

*Incandescence lamps; Crystals in filament form as used in electric —.* F. Schröter. J. Gasbeleucht., 1918, 61, 44—45.

THE effect of work on a tungsten filament is to convert the metal into the labile elastic form which reverts to the crystalline at high temperatures. Crystal boundaries are produced which are points of weakness and readily fracture. In the process of J. Pintsch (Fr. Pat. 469,212 and Ger. Pat. 304,857; this J., 1915, 36; 1918, 331 A) this is avoided by producing a filament consisting of one homogeneous elastic crystal. A drawn and sintered



thread is made of tungsten with the addition of 2% of thorium oxide which acts as a catalyst, and its micro-structure is found to resemble a bundle of crystals of octagonal section. The filament is drawn through a cylindrical electrically heated furnace with a zone at 2400°–2600° C. and an atmosphere of hydrogen. The rate of motion—about 2.5 metres per hour—is somewhat slower than that of crystal growth. The crystals, already existing, grow and coalesce so that at the end the whole filament consists of one crystal, which cannot therefore grow on further heating or develop new crystal junctions. The filaments show a high degree of pliability which is very permanent in use.  
—H. J. H.

*Manufacture of paper pulp, etc., from dead leaves.*  
Bramson. See V.

#### PATENT.

*Seal for retorts in distillation plants.* W. Schacht, Weissenfels. Ger. Pat. 302,754, Aug. 26, 1916.

THE lid of a retort used for the dry distillation of residues of cellulose factories may be sealed with a metal or alloy liquid at the temperature of distillation. Up to 500° C. loss by volatilisation is negligible. An outer seal of high-boiling oil may be provided in addition to the seal of molten metal.  
—H. J. H.

### III.—TAR AND TAR PRODUCTS.

*Toluol [and cumene] from spruce turpentine.* A. S. Wheeler. J. Ind. Eng. Chem., 1918, 10, 359–360.

SPRUCE turpentine (see this J., 1918, 296 A) was purified by distillation in superheated steam, the vapour being passed through 10% caustic soda and then condensed. The oil was separated from water, shaken with 0.5% potassium permanganate and then with strong sulphuric acid. The colourless purified oil, consisting mainly of cymene, was mixed with benzol 10 parts by weight and aluminium chloride 0.05 part by weight and boiled for 10 hours on a water-bath, the solution becoming dark red. The product was fractionated with a 3-section Young still head, and the portion distilling at 83°–95° C. was redistilled. Accumulations occurred at about 110° and 153° C., the boiling points of toluene and cumene. The cumene may be utilised by direct oxidation to benzoic acid.—W. F. F.

*Action of concentrated sulphuric acid on olefines with particular reference to the refining of petroleum distillates.* Brooks and Humphrey. See IIa.

#### PATENTS.

*Distillation of tar, crude petroleum, resin, and similar materials; Method and plant for the —.* T. Weickel, Weinsheim Customs House. Ger. Pat. 302,323, Jan. 16, 1917.

HORIZONTAL stills for tar distillation, etc., need not be walled in if the fire gases approach and leave at the same region.—J. C. W.

*Method of obtaining motor fuels and light paraffin oils from shale, and benzene, toluene, and solvent naphtha from coal.* Eng. Pat. 115,573. See IIa.

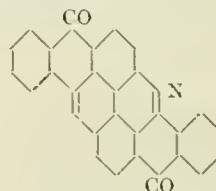
*Concentration and purification of sludge acid.*  
U.S. Pat. 1,263,950. See IIa.

*Process for the fractional separation of tarry substances such as pitch or steel-works tar and tar oils from superheated crude gases.* Ger. Pat. 303,195. See IIa.

### IV.—COLOURING MATTERS AND DYES.

*Pyranthridone.* R. Scholl and O. Dischendorfer. Ber., 1918, 51, 441–452.

THE synthesis of pyranthridone,



which is intermediate between pyranthrone,  $C_{16}H_{14}O_2$ , and flavanthrene,  $C_{22}H_{12}O_2N_2$ , is described. The substance sublimes without melting in brownish-yellow needles, and forms a violet-blue hydrosulphite vat from which cotton is dyed orange-red, after hanging in the air. The parent base, pyranthridine,  $C_{22}H_{17}N$ , is also described. (See further J. Chem. Soc., 1918, I., 307.)—J. C. W.

*Flavanthrene; Synthesis of —.* R. Scholl and O. Dischendorfer. Ber., 1918, 51, 452–453.

THE immediate precursor of flavanthrene in various syntheses is 2,2'-diamino-1,1'-dianthraquinonyl. This has now been isolated for the first time, in microscopic, red needles, which change into flavanthrene at 250° C. (See further J. Chem. Soc., 1918, I., 308.)—J. C. W.

*Rhoduline; The simplest —.* F. Kehrmann and M. Ramm. Ber., 1918, 51, 385–388.

WHEN 3,7-diacetylaminophenazine is heated with methyl sulphate and nitrobenzene, a mixture of three methyl derivatives is formed, from which it is possible to obtain 3,7-diamino-5-methylphenazine in the form of salts. The solution of the perchlorate in concentrated sulphuric acid is green but becomes violet-blue and then red on dilution with water. (See further J. Chem. Soc., 1918, I., 313.)—J. C. W.

*Phencyazonium compounds.* F. Kehrmann and M. Sandoz. Ber., 1918, 51, 388–391.

WHEN a warm solution of Kaufmann and Albertini's 5-cyano-10-methyldihydroacridine (Ber., 1909, 42, 1999) in glacial acetic acid is exposed to the air, it becomes deep yellow owing to oxidation to 5-cyano-10-methylacridinium acetate. The corresponding crystalline perchlorate may be isolated. The base is now designated 10-methylphenacyazonium as it greatly resembles phenazonium as a chromogen. It may be regarded as the parent of Ehrlich and Benda's tryptaflavine (this J., 1913, 745. See further J. Chem. Soc., 1918, I., 313).  
—J. C. W.

*Constitution and colour. VI. Triphenylmethane dyes.* F. Kehrmann. Ber., 1918, 51, 468–474.

It is suggested that triphenylmethane bases form salts in strongly acid solutions in which the acid residues are attached not only in the usual way, as in solutions in more dilute acids, but also to the central carbon atom, by the exercise of a fifth and sixth valency or by residual affinity. (See further J. Chem. Soc., 1918, I., 311.)—J. C. W.

*Jellies formed by dyes.* R. Haller. *Kolloid Zeits.*, 1918, 22, 49—53.

WHEN certain substantive dyes are dissolved in hot water, solutions are obtained which form jellies on cooling. The structure of the jellies formed by Benzopurpurin B and Chrysophenin B has been examined and found to be very similar to that of the soap jellies investigated by Zsigmondy and Bachmann (this J., 1912, 1084). The formation of the jellies seems to be dependent on the presence of small quantities of electrolyte in the dyes, for when the solutions are purified by dialysis, coagulation takes place and the gelatinous coagulum is no longer soluble even in hot water. The addition of sodium chloride is however sufficient to bring the dye into solution. If the purified gelatinous coagulum is dried and powdered, the product resembles closely the original material but is sharply distinguished from this by its insolubility in hot water. The experiments show that the small quantities of electrolytes (sulphates and chlorides) which are present in the commercial substantive dyes are of very important significance in the technical application of these substances, but as yet no satisfactory explanation of the phenomena can be given. (Compare *J. Chem. Soc.*, July, 1918.) —H. M. D.

#### PATENTS.

*Vat dyestuffs of the carbazole-naphthoquinone series; Manufacture of —.* M. P. Schmidt, Assignor to Kalle und Co., Biebrich, Germany. U.S. Pat. 1,261,858, Apr. 9, 1918. Date of appl., Apr. 28, 1916.

CARBAZOLE-NAPHTHOQUINONES are condensed with compounds containing reactive hydrogen atoms, capable of condensation with a benzo- or naphthoquinone, giving brown to black powders, insoluble in water, sparingly soluble in the common organic media, and forming yellow to brown vats with alkaline reducing agents, from which fibres are dyed brown to black tints. The condensation products of carbazole-naphthoquinone (including toluencarbazole-naphthoquinone) with oxythionaphthene dissolve in concentrated sulphuric acid with greenish-blue colours, form yellow vats, and dye wool violet-black tints, the shades becoming redder on treatment with hot acids.—J. C. W.

*Vat dyes of the anthracene series; Manufacture of nitrogenous —.* Chem. Fabr. Griesheim-Elektron. Ger. Pat. 302,259, Dec. 22, 1914. Addition to Ger. Pat. 301,554 (see Eng. Pat. 14,103 of 1915; this J., 1916, 1104).

THE alkali salts of the partially alkylated dyes mentioned in the chief patent are alkylated further, different alkyl groups being introduced.—J. C. W.

*Vat dyestuff of the anthracene series; Manufacture of a golden-orange —.* Chem. Fabr. Griesheim-Elektron. Ger. Pat. 302,260, June 6, 1916.

THE benzyl derivative of Pyrazole-anthrone Yellow (Eng. Pat. 14,103 of 1915; this J., 1916, 1104) is warmed with sulphuric acid. The product gives a bluish olive green vat which dyes cotton fast golden orange.—J. C. W.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Cellulose of cereal straw; Nature of the —.* E. Heuser and A. Haug. *Z. angew. Chem.*, 1918, 31, 99—100, 103—104.

IN the determination of cellulose in straw by the chlorine process the separation of the last residue of lignin presents some difficulty, and if the usual

sulphite solution be employed for extracting the chlorinated product, a large number of chlorine treatments is necessary, so that the cellulose is abnormally attacked. On the other hand, if a 1.0% solution of sodium hydroxide is used after each chlorination, a pure cellulose can be isolated in a reasonable time. According to the method recommended by the authors, 2 grms. of chopped straw (internodes) is placed in the Gooch crucible (see this J., 1914, 71) and treated with a strong current of steam for 10 mins. to soften the material. It is then thoroughly washed and treated with chlorine gas for half an hour. The hydrochloric acid produced is washed out, and the crucible placed in a beaker containing about 100 c.c. of 1% caustic soda solution which is heated for 10 mins. at a temperature not exceeding 70° C. The treatment with chlorine gas and caustic alkali is performed four times in all, for half an hour each time, and the cellulose is finally bleached with 0.1% permanganate. The yield of cellulose, calculated on the dry and ash-free basis, is 54.75—55.60%; the product is free from lignin and oxycellulose but still contains pentosans (furfuroids). In an example given, the amount of hydrochloric acid produced in the first four chlorinations of half an hour each was 28.5%; the total after 10 chlorinations was 32.34%. After 5 chlorinations, the amount of hydrochloric acid produced per treatment became constant (0.55—0.60%). The amount of chlorine combining with the lignin ranged from 9.50 to 10.81%. The action of the chlorine gas on the pentosans has been studied by the treatment of an isolated preparation of xylan. The quantity of hydrochloric acid produced after 4 hours' treatment with chlorine was 9.0% and after 15 hours, 16.9%, and the amount of chlorine fixed as xylan chloride was 16.8%. These figures suggest combination of the xylan with chlorine by substitution, but they cannot be regarded as final. This reaction is much slower than the reaction of chlorine with lignin, and only a small portion of the acid produced in the chlorination of straw can be attributed to the xylan. In the chlorination of straw, a portion of the xylan is hydrolysed in the initial treatment with chlorine and appears in the extract in the form of pentose. In the later treatments it is dissolved without hydrolysis.—J. F. B.

*Wood pulp.* C. G. Schwalbe. *Z. angew. Chem.*, 1918, 31, 50—56, 57—60.

COMMERCIAL wood pulps contain about the same quantity of cellulose, namely 91 to 92%. Mitscherlich pulp and Ritter-Kellner sulphite pulp contain the least quantity of pentosans, soda pulp the most. There is no great difference in the lignin content of the pulps. Sulphite pulps yield the most ether-extract (0.7%), whilst soda pulps give much less (0.2%). Half-stuff and three-quarter-stuff contain a considerable quantity of pentosans, and lignin or sugar, but there are, possibly, two kinds of half-stuff, the one rich in lignin and the other in pentosan. A true half-stuff, with a yield of 80 to 90%, might be prepared from half-stuff rich in pentosan but a suitable solvent or hydrolytic agent for the purpose is not yet known. The cell-length of pulp is about 2 to 3 mm., which is one-half the minimum length required for good spinning fibres; probably bundles of supple fibres provide the necessary length in the case of pulp. —W. P. S.

*Wood pulp; Colour reaction of mechanical — or of the incrusting substances of wood with phenylhydrazine hydrochloride.* S. Jentsch. *Z. angew. Chem.*, 1918, 31, 72.

THE lignocellulose of wood combines with phenylhydrazine hydrochloride in aqueous solution in



definite proportions capable of quantitative measurement. The orange-yellow compound so produced, when dried with free exposure to air, acquires a characteristic bright green colour which is more definite than the crimson coloration produced by phloroglucinol and hydrochloric acid. The development of the green coloration is hastened by heating, so that it may appear after a few seconds with great clearness. Pure cellulose fibres, on treatment with phenylhydrazine hydrochloride, are coloured only pale yellow and assume a characteristic light brown colour on drying. The phenylhydrazine reagent possesses several advantages over the strongly acid phloroglucinol reagent and is recommended as an indicator for the presence of woody fibre in paper and for the estimation of the degree of purification of cellulose fibres from lignified materials.—J. F. B.

*Sorghum vulgare* (millet); Use of — in the cellulose industry. B. Haas. Z. angew. Chem., 1918, 31, 84.

THE stems and branches of *Sorghum vulgare* are being extracted for the purpose of beer brewing. When treated like esparto the residue has been found to yield a fibre well adapted to the manufacture of paper and textile fabrics. The treatment is, however, simpler, the time of heating, steam pressure, and quantity of chemicals required all being smaller. The manufacture might be linked up with that of alcohol.—H. J. H.

*Paper-pulp, etc.; Manufacture of — from dead leaves.* K. Bramson. Comptes rend., 1918, 166, 853–854.

THE process is simple, rapid and not costly. The leaves are crushed and separated into two parts, i.e., the veins and ribs, and a powder. The veins and ribs, which yield the paper pulp, are submitted to a rapid washing with alkali, followed by washing with water and bleaching, and then made into pulp. The powder, which has a feeding value almost equal to that of hay, may be mixed with molasses and compressed into cakes and fed to animals. An alternative use for the powder is to submit it to dry distillation, when it yields a relatively pure, porous carbon, with a high calorific value, tar, acetone, and pyroligneous acid. In this way 1000 kilos. of leaves yields 250 kilos. of paper pulp and 500 kilos. of fodder or 200 kilos. of carbon, 30 kilos. of tar, 1 kilo. of pyroligneous acid, and 600 grms. of acetone.—W. G.

*Artificial silk; Treatment of washing water in the production of —.* M. de Chardonnet. Comptes rend., 1918, 166, 753.

IN the manufacture of artificial silk from a collodion base about 4 cubic metres of waste liquor is produced per kilo. of silk. The washings from the denitration partly neutralise the liquor obtained by washing the pyroxylin. The waste liquors contain per cubic metre: nitric acid, 0.650; sulphuric acid, 1.10; sulphur in a finely divided state, 0.175; calcium, 0.11 kilo.; the free acid being from 1 to 1.50 kilo. per cubic metre. The washings are conveyed through separate stoneware pipes to a small pond in which admixture takes place in a closed space so as to avoid a nuisance from the nitrons gases evolved on mixing. The partly neutralised liquor then passes over a weir into tanks in which neutralisation is completed by the addition of ground limestone or quicklime. It is unnecessary to clean out the tanks as sufficient water is always present to ensure complete solution of the calcium

nitrate and sulphate produced, and the finely divided sulphur remains in suspension. The neutralised liquor is said to be a good fertiliser.

—C. A. K.

*Toluol [and cumene] from spruce turpentine.* Wheeler. See III.

#### PATENTS.

*Fabrics; Machine for drying — in open form.* C. Taylor, West Nutley, N.J., U.S.A. Eng. Pat. 110,747, Oct. 6, 1917. (Appl. No. 14,474 of 1917.) Under Int. Conv., Oct. 21, 1916.

AN enclosed casing through which a drying medium, such as hot air, is circulated contains an endless travelling member with rollers for hanging the fabrics, and means are provided for rotating the rollers while they are carried through the casing by the travelling member. The fabric thus hangs without tension in festoons and receives a vertical travelling motion through the rotation of the rollers as well as a horizontal motion forward with the rollers. The lower portions of the loops of fabric are supported by a travelling platform which moves forward through the casing at the same speed as the rollers. Means are provided to guide the fabric on the rollers, and to draw the fabric into and deliver it out of the casing.—J. F. B.

*Rice straw; Process of producing fibrous material from —.* J. K. Toles, San Francisco, Cal. Assignor to Union Fibre Co., Winona, Minn. U.S. Pat. 1,262,872, Apr. 16, 1918. Date of appl., Dec. 20, 1915.

RICE straw is immersed in a "nominally weak" alkaline solution which is boiled for a sufficient time to dissolve the gummy substances surrounding the rice fibres without pulping or disintegrating them; the fibres are then freed from excess of gums and alkaline solution.—J. F. B.

*Fibres; Process for obtaining textile —.* A. Frohmader, Nuremberg. Ger. Pat. 301,233, Aug. 30, 1916.

GRASS-LIKE or herbaceous plants are treated with dilute caustic soda at the ordinary or a moderate temperature; the fibres are loosened by a gentle mechanical movement during or after steeping, then separated from the swollen or dissolved incrusting matters, washed, if necessary with further agitation, dried, and worked up in the ordinary manner.—J. F. B.

*Wool; Process for testing the wearing quality of —.* P. von Allwörden, Osterode. Ger. Pat. 302,808, Jan. 30, 1916.

WOOL is moistened with chlorine water and examined under the microscope in order to see whether the swellings, which are characteristic of good wool, appear under the scales. These swellings are due to the presence of "elastium" between the fibre cells and the epidermal scales, the removal of which in the scouring process makes the wool tender and brittle and incapable of fulling and finishing. (See also this J., 1916, 416; 1917, 706.)—J. F. B.

*Fibres and textiles; Process for making woolly artificial —.* P. Kraiss, Tübingen. Ger. Pat. 303,731, Mar. 29, 1917. Addition to Ger. Pat. 302,611.

PARCHMENTISED paper pulp is employed as a binding medium in the process described in the chief patent

(this J., 1918, 332 A). Parchment papers may be prepared containing 50% or more of wool; these are very strong and may be made pliable and water-resistant by suitable treatment and additions.

—J. F. B.

*Waterproof [fibrous] composition and process for making such composition [from spent bark].*

V. A. Wallin, Grand Rapids, Mich., and O. A. Heppes, La Grange, Ill. U.S. Pat. 1,263,823, Apr. 23, 1918. Date of appl., Mar. 6, 1917.

Spent bark is finely divided crosswise of the grain into pieces so short as to be capable of saturation from end to end; the product is mixed with a fibre such as will afford tensile strength and the mass is felted together to form a sheet, to which a waterproofing material is intimately applied.—J. F. B.

*Cellulose acetate and a process of making same.*

F. Ruppert, Mainz-Mombach, Assignor to Verein für Chemische Industrie in Mainz, Frankfurt, Germany. U.S. Pat. 1,263,119, Apr. 16, 1918. Date of appl., Nov. 13, 1914.

CELLULOSE is treated with acetic anhydride or with acetic acid and anhydride, below 20° C., in presence of a metalloids chloride as a catalyst but without the addition of a diluting agent, avoiding the solution of the reaction product, and the reaction is interrupted as soon as the mass forms an unliquefied jelly soluble in tetrachloroethane but insoluble in cold glacial acetic acid.—J. F. B.

*Cellulose acetates; Process for the manufacture of masses or films of low melting point from infusible —.* Verein für Chemische Industrie, Frankfurt. Ger. Pat. 303,018, Nov. 7, 1912.

CELLULOSE acetate is incorporated with triphenyl phosphate to form a homogeneous mass, which melts under the action of heat and falls away without bursting into flame.—J. F. B.

*Viscous liquids; Process for spinning — with use of liquids in motion for drawing off the threads.* J. P. Bemberg A.-G., Barmen-Rittershausen. Ger. Pat. 303,047, Aug. 26, 1916.

A MOVING liquid is used which is free from dissolved gases or from which these have been removed by evacuation, heating, or the like. (Compare Eng. Pat. 113,010 of 1917; this J., 1918, 146 A.) —J. F. B.

*Plants; Process for treating parts of —.* C. Melhardt, Starnberg. Ger. Pat. 303,730, Apr. 19, 1917.

Plants of plants are treated for the separation of fibres, cellulose, or cell-contents, by steeping in dilute alkali solutions, with suitable additions to prevent putrefaction and fermentation, in pits from which air is excluded, until the soluble matters can be separated by washing or leaching and the insoluble matters by pressing.—J. F. B.

*Sulphite pulp process; Reclaiming system in a —.* C. B. Thorne, Hawkesbury, Ont., Canada. U.S. Pat. 1,263,486, Apr. 23, 1918. Date of appl., Nov. 22, 1913. Renewed, Sept. 22, 1917.

THE system comprises a number of acid-forming tanks, an acid receptacle, connected with a gas-generating plant, receiving liquor from the digester and delivering gases to the acid tanks, and a second receptacle receiving liquor from the acid receptacle and gases from the digester. The gases exhausted from the digester pass to a separator, the gas outlet

of which is connected with a recovery tower and the liquor outlet with an acid tower which in turn is connected with the recovery tower and with the acid-forming tanks, and means are provided for delivering the liquor from the tanks to the acid tower and from the latter to the recovery tower.

—J. F. B.

*Waste gases from chemical operations [e.g., from the sulphate-cellulose process]; Method of disposing of —.* A. G. Bloxam, London. From Zellstoff-fabrik Waldhof, Mannheim-Waldhof, Germany. Eng. Pat. 115,350, Aug. 10, 1917. (Appl. No. 11,539 of 1917.)

MALODOROUS waste gases, such as those produced in the manufacture of cellulose by the soda-sulphate process, are led directly into the primary air-intake of the gasifying chamber of a fuel gas producer. The gases are thus forced to pass through a relatively deep combustion zone and any small residue which escapes combustion is again subject to combustion when the fuel gas is burnt.

—J. F. B.

*Impregnating material for leather, leather substitutes, fabrics, etc.* Ger. Pat. 302,158. See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Jellies formed by dyes.* Haller. See IV.

*Electrical endosmosc.* II. Briggs and others. See XI.

### PATENTS.

*[Aniline] Black on vegetable fibres; Production of —.* The Calico Printers' Association, Ltd., and E. A. Fourneaux, Manchester. Eng. Pat. 115,278, May 5, 1917. (Appl. No. 6114 of 1917.)

MIXTURES are used consisting of (a) aniline, (b) one or more primary amines of the benzene series, comprising the substitution products of aniline and their homologues, benzidine and its homologues and substitution products, but not diphenylamine compounds nor phenylenediamine, *p*-aminophenol and their homologues, (c) hydrochloric acid and/or nitric acid, (d) formic acid, which may be replaced to the extent of one-fifth by other carboxylic acids, (e) chlorates of the alkali metals, (f) one or more catalytic agents comprising copper and/or vanadium salts alone or in conjunction with *p*-phenylenediamine or *p*-aminophenol or their homologues, preferably in the form of hydrochlorides. Example: —Aniline, 39.5; aniline salt, 6.5; benzidine dihydrochloride, 20.5; *p*-phenylenediamine dihydrochloride, 1.75; formic acid (90%), 65; lactic acid (30%), 30; sodium chlorate, 28; copper chloride, 7.75; starch, 35; gum dragon mucilage, 150 parts by weight; water to 1000 parts by volume.—J. F. B.

*Dyestuff pastes, solutions, and vats; Process for preparing —.* S. Aschkenasi, Berlin. Ger. Pat. 303,121, Aug. 8, 1916.

THE so-called fat-splitting agents are used as additions to the dye-bath to produce emulsification. Neutral or acid hydrosulphite vats are prepared by adding to the ordinary alkaline vats, after reduction by hydrosulphite, a fat-splitting agent or its aqueous solution at a temperature not exceeding 40° C. In the case of indigo a fat-splitting agent, such as Twitchell's reagent, is added, in the



proportion of 1–3% of the dyestuff, and the mixture gives a homogeneous emulsion on stirring with water. This paste is stirred with concentrated alkali hydroxide and then reduced with hydro-sulphite; the product on dilution gives an alkaline vat.—J. F. B.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Hydrochloric acid from the waste liquors of the potash industry; Manufacture of —.* B. Lepsius. *Z. angew. Chem.*, 1918, **31**, 93–95.

According to Hepke's process (Ger. Pat. 278,106; this J., 1915, 227) the final liquors from the German potash salt factories, containing magnesium chloride, are evaporated to about two-thirds of their bulk, mixed with 15% of crude magnesia, and the mass is allowed to set in moulds. The magnesium oxychloride is broken up and calcined in a gas-heated rotary tube furnace, 35 m. long. The maximum temperature required is 600° C. The products are crude magnesia, containing 80–90% MgO, and hydrochloric acid which is easily collected and condensed in long quartz pipes. The yield is 75% of the theoretical. The question whether this process, which is admittedly a practical one, is capable of solving the difficulty in connection with the disposal of the waste liquors and the contamination of the rivers with soluble chlorides, depends on the commercial outlets for the products. With regard to the magnesia, no fundamental difficulty is expected. The German iron industry consumed about 350,000 tons of magnesia annually, manufactured from magnesite imported from Greece; the use of magnesium oxychloride cements for making artificial wood plasters accounted for about 30,000 tons. With regard to the hydrochloric acid, the outlook is not favourable. The transport of this article limits its utility, as the centres of consumption are far removed from those of production. Leblanc hydrochloric acid will not be easily displaced, because the salt-cake is required for the glass industry. Whether hydrochloric acid can replace sulphuric acid for the absorption of ammonia from coke and gas ovens for agricultural purposes is not yet clear, but in any case the chlorides would ultimately find their way to the rivers and the situation would not be relieved. A large extension of the oxychloride cement industry would be favourable, but this product is not weather-proof and its utilisation cannot become sufficiently general.

—J. F. B.

*Nitric acid; Apparatus for the determination of — by the Schulze-Tiemann method.* K. Leuchs. *Chem.-Zeit.*, 1918, **42**, 235.

The apparatus described is constructed entirely of glass. The decomposition flask is provided with a glass stopper carrying a tap-funnel and a delivery tube, the stopper being surrounded by a water-seal. A non-return glass valve is fitted in the delivery tube midway between the flask and the gas burette. (See also *J. Chem. Soc.*, July, 1918.)—W. P. S.

*Ammonia; Synthesis of — at high temperatures.* H. E. B. Maxted. *Chem. Soc. Trans.*, 1918, **113**, 386–389.

In extension of the earlier investigation (compare this J., 1918, 105 r) it was found that by submitting a current of hydrogen (3 vols.) and nitrogen (1 vol.) inside a capillary glass tube to the action of an induction discharge modified so as to form a small high-tension arc, it is possible to obtain at atmospheric pressure a yield of ammonia amounting

to 1.5% of the gaseous mixture treated. The effect of induction sparks as such is feeble, and for the energetic production of ammonia it is necessary to bring the electrodes sufficiently close together to produce a small high-tension arc accompanied by a visible and apparently continuous flame of high temperature.—D. F. T.

*Sulphur dioxide and ammonia; Oxidation of — in the presence of platinum and rhodium.* P. Wenger and C. Urfer. *Ann. Chim. Analyt.*, 1918, **23**, 97–104.

When a mixture of sulphur dioxide and oxygen is passed over heated platinum black, maximum oxidation occurs at 432° C., 96.8% of the dioxide being converted into trioxide, whilst with rhodium black the maximum oxidation (91.1%) takes place at 610° C. Ammonia is oxidised to the extent of 97.7% by platinum black at 562° C., but the maximum conversion by rhodium black is only 69.7% at 662° C.; the products in both cases are nitrous and nitric acids.—W. P. S.

*Potassium carbonate; Causticising —.* E. Belloni. *Annali Chim. Appl.*, 1918, **9**, 115–149.

The method of preparing potassium hydroxide by treating potassium carbonate with lime, which prior to the war had been largely superseded by the electrolytic process, is again being used extensively, and in Italy potassium carbonate obtained by calcining molasses is treated by this method. Increasing the proportion of lime materially beyond the amount required to convert the potassium carbonate does not yield any increase in the proportion of alkali, and may cause a considerable loss. For example, in works where an excess of 140% of lime was used there was a loss of 25.65%. Provided there is efficient agitation of the mixtures, e.g., by means of steam, the reaction proceeds rapidly without excess of lime. Treatment of the potassium carbonate solution under pressure (3 to 4 atmos.), as claimed in several patents, has no influence upon the final reaction, although it enables the process to be effected at 125° to 150° C., and thus accelerates the velocity of reaction. The concentration of the potassium carbonate solution should not exceed 12%, in order that the quantity of unconverted potassium carbonate may not exceed 5%. The loss frequently noted in the process is to be attributed, not to the formation of a double carbonate of calcium and potassium, which cannot exist under the working conditions, but to the excess of calcium oxide generally used, which mechanically carries down and retains some of the alkali. Temperature has no influence on the conditions of equilibrium, at all events in solutions of medium concentration. The best results are obtained at 100° C., care being taken to keep the volume of the solution constant. Under these conditions the filtered product after concentration to 40° B. (sp. gr. 1.384) ought not to contain more than 1 to 1.5% of unaltered potassium carbonate. The chief impurities in potassium hydroxide prepared in this way are potassium carbonate, potassium chloride, and traces of silica, iron, and aluminium. If the solution at 40° B. (sp. gr. 1.384) be concentrated to 47° to 48° B. (sp. gr. 1.483–1.498) and cooled, crystals of the hydrate KOH.2H<sub>2</sub>O mixed with part of the potassium carbonate are obtained, whilst the potassium chloride and other impurities remain in the mother liquors. As a rule, a single crystallisation is sufficient to remove the whole of the potassium chloride. The crystals are washed with a little water, dissolved in water, the solution treated with barium hydroxide to convert the potassium carbonate into hydroxide and filtered, and the filtrate concentrated in silver vessels. This new method gives better

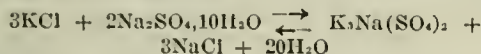


results than the old method of purification with alcohol followed by treatment with barium hydroxide. (See also J. Chem. Soc., July, 1918.)

—C. A. M.

**Mixtures of salts; Equilibria in solutions containing —.** 1. *The system water and the sulphates and chlorides of sodium and potassium.* W. C. Blasdale. J. Ind. Eng. Chem., 1918, 10, 344–347.

The conditions which govern the separation by fractional crystallisation of potassium salts from other salts frequently associated with them were investigated by the aid of the phase rule diagrams for such solutions. The equilibrium conditions of the reversible reaction

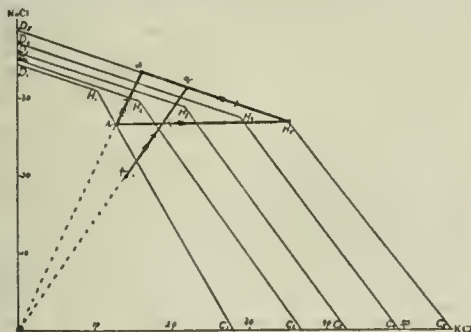


were studied, and the compositions of the solutions saturated with one or more of these salts at 0°, 25°, 50°, 75°, and 100° C. were plotted with respect to four axes representing sodium sulphate, potassium sulphate, potassium chloride, and sodium chloride, the ordinates being in mols. of salt per 1000 mols. of water, for solutions saturated at the given temperatures with each of the constituents. The diagram for 25° C. is very similar to that previously given by Meyerhoffer (Z. physik. Chem., 1899, 28, 453) and the diagrams for 50°, 75°, and 100° C. are closely similar and differ from that for 25° C. in the way that would be expected from the known changes in the solubility of the four simple constituent salts. The diagrams indicate the composition of all possible solutions which can be in equilibrium with the six salts, sodium sulphate, sodium chloride, sodium potassium sulphate (glaserite), Glauber's salt, potassium chloride, and potassium sulphate. All these diagrams indicate a very large field representing the composition of the solution with which solid glaserite is in equilibrium. (See also J. Chem. Soc., July, 1918.)

—W. F. F.

**Sodium and potassium chlorides and sulphates; Separation of — by fractional crystallisation.** W. C. Blasdale. J. Ind. Eng. Chem., 1918, 10, 347–353.

The application of the principles described in the preceding abstract to the separation of mixed solutions is shown. As an example, illustrated by the diagram, the points D<sub>1</sub>–D<sub>5</sub> represent the compositions of solutions saturated with sodium chloride at 0°, 25°, 50°, 75°, and 100° C., C<sub>1</sub>–C<sub>5</sub> represent the composition of solutions saturated



with potassium chloride, and H<sub>1</sub>–H<sub>5</sub> that of solutions saturated with both salts. The lines D<sub>1</sub> H<sub>1</sub>, etc., represent solutions saturated with sodium chloride with varying amounts of potassium chloride, and C<sub>1</sub> H<sub>1</sub>, etc., solutions saturated with potassium chloride with varying amounts of sodium chloride. If an unsaturated solution represented by p is evaporated at 100° C. it will become saturated

with sodium chloride at the point q, and this will be deposited till the saturation point, H<sub>5</sub>, for potassium chloride is reached. On cooling to 0° C. potassium chloride only will separate till the point r is reached. If this is removed and the solution then evaporated at 100° C. sodium chloride will again separate when the point s is reached. This cycle may then be repeated. The results of working at other temperatures may also be calculated from the diagram, and the relative efficiencies compared. Similar diagrams are given for mixtures of potassium chloride and potassium sulphate, of potassium sulphate and sodium sulphate, of sodium sulphate and sodium chloride, and of sulphates and chlorides of sodium and potassium. These diagrams indicate possible cycles for the efficient commercial separation of the salts. The applications of the diagrams to the most efficient separation of potash from the ash of kelp and from a desert bribe are given in detail. (See also J. Chem. Soc., July, 1918.)—W. F. F.

**[Wood] ash leaching plant; Inexpensive —.** W. D. Turner and B. G. Nichols. J. Ind. Eng. Chem., 1918, 10, 374–376.

A NUMBER of wooden buckets perforated at the bottom are arranged in tiers of six buckets, the uppermost being a water reservoir, and a non-perforated bucket being provided at the bottom for the liquor. The bottom of each bucket is covered with about 3 in. of "excelsior," and a layer of cloth, and the buckets are then filled with wood ashes. Water is allowed to drip from the reservoir till the ash in all the buckets is saturated, and a measured quantity of water equal to half the weight of ash in one bucket is then placed in the reservoir and allowed to drip through the column. An equivalent amount of liquor is forced out at the bottom and the ash and filters then removed from the top bucket and fresh ash substituted. This bucket is then placed at the bottom, the others being raised a step and water equal to 1.5 times the weight of ash placed in the reservoir and allowed to drip through. The operation is repeated as many times as desirable. The liquor, without concentrating, is boiled with the requisite amount of lime and allowed to settle. The lime mud is removed, and spread over the ash in the lowest bucket so that any soluble potash is subsequently leached out. The potash in the liquor may alternatively be converted directly to sulphate by the addition of sulphuric acid. The apparatus is simple, cheap, and rapid, and about 99% of the available potash is recovered.—W. F. F.

**Carbon dioxide in carbonates; Determination of — by Dittrich's method.** B. von Horvath. Chem.-Zeit., 1918, 42, 121.

IN this method borax is heated at 1000° C., cooled, the carbonate is added, and the mixture re-heated at dull redness until evolution of carbon dioxide ceases; the loss in weight gives the amount of carbon dioxide present. The method is rapid and suitable for the determination of carbon dioxide in sodium carbonate and barium carbonate. Allowance must be made for any water or other volatile substance in the carbonate. (See also J. Chem. Soc., July, 1918.)—W. P. S.

**Reactions between solid substances.** L. H. Parker. Chem. Soc. Trans., 1918, 103, 396–409.

By investigating the velocity of reaction of barium sulphate, silver nitrate, and cuprous chloride respectively with sodium carbonate at different temperatures, it was found that at the point of fusion a marked increase is observable in the rate of reaction. This result indicates that the increased



reactivity of substances in the fused state is due to the existence of at least one of the reagents in the liquid state and is not merely a result of the high temperature necessary to cause fusion. The interaction of the ingredients of solid binary mixtures when these are submitted to shearing stress, *e.g.*, by means of a mortar and pestle (Parker, this J., 1914, 770), is therefore probably correctly attributed to local surface fusion of the reacting substances under the mechanical strain.

—D. F. T.

*Sodium gold chloride; Adsorption of — by charcoal and the estimation of gold in sea water.* H. Koch. *Kolloid Zeits.*, 1918, 22, 1–22.

GOLD is readily adsorbed from dilute solutions of gold chloride by various commercial forms of carbon. Experiments were made with animal charcoal, wood charcoal, coke, Indian ink, and lamp-black with results which show that adsorption occurs even when the quantities of gold in solution are extremely small. Results obtained with solutions of varying concentrations of gold chloride in 3% sodium chloride solution can be satisfactorily represented by means of the ordinary adsorption formula, and since wood charcoal was found to be the most suitable adsorbent, this material was employed in the experiments with sea water. Results obtained with samples of sea water from the Adriatic and the Riviera show that gold is present to the extent of 2.5 to 4 mgrms. per cubic metre. Although the author's results agree with those recorded by certain previous observers, it should be noted that gold concentrations of the order of 50 mgrms. of gold per cubic metre have been found by Liversidge. (Compare *J. Chem. Soc.*, July, 1918.)—H. M. D.

*Thionyl and sulphuryl chlorides; Action of — on sulphur and phosphorus.* H. B. North and J. C. Thomson. *J. Amer. Chem. Soc.*, 1918, 40, 774–777.

WHEN heated in a sealed tube at 150°–180° C. thionyl chloride reacts with sulphur in accordance with the equation:  $2\text{SOCl}_2 + 3\text{S} = 2\text{S}_2\text{Cl}_2 + \text{SO}_2$ . Sulphuryl chloride reacts similarly and at a lower temperature (100°–125° C.), sulphur monochloride and sulphur dioxide being formed:  $\text{SO}_2\text{Cl}_2 + 2\text{S} = \text{S}_2\text{Cl}_2 + \text{SO}_2$ . Thionyl chloride reacts with both red and yellow phosphorus at 150°–180° C. in accordance with the equation:  $4\text{SOCl}_2 + 2\text{P} = 2\text{PCl}_3 + 2\text{SO}_2 + \text{S}_2\text{Cl}_2$ . Sulphuryl chloride under similar conditions reacts according to:  $3\text{SO}_2\text{Cl}_2 + 2\text{P} = 2\text{PCl}_3 + 3\text{SO}_2$ . By prolonged heating in presence of excess of thionyl or sulphuryl chloride, the phosphorus trichloride is oxidised in accordance with the equations:  $3\text{PCl}_3 + 4\text{SOCl}_2 = 3\text{PCl}_3 + 2\text{SO}_2 + \text{S}_2\text{Cl}_2$ ,  $\text{PCl}_3 + \text{SO}_2\text{Cl}_2 = \text{PCl}_3 + \text{SO}_2$ . Since the product of the reaction with thionyl chloride yields hydrogen sulphide on treatment with water, it seems that reaction takes place to some extent according to the equation:  $3\text{PCl}_3 + \text{SOCl}_2 = \text{PCl}_3 + \text{POCl}_3 + \text{PSCl}_3$ .—H. M. D.

*Bromine; Method of extracting — from the mother liquors of marine salt works.* G. Magnanini, G. Bianchi, and G. A. Venturi. *Annali Chim. Appl.*, 1918, 9, 109–113.

A PLANT for the direct extraction of bromine, without preliminary concentration of the mother liquors, has been installed in the marine salt works at Corneto Tarquinia, where the mother liquors in the salt pans have a density of 15° B. (sp. gr. 1.116) and contain about 1 gm. of sodium bromide per litre. A measured current of air is pumped into the lower part of a stoneware tower communicating at the top with a horizontal tube, which is slightly inclined, and is surrounded by a jacket through

which passes cold mother liquor to cool the liberated bromine vapour. This tube is connected with an iron tube containing iron turnings, and the aqueous solution of iron bromide here formed is collected in a bottle at the outlet of the tube. A current of slightly acidified chlorine water is introduced into the tower from an overhead tank, and mixes with a current of heated mother liquor. The cold mother liquor receives a preliminary heating by passing through a system of double leaden pipes, the inner pipe of which conveys from the tower the hot liquid from which the bromine has been removed. The mother liquor is then conducted through a copper coil, where it is heated to over 80° C. before entering the tower. The liberated bromine is expelled by the current of air and forced into the tube of iron turnings above the tower. The solution of iron bromide is neutralised with sodium carbonate and evaporated to dryness, leaving a black granular mass from which boiling water will extract a white salt consisting mainly of sodium bromide with a variable amount of sodium chloride. The latter may be removed by boiling the salt with water in only sufficient quantity to dissolve the sodium bromide.—C. A. M.

*Concentration of potash from raw materials containing only a trace of this element by means of the electrical precipitation of flue dust and fume from cement kilns.* Erdahl. See IX.

*Iron-silicon alloys unattacked by acids.* Matignon. See X.

*Use of "mine run" phosphates in the manufacture of soluble phosphoric acid.* Waggaman and Wagner. See XVI.

*Contributions to gravimetric analysis. Determination of chlorides, bromides, and iodides.* Winkler. See XXIII.

#### PATENTS.

*Sulphuric acid; Process of producing nitrous gases in lead chambers for the manufacture of —.* R. Vetterlein, Schöningen. Ger. Pat. 303,557, Feb. 26, 1916.

THE hot gases passing from the roasting furnace to the Glover tower are made to act upon potassium nitrate or nitrite, or other nitrate or nitrite, or mixtures of such salts, preferably by spraying aqueous solutions of the salts into a tower through which the gases are passed. The resulting nitrous gases pass into the lead chamber, whilst the non-volatile constituents of the salts subside in the reaction chamber, but it is advisable to interpose a dust-catcher to prevent solid particles entering the Glover tower.—C. A. M.

*Gases containing nitrogen oxides; Prevention of the premature formation of nitric acid in apparatus used for cooling —.* J. Straub, Bochum. Ger. Pat. 304,002, July 18, 1915.

SULPHUR dioxide or trioxide or substances which yield those compounds on heating are introduced into the hot gases. The sulphuric acid which is thus formed and deposited on the cooling surfaces, unlike nitric acid, has but little action on metals such as wrought iron.—C. A. M.

*Acetic acid; Preparation of concentrated from dilute —.* Akt.-Ges. für Anilinfabrikation. Ger. Pat. 302,673, June 11, 1915.

DILUTE acetic acid is dehydrated by mixing it with kleserite,  $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ , and a solvent such as chloro-

form, di- or tri-chloroethylene, or benzene, the anhydrous solution is removed from the solid mass, and distilled.—J. C. W.

*Nitre cake in a disintegrated or ground form; Method for the obtaining of —.* J. Grossmann, Manchester. Eng. Pat. 115,340, July 17, 1917. (Appl. No. 10,286 of 1917.)

At a certain critical temperature—about 50° C. for nitre cake containing 27% of free sulphuric acid, but varying with the content of free acid—nitre cake may be disintegrated by crushing or grinding more readily than under ordinary conditions. Accordingly, solidified or molten nitre cake is heated or cooled to the required degree and then disintegrated, the finer grinding being effected at or near atmospheric temperature.—W. E. F. P.

*Pyrophosphoric acid and acid pyrophosphates; Manufacture of —.* B. Levin, Hale, Chester. Eng. Pat. 115,693, May 16, 1917. (Appl. No. 7014 of 1917.)

To render pyrophosphoric acid and acid pyrophosphates stable and non-hygroscopic, suitable boron compounds (e.g., boric anhydride and pyrophosphoric acid) are incorporated with them during or after manufacture.—W. E. F. P.

*Zinc sulphate solutions [containing arsenic and antimony]; Method of purifying —.* R. B. Caples, Great Falls, and F. F. Frick, Anaconda, Mont., Assignors to Anaconda Copper Mining Co. U.S. Pat. 1,263,852, Apr. 23, 1918. Date of appl., Sept. 4, 1917.

A solution of zinc sulphate, containing a dissolved copper salt in the proportion of at least one part by weight of copper for each part of arsenic or antimony trioxide to be eliminated, is treated at about 80° C. with metallic zinc, whereby arsenic and antimony are precipitated with the copper.

—W. E. F. P.

*Bauxites, kaolins and other alumina-containing minerals; Method for the preliminary treatment of —.* A. Prager, Leipzig. Ger. Pat. 299,072, June 8, 1915.

The method of introducing the cold bauxite, etc., into the hot or cold alkaline flux effects only partial decomposition in the case of certain kinds, notably Hungarian bauxites. More rapid and complete decomposition is effected by roasting the bauxite and introducing it while hot into the flux.

—C. A. M.

*Salt and other substances; Process for preventing — from becoming damp and lumpy.* P. Immerwahr, Berlin. Ger. Pat. 303,449, Nov. 22, 1916.

Amorphous silicic acid is added to salt for this purpose. It absorbs more moisture than other addition agents without the formation of lumps and is tasteless, without smell, and not injurious to health.—H. J. H.

*Hydrogen peroxide; Process for obtaining stable compounds of —.* E. Merck Chem. Fabrik, Darmstadt. Ger. Pat. 303,680, Sep. 23, 1911.

Dilute solutions of commercial hydrogen peroxide are evaporated with neutral inorganic or organic substances (e.g., urea) to obtain stable compounds without the necessity of concentrating or purifying the hydrogen peroxide solution.—C. A. M.

*Sulphurous acid; Process of preparing reduction products of —.* Chem. Fabr. von Heyden A.-G. Ger. Pat. 304,107, June 6, 1914.

REDUCED iron or other form of iron containing little or no carbon is used in a finely divided condition for the preparation of hydrosulphites and sulphoxylates. The resulting solutions are free from iron, and better yields are obtained than when iron turnings, etc., containing carbon are used for the reduction.—C. A. M.

*Method of operating gas producers [with recovery of ammonium chloride].* Eng. Pat. 101,220. See IIA.

*Concentration and purification of sludge acid.* U.S. Pat. 1,263,950. See IIA.

*Process of decomposing alkali-containing minerals [for production of cement with volatilisation of the alkali].* Ger. Pat. 304,080. See IX.

*Electrolytic cells particularly adapted for the electrolysis of salt or similar material.* Eng. Pat. 114,974. See XI.

*Treatment of nitrate of ammonia.* U.S. Pat. 1,263,363. See XVI.

## VIII.—GLASS; CERAMICS.

*Refractoriness of firebricks, etc.; Effect of load on the —.* J. W. Mellor and W. Emery. Report of Refractory Materials Research Committee of Inst. Gas Engineers. Gas J., 1918, 142, 478—481.

To determine the refractoriness of firebricks under load, a test-piece is cut from the brick and ground to a rectangular block  $3\frac{1}{2}$  in. long and 2 in. cross section so as to present a surface of 4 sq. in. to the load. The block must be accurately measured and its top and bottom surfaces must be parallel. The test-piece is placed centrally and level on a solid refractory support, covered with a thin layer of powdered carborundum, in a modified Hirsch electric furnace. Small Seger cones, arranged in series of four, are fixed on the support around the test-piece. A cube of  $2\frac{1}{2}$  in. side, of similar material to the support, is placed on top of the test-piece and, above this, a cylinder of magnesite and finally, a carbon rod mounted on an iron ring, the rod being attached to a lever-arm. As the temperature of the test-piece rises, its thermal expansion causes the lever-arm to rise, but later it begins to fall, the rate of deformation depending on the character of the test-piece. When the pre-arranged limit has been reached, the current is switched off, the weights, lever, etc., removed, and the cones examined. It was found impossible to have the inside and outside of the test-piece at the same temperature, but if the heating is arranged so that Seger cone 16 bends in four hours, the difference between the interior and exterior of the test-piece will be about 20° C. Tests of 20 fireclay bricks, which lost their shape without load at Seger cone 26—33, were made under a load of 50 lb. per sq. in. at Seger cones 14 to 26, most of the samples deforming steadily throughout the heating. Similar tests on six silica bricks which, without a load, lost their shape at Seger cone 29—34, were made under the same load at cones 3 to 31, four of the pieces collapsing suddenly, and the other two more steadily. The coarser-grained bricks gave the best results. The worst result (collapse at cone 3 under load) was obtained with a soft, red silica brick of fine, even



grain which has given good results in practice in settings of gas retorts. A commercial magnesia brick which, unloaded, had a refractoriness above cone 40, deformed slightly and collapsed at cone 18 when loaded with 50 lb. per sq. in. A German chromite brick behaved similarly at cone 13–14, and a zirconia brick with a normal refractoriness greater than cone 40 was deformed at cone 26–27 with a load of 25 lb. per sq. in., at cone 15–16 at 50 lb. per sq. in., and at cone 14–15 at 75 lb. per sq. in. The authors consider that a load of 50 lb. per sq. in. is too severe for fireclay bricks, and suggest one of 20–30 lb. The collapse of firebricks under load appears to be of two kinds: fusion collapse due to the gradual melting of the matrix, and mechanical collapse of an abrupt nature. The former predominates in fireclay, alumina, and zirconia bricks and the latter in silica, magnesia, and chromite bricks. A firebrick containing silica-grog may not show any difference from a brick made without grog when heated without a load but, when loaded, the former will show a higher result. The variations in refractoriness under working conditions are partly due to the different temperatures at various parts of the brick. One brick examined was found to have a steady temperature of 1000° C. at the hot end, 720° C. at a point  $3\frac{1}{2}$  in. nearer the cool end, 570° C. at  $4\frac{1}{2}$  in. from the hot end, and 350° C. at  $6\frac{1}{2}$  in. from the hot end. If a brick is not inclined to spall, the softening of the hot face appears to do no harm. Some of the least resistant bricks when re-fired at cone 15–16 for two hours became much more resistant (cone 14 as compared with cone 9).—A. B. S.

*Silica bricks; Influence of iron and lime as agglomerants in the manufacture of* — M. Bied. *Comptes rend.*, 1918, 166, 776–778.

The addition of 3, 4, and 5% of iron oxide respectively to a silica brick mixture did not induce the formation of a network of tridymite in the absence of lime, and no agglomerating action was observed. The inclusion of iron oxide in a silica brick mixture does not appreciably lower the fusibility of the brick, an addition of 3%  $\text{Fe}_2\text{O}_3$  and 1% lime depressing the fusibility by only 5° C., which is within the error of determination. A ferruginous brick had expanded 1.8% after several days' exposure in an acid steel furnace and was sound, but the quartz grains had lost their individuality. —C. A. K.

*Silica bricks; Composition of* — after use in a Martin furnace. E. Rengade. *Comptes rend.*, 1918, 166, 779–781.

EXAMINATION of silica bricks from the crown of a Martin furnace shows that considerable changes have taken place, one of the most important of which is the conversion of quartz into cristobalite and tridymite (Le Chatelier, this J., 1917, 963, 964). Changes in chemical composition of different parts of the brick were studied after the brick had been subjected to the action of the furnace dust, and from the examination of a large number of such bricks the author differentiates four distinct zones. (A) The portion of the brick which has been immediately in contact with the flames, and is glazed, or at times has partially fused with the formation of stalactites. It is apparently homogeneous in structure, sometimes honeycombed, and grey in colour. (B) A black or dark grey section beneath A, usually distinctly separated from the latter, homogeneous, and of considerable hardness. (C) A transition zone often showing the larger grains of unconverted quartz in a black matrix, which latter recedes into a clear brown melt showing up the heterogeneous structure of the original brick.

(D) The remaining portion of the brick, which is apparently unchanged. Microscopic examination shows large transparent tridymite crystals in section B surrounded by an opaque black material, while in A these crystals are rounded by fusion but still distinct from the black matrix. As the cooler portion of the brick is approached tridymite occurs in diminishing quantity until the normal structure of the original brick is reached. Tabulated analyses of the different sections of silica bricks after use in acid and basic steel furnaces show considerable variations in the quantities of iron in the regions A and B and also in its state of oxidation, but generally B is richer in iron than A, and this can be explained by the black ferruginous constituent of the molten zone A moving into the region B between the crystals of tridymite under the influence of capillarity. A similar explanation would account for the transference of lime, as a fusible silicate, into the zone C. The presence of a considerable quantity of iron oxide does not appreciably affect the fusibility of silica bricks; even highly impregnated bricks stand well under furnace conditions.—C. A. K.

*Silica; Action of iron oxide on* — H. Le Chatelier and B. Bogitch. *Comptes rend.*, 1918, 166, 764–769.

THE necessary temperature for the production of steel is approximately 1650° C. and the fusibility of silica bricks rarely reaches 1750° C. Comparatively small increases in the temperature during steel making are therefore liable to cause fusion of the furnace lining. This difficulty is accentuated by the action of particles of iron which assist in the fusion of the crown of the furnace. The authors' results confirm those obtained by Rengade (see preceding abstract) and they use his classification of the differently heated regions A, B, and C, of silica bricks after use in a steel furnace, though preferring a division of zone C into two separate zones, as the yellow matrix differs from the black matrix in chemical composition. In the example of the different chemical composition of the zones given below, the figure for  $\text{Fe}_2\text{O}_3$  includes about 0.5%  $\text{Al}_2\text{O}_3$ .

	Brick prematurely fused, impregnated 9 cm. in depth.			Brick after long use, impregnated to a depth of 18 cm.		
	Sulphates	$\text{Fe}_2\text{O}_3$	CaO	Sulphates	$\text{Fe}_2\text{O}_3$	CaO
Original brick	10.5	1	2	8.5	1.5	1
Zone C ..	29.5	4.5	5	22	5.0	1.5
Zone B ..	29.5	4.5	5	26	6.5	2
Zone A ..	15	3.5	1.5	18	4.5	1.5

The grey zone directly in contact with the flame and exposed to the ferruginous particles contains less basic oxides than the zones further removed from the atmosphere of the furnace. The brown and yellow zones are much higher in lime, the increase being more marked by longer exposure of the brick in the furnace. Laboratory experiments indicated that the penetration of iron oxide is due to capillarity and by heating (to 1600° C.) a piece of the ferruginous brick on which was placed a similar piece of non-ferruginous brick, penetration of iron oxide was observed in the upper piece to a depth of 5 mm. The penetration of iron oxide into a silica brick heated in an oxidising atmosphere was not observed up to 1400° C., but reducing conditions caused considerable attack at 1200° C. The lessening of the proportion of basic oxide in the zone heated directly by the flame is due to the contraction of that portion of the brick, whereby the porosity is reduced and the ferruginous slag expelled into the cooler regions.

A briquette cut from the region B contracted 5% when subjected to a temperature of 1600° C. for one hour, whereas the original brick before contamination with iron oxide expanded 5% under the same conditions owing to the transformation of quartz into silica of lower density. This contraction becomes greater with increase in temperature even though the tridymite crystals expand under this treatment. In practice this contraction is readily observed, the face of the brick contracting (up to 10%) and falling away in a state of semi-fusion. If by faulty management of the furnace complete fusion occurs, the surface of the brick drips into the furnace with complete collapse of the crown, unless the conditions are altered. The aluminosilicates of calcium in the original brick are carried in advance of the ferruginous slag and concentration of lime occurs in the cooler regions. The region C containing 5% of lime would have a fusibility in the neighbourhood of 1600° C., with a fusibility of 1700° C. for the corresponding zone A. An increase in the temperature above 1700° C. would melt away the surface zone, and the region behind would in turn be quickly destroyed. A silica brick impregnated with iron oxide is regarded as being in a kind of unstable equilibrium, and its successful working necessitates a careful judgment of the temperature in the furnace.—C. A. K.

*Constructional materials; Use of Brinell ball in testing [hardness of] —.* H. Le Chateller and B. Bogitch. *Comptes rend.*, 1918, 166, 840—843.

The authors have adapted the Brinell ball test to the measurement of the hardness of bricks, by laying thereon a piece of thin lead foil, 30 mm. square and 0.05 mm. thick, previously blackened by treatment with hydrogen sulphide in a faintly acid solution, dried, and smeared with vaseline, most of which is wiped off again so that a matt surface is produced. The ball polishes the surface of the metal with which it comes in contact and this facilitates accurate measurement of the diameter of the depression. Tests with lead and copper showed that the foil does not interfere with the size of the depression produced. The ball used is 17.5 mm. diameter and is applied for 1 min. under a pressure of 500 kilos. The results are much more concordant than those obtained in determining the crushing strength of bricks. The test is useful in controlling the manufacture of bricks as it will show notable differences in the hardness of different parts of the same brick, due to irregularities in making or burning, or to an insufficient taper in the mould.—A. B. S.

#### PATENTS.

*Graphite crucibles, bricks, and other refractory articles having graphite as a base; Baking —.* Soc. Nouvelle des Etablissements E. Muller, Ivry-Port, France. Eng. Pat. 109,247, June 14, 1917. (Appl. No. 8536 of 1917.) Under Int. Conv., Aug. 29, 1916.

CRUCIBLES or other refractory objects having graphite as a base are piled up in a kiln of existing type and are burned in direct contact with the flames and products of combustion instead of being placed in muffles or saggars. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 3289 of 1871.)—A. B. S.

*Furnaces or kilns for calcining limestone, fireclay, and other refractory materials.* E. J. W. Richards, Glasgow. Eng. Pat. 115,316, June 7, 1917. (Appl. No. 8126 of 1917.)

A RECTANGULAR kiln is provided with hinged charging doors at the top and discharging doors at the

bottom along each side. Along the bottom of the kiln is a flue of approximately triangular cross section, with holes through which air is forced into the kiln so as to secure complete combustion of the fuel mixed with the material to be calcined. As the heating proceeds, the contents of the kiln descend and finally fall towards the discharging doors, through which they are removed from time to time.—A. B. S.

*Kilns; Continuous or series —.* T. West, Durham. Eng. Pat. 115,696. (Appl. Nos. 7068, May 17, and 13,925, Sept. 27, 1917.)

A NUMBER of round or rectangular up- or down-draught kilns of the customary type are connected by damper-controlled flues so that all the kilns may be worked with either up- or down-draught or the kilns may be worked up- and down-draught alternately.—A. B. S.

*Porous masses and objects; Process for the preparation of —.* N. V. Nederlandsche Gist- en Spiritusfabriek, Delft, and H. G. Monkhorst, Rijswijk, Holland. Ger. Pat. 304,854, Feb. 18, 1917.

THE gas-developing properties of yeasts or other micro-organisms in the presence of nutrient media are utilised as a means of obtaining a porous structure in objects, e.g., moulds and the like.

—A. de W.

*Kiln.* U.S. Pat. 1,263,620. *See I.*

#### IX.—BUILDING MATERIALS.

*Potash; Concentration of — from raw materials containing only a trace of this element by means of the electrical precipitation of flue dust and fume from cement kilns.* B. F. Erdahl. J. Ind. Eng. Chem., 1918, 10, 356—359.

IN a cement plant using limestone and blast-furnace slag, with powdered coal as fuel, a series of analyses of the limestone, slag, coal ash, clinker, and precipitated dust were made to determine the potash content. It was found that the potash content of the limestone and clinker was constant, that of the slag varied from 0.28 to 0.69%, that of the coal ash varied from 1.20 to 1.52% and the potash in the precipitated dust increased with the number of electric precipitators used. The charge becomes richer in potash as the dust is continuously returned to it. About 30% of the total potash remains in the clinker and 70% is volatilised. About 30% of the volatilised potash is deposited in the rear end of the kilns, the housings, and gas coolers, and less than 2% is lost through the stacks. The analyses of the returning dust from the precipitators gave a decreasing potash content for successive cycles. The deposited dust in the rear end of the kilns increased in amount with each cycle, indicating that choking would eventually occur. The potash content of the dust escaping through the stacks decreased for successive cycles.—W. F. F.

*Influence of iron and lime as agglomerants in the manufacture of silica bricks.* Bied. *See VIII.*

*Use of Brinell ball in testing [hardness of] constructional materials.* Le Chateller and Bogitch. *See VIII.*



## PATENTS.

*Shaft-furnaces for burning cement, lime and similar substances.* A. and W. W. Steiger, Zürich, Switzerland. Eng. Pat. 109,266, Aug. 28, 1917. (Appl. No. 12,370 of 1917.) Under Int. Conv., Aug. 30, 1916.

A ROTARY hearth, fitted with a stepped helical surface and two groups of cutting edges, is placed in the lower part of a shaft furnace to form an automatic discharging device.—A. B. S.

*Kilns for burning cement, lime, or the like.* A. Steiger, Zürich, Switzerland. Eng. Pat. 111,854, Nov. 27, 1917. (Appl. No. 17,543 of 1917.) Under Int. Conv., Dec. 4, 1916.

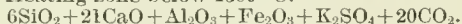
COMPRESSED air is passed through a series of ducts in the walls of a shaft kiln and thence through numerous adjustable openings into the interior of the kiln. The gaseous products of combustion are drawn off by an exhaustor connected to openings near the top of the kiln. The air supplied through the conduits close to the sole of the kiln is at a higher pressure than that supplied through the conduits nearer the burning zone.—A. B. S.

*Alkali-containing minerals; Process for decomposing* — [for production of cement with volatilisation of the alkali]. F. Krupp A.-G. Grusonwerk, Magdeburg-Buckau. Ger. Pat. 304,080, Apr. 9, 1914.

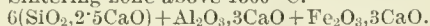
THE mineral is heated with calcium carbonate and the calcium salt of another acid, and in certain cases with a corrective agent—e.g., silica—so as to obtain simultaneously the total alkali and a cement. The calcium salt of the other acid must be added in the proportion necessary for combining with the alkali, and if the mineral does not contain sufficient iron to form about a quarter of the weight of the total sesquioxides present, a corresponding quantity of ferric oxide must be introduced. Under these conditions the decomposition is practically quantitative at a lower temperature than is required for the formation of clinker. In the case of a mixture of feldspar, chalk, calcium sulphate, and ferric oxide, the reaction takes place in two stages—



(1) Heating zone below 1300° C.



(2) Sintering zone above 1300° C.



—C. A. M.

*Furnaces or kilns for calcining limestone, fireclay, and other refractory materials.* Eng. Pat. 115,316. See VIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Hematite; Concentration of silicious red* —. J. T. Singewald, jun. U.S. Bureau of Mines, Bull. 110. 84 pages.

THE Birmingham district of Alabama possesses large beds of ferruginous sandstone, consisting of red hematite with varying percentages of silica and other impurities. Where the bed has not been leached by surface water, the ore is hard and ranges in composition from 32 to 45% Fe, 5 to 20% CaO, 2 to 25% SiO<sub>2</sub>, 2 to 5% Al<sub>2</sub>O<sub>3</sub>, 1 to 3% MgO, 0.25 to 1.5% P, and up to 0.5% S. The value of the ore depends to a large extent on the approximation to a self-fluxing character, that is, on the relative quantities of SiO<sub>2</sub> and CaO. In the concentration

tests the ore was crushed to pass a 20-mesh screen, and a screen analysis was then made (by the Tyler standard) ranging from particles of 0.833 mm. diam. to particles of less than 0.074 mm., the latter being regarded as slime. The heavier particles high in iron were separated by the use of "Thoulet solution" of sp. gr. 3.0, and by the combined processes each sample yielded seven sizes of concentrates and of tailings, besides the slime. 39 samples were examined in this way and the detailed results are given. The hard ores cannot be profitably worked if the content of SiO<sub>2</sub> exceeds that of CaO by more than 8%, and by concentration the quality of the concentrates can be made at least equal to that of ores which at present are successfully worked. In the case of an extremely poor sample from the Ruffner mine, with silica in excess of the iron, averaging 28.8% Fe and 30.9% SiO<sub>2</sub>, the concentrates averaged 40.5% Fe and 17.1% SiO<sub>2</sub> for example. Of the total iron in the crude ore, in all the samples tested, that in the concentrates averaged 80.6% and that in the slimes 13.1%, making a recovery of 93.7%. The average composition of the concentrates was 46.4% Fe and 13.4% SiO<sub>2</sub>, and of the slimes 44.7% Fe and 13.5% SiO<sub>2</sub>. The method of concentration employed would not be feasible in commercial working but as a definite concentration of iron is indicated, the devising of a separation on commercial lines may be possible.

—C. A. K.

*Carbon; Rapid determination of total* — in steel and cast iron. E. Sernagiotto. Annali Chim. Appl., 1918, 9, 113—114.

FROM 1 to 3 grms. of the finely-divided metal is treated with 50 c.c. of a 30% aqueous solution of the double chloride of copper and potassium (CuCl<sub>2</sub> + 2KCl) acidified with a few drops of hydrochloric acid, the reagent being added in one portion with constant shaking. As soon as the iron has dissolved, the beaker is heated and the solution filtered with the aid of suction through an asbestos filter contained in a perforated copper thimble resembling a Gooch crucible. The filter is washed with a little of the reagent acidified with hydrochloric acid and then with water, and the thimble dried at 100° C. The carbon is then determined by combustion in a current of oxygen.—C. A. M.

*Iron alloys; Influence of the chemical composition and heat treatment of — on their magnetic properties, specific resistance, and density.* Gumlich. Chem.-Zeit., 1918, 42, 200—201.

THE density and specific resistance of the alloys investigated containing iron with carbon, silicon, aluminium, and manganese, generally vary in a regular manner with alteration in the percentage of the added elements, so that it is possible, with good agreement with the results of direct measurement, to calculate from the figures for the alloys the corresponding values for pure iron. Irregularities in the course of the curve showing the relation between these physical properties and the composition are encountered only with alloys containing a high percentage of carbon or manganese, the latter at a proportion of 8—10% and the former at the point at which cementite may be formed in a matrix of pearlite or martensite. Analogous irregularities are observed in the curves for coercive force and saturation value, the latter having its maximum for pure iron. The view that the addition of silicon or aluminium improves the magnetic properties of iron is therefore erroneous, this belief having arisen from the effect of these elements in reducing the oxygen content and in preventing the presence of the deleterious dissolved carbon. Aluminium and silicon also possess the technically important characteristic of increasing

the specific electrical resistance and thereby reducing the tendency to eddy currents. Manganese, on the other hand, checks the separation of dissolved carbon and is therefore undesirable. All four ingredients cause a reduction in the true remanence of iron. Electrolytic iron after being heated and then cooled slowly, exhibits high remanence and permeability whereas after quenching the remanence and permeability are slight; on repeated heat treatment the hysteresis loops gradually become more oblique and narrower; rolled sheet iron, however, does not exhibit this behaviour. Manganese and quenched carbon alloys possess several analogies; both show a simultaneous increase in the coercive force and decrease in the remanence when the percentage of the characteristic elements is raised; in order to obtain high coercive force and high remanence simultaneously it is necessary to introduce tungsten, chromium, or molybdenum. Unlike the higher manganese alloys, the carbon alloys on quenching from above 700° C. become magnetically harder than when cooled slowly.—D. F. T.

*Iron-silicon alloys unattacked by acids.* C. Matignon. Comptes rend., 1918, 166, 815–818.

VARIOUS iron-silicon alloys produced in different countries in 1913 were compared with one another and with samples of ferrobiron and Borchers' metal. The specimens examined had the following compositions:—

	French	Italian		American		
	Métallure	Ellanite I	Ellanite II	Ironac	Duriron	Ferrobiron
Si ..	16.92	15.07	15.13	13.16	15.51	4.9
Fe ..	81.05	82.40	80.87	83.99	82.23	69.8
Mn ..	0.88	0.62	0.53	0.77	0.66	3.3
Ni ..	—	—	2.23	—	—	—
Al ..	0.25	—	—	—	—	3.1
C ..	0.592	—	0.82	1.08	0.83	—
P ..	0.173	—	0.06	0.78	0.57	—
S ..	0.01	—	0.03	0.05	0.01	—
Ca and Mg	—	—	—	—	—	—
B ..	—	—	—	—	—	15.4
Sp. Gr. ..	6.71	6.87	7.14	6.71	6.94	—

Borchers' metal was prepared from the following mixture: Ni 64.6; Cr 32.3; Ag 0.5, Mo 1.8 parts. As regards resistance towards nitric acid the ferrobiron did not resist the action of the acid either weak or strong. Contrary to the general rule Borchers' alloy was more resistant to the weak acid (20° B., sp. gr. 1.16) than to the strong acid (36° B., sp. gr. 1.33), but it did not show any advantage over the iron-silicon alloys, which are cheaper. The introduction of 2.0% of nickel improved the resistance of the alloy to acid as between Ellanite I and II. Métallure gave the best results, it being a homogeneous alloy with regular texture and apparently more carefully refined. None of the alloys resisted the action of hydrochloric acid. (See also Tungay, this J., 1918, 87 T.)

—W. G.

*Copper melts containing gases; Effect of tin on —.* W. Stahl. Metall u. Erz, 1918, 15, 67.

Tin separates from molten copper in an oxidising atmosphere as tin dioxide, or as a stannate if other metals are present. After the commencement of the reaction between cuprous oxide and sulphide, the tin reacts with the former according to equation:  $2\text{Cu}_2\text{O} + \text{Sn} = 4\text{Cu} + \text{SnO}_2$ . When sulphur dioxide is present in solution the following reaction occurs:  $\text{SO}_2 + \text{Sn} + 2\text{Cu} = \text{SnO}_2 + \text{Cu}_2\text{S}$ . The addition of 1.3% of tin raises the specific gravity of refined copper from 8.625 to 8.9135. Small quantities of tin added to copper act as a deoxidiser similarly to phosphor-copper.—F. C. Th.

*Copper; Influence of the temperature of rolling, the amount of work, and annealing on some properties of —.* P. Oberhoffer. Metall u. Erz, 1918, 15, 47–56.

THE material used was intended for locomotive fire-boxes and had the following composition: 99.46% Cu, 0.28% Sn, 0.231% As, 0.11% O. Specimens quenched in water after rolling possessed similar properties to those cooled in air. In the case of samples hot rolled at temperatures between 300° and 800° C., the increase of breadth was relatively large while the elongation was small, but above and below these temperatures the broadening decreased and the elongation increased.

*Influence of heating to the temperature of rolling.* In the case of the cast metal, the tensile strength and hardness after the cooling down altered very little as the temperature to which the samples were heated was raised. The elongation and the reduction of area remained practically constant to 800° C. but then decreased. The structure of the cast metal showed dendritic crystals which appeared bright under oblique illumination, and were low in arsenic and oxygen. The arsenic and the cuprous oxide eutectic were concentrated between the branches of the dendrites rendering the crystals much finer grained. The crystal size did not much affect the strength of the cast metal, but influenced the external appearance of the broken tensile test-pieces. Annealing did not change the crystal size of the cast copper nor did it produce equilibrium in the arsenic content by diffusion. In the case of copper which had been previously reduced 85% in thickness by rolling, the strength and hardness were hardly influenced at all by subsequent heating to the rolling temperature, the tensile strength showing a reduction of only 2 kilos. per sq. mm. after heating to 1000° C. The elongation and reduction of area fell as the temperature was raised to 200° C., then increased, and remained constant from 300° C. to 500° C. A rapid rise occurred between 500° and 600° C., when the values remained constant to 900° C. after which a fall again set in. The structure of the metal showed no correlation with these changes, being more in agreement with the strength and hardness curves. The crystal size was almost constant till an increase occurred from 600° C. to 1000° C. In spite of the large amount of work done upon the specimens, traces of the original dendritic structure were clearly visible, and the parallelism of the zones rich in cuprous oxide and arsenic gave a banded structure analogous to that of hot-rolled structural steels. The increase of strength (7 kilos. per sq. mm.) and hardness (10) compared with the cast and unworked metal, is due not only to the change in the crystal size but also to that in the arrangement of the oxide- and arsenic-rich areas.

*Influence of the temperature of rolling.* In the case of cast copper, the hardness and tenacity fell slowly as the temperature was increased to 500° C. and then more quickly to 700° C., remaining almost constant to 900° C., after which a further fall occurred. The elongation and reduction of area increased rapidly from 600° C. to 700° C. and no decrease from 900° C. to 1000° C. was observed. Between 600° C. and 900° C. the resistance to impact increased rapidly. The structure of the pieces rolled up to 600° C. showed distinct elongation of the crystals, but between 600° C. and 700° C. a sudden change in structure occurred which was clearly expressed in the elongation curve. Above 700° C. the crystals were no longer elongated. Above 900° C. a noteworthy increase in the coarseness of the crystallisation occurred. In the case of previously worked material the curves were similar to the foregoing, but the impact value rose more sharply and the increase occurred not between 600° C. and 700° C. but between 400° C. and 500° C.



The elongation curve showed a rise between 400° C. and 500° C., but only attained its maximum at 700° C. Further, elongation of the crystals was observed only up to 400° C., thus clearly showing that the smaller crystals of the worked metal need a distinctly lower temperature to produce recrystallisation than do the coarser ones of the cast material. With the worked copper rapid crystal growth did not occur till 900° C., but at 1000° C. the crystallisation in the cast and worked samples was almost identical. The elongation, however, of the worked metal was 20% higher than that of the cast. In this material the tensile strength is not determined solely by the crystal size, the amount of work exerting a considerable influence.

*Influence of the rolling:*—The curves of both the cast and previously worked materials were similar, but the hardness and strength in the latter case were consistently higher than in the former. Below 650° C. the rolling operations correspond to "cold-rolling" and above that temperature to "hot-rolling." Annealing at 650° C. after rolling at temperatures from 600° C. to 1000° C. produced no improvement. For a reduction of 85% 500° C. is the lowest temperature that can be used, and 600° C. is better; 800° C. should not be exceeded as an upper limit. At 900° C. the structure, tensile and impact properties deteriorate and a sudden change in the relations of broadening to elongation during rolling occurs. The influence of the extent of the rolling upon the mechanical properties is summarised in the following table:—

Reduction %	Maximum stress, kilos per sq. mm.	Elongation, % on 100 mm.	Reduction of area, %	Hard- ness
0	17.0	18.0	18.0	46
50	21.5	27.0	27.0	63
92.5	23.0	48.0	49.0	60

—F. C. Th.

*Oxygen in copper; Estimation of* — P. Oberhoffer. Metall u. Erz, 1918, 15, 33–35.

AFTER briefly reviewing other methods suggested for the determination of oxygen in copper, a modification of Hampe's method (Z. anal. Chem., 1874, 13, 188) is described, whereby the time required is reduced to  $\frac{3}{4}$  hour and the necessary apparatus is simplified. The apparatus consists of an electrolytic hydrogen generator with purifiers, a mercury air-pump, an electric oven with silica tube in which the sample is heated, and a phosphorus pentoxide guard-tube. A temperature of 800° C. is used, and 5 minutes is sufficient for the complete reduction of the cuprous oxide present, the complete estimation taking 45 minutes.—F. C. Th.

*Zinc blende and some other minerals; Magnetic properties of* — F. Stutzer, W. Gross, and K. Bornemann. Metall u. Erz, 1918, 15, 1–9.

MANY zinc blendes are sufficiently magnetic to be attracted by strong magnetic fields. The authors have devised a method by which the susceptibility of such materials can be measured and have carried out determinations on 19 samples of zinc blende and on several other minerals. Minerals having susceptibilities less than  $20 \times 10^{-6}$  are practically unmagnetisable, and to this class belongs pure zinc blende in addition to gangue materials such as quartz, calcite, fluorspar, and barytes. The presence of iron in zinc blende increases the susceptibility, though not in any definite manner; with 14.7% Fe the susceptibility is over  $500 \times 10^{-6}$ . Manganese, nickel, and cobalt also increase the susceptibility as in the case of copper ores and salts. Some measurements are also given for ferro-magnetic minerals such as magnetite, but these are less reliable.—F. C. Th.

*Zinc; Electrothermal production of* — H. Nathusius. Metall u. Erz, 1918, 15, 87–93, 108–111.

ATTEMPTS made hitherto to apply electric heating to the extraction of zinc from its ores have, in the opinion of the author, been unsuccessful. The principal cause lies in the fact that electric furnaces hitherto constructed have not solved the problem of the condensation of the zinc vapour to liquid metal. In most cases a high percentage of zinc dust is formed which must be remelted, rendering the process uneconomical. No resistance furnace has previously been constructed which will attain the high temperature, 1200° C. to 1300° C., which is required, and the arc type of furnace must be entirely excluded since, apart from other disadvantages, at the temperature of the electric arc the temperature and pressure relations of the reduction are completely changed and cannot be allowed for.

The author has designed a furnace in which the following points have been considered:—The furnace must be gas-tight during reduction, distillation, and condensation; the gases formed must be at once removed; the heating must be gradual and uniform throughout the charge; the temperature reached should not exceed 1300° C., and the process should be continuous.

The furnace is described in detail and illustrated. It consists essentially of a shaft, preferably vertical, in which the reduction by carbon occurs. Zinc vapour distils off through side flues into a condensing chamber maintained at 500° C., from which the molten zinc is tapped off. The heating is produced by the current introduced through cast steel electrodes built into the walls. An alternating current at from 5 to 15 volts is used. The whole furnace is cased in an iron sheath and is gas-tight. The zinc vapour and carbon monoxide are drawn into the condenser by an exhaust pump. The charge is preheated (by the hot products of the reaction) to about 900° C. in a conical continuation of the reducing chamber. The heating is claimed to be uniform and very efficient. In a test a charge of 1000 kilos. of ore, containing 64% ZnO, and 267 kilos. of coal was treated with an expenditure of 920 kilowatt-hours. The cost is claimed to be little more than that of the gas-heated muffle furnace, while advantages are obtained in the directions of purity of metal, uniformity of heating, economy of space and labour, the possibility of working poor ores containing lead and copper, economy in carbon and refractory materials, smaller loss of metal, and simplification of transport.

—F. C. Th.

*Tin; Recovery and losses of* — at Giew Mill.

Cornish Sub-Committee, Tin and Tungsten Research Committee. Bull. 164, Inst. Min. and Met., May, 1918. [Advance copy.] 11 pages.

THE mill was run for 71 days under the supervision of the Sub-Committee, in order that exact data on the losses in milling might be obtained. The tin recovered in the concentrate was 67.5%, while the direct determination of the loss in the effluent gave 35.5%. The original ore contained 1.4% SnO<sub>2</sub>.

—W. R. S.

[Tin.] *Giew Mill test. Elutriation of the crushed products.* S. J. Truscott. Inst. Min. and Met., Bull. 164, May, 1918. [Advance copy.] 6 pages. (See preceding abstract.)

THE battery feed, the tubemill feed and tailing, and the final tailing and concentrate were elutriated, and the products assayed. The bulk of the tin was found in the coarser sizes of the feed (down to -200 mesh), proving that cassiterite does not suffer undue comminution in crushing and re-grinding. More than two-thirds of the concentrate was coarser than 300-, and nearly one-half coarser than 200-mesh.

—W. R. S.

*White metals; Analysis of* —. P. Drawe. Z. angew. Chem., 1918, 31, 88.

THE approximate composition of white metals may be ascertained as follows. One gram. of the sample is decomposed with nitric acid, the solution is diluted and boiled, and the hydrated tin and antimony oxides are collected on a filter. The mixed oxides are then heated at 80° C. for 1 hour with hydrochloric acid and iron dust, cooled, the precipitated antimony collected on a filter, and the stannous chloride in the filtrate titrated with ferric chloride solution. The antimony precipitate is washed with dilute hydrochloric acid until free from iron, ignited, and weighed. The filtrate from the tin and antimony oxides is treated in the usual way with sulphuric acid to separate the lead, copper is precipitated as sulphide in the filtrate from the lead sulphate, iron is precipitated with sodium hydroxide after oxidation with bromine, and zinc as carbonate.—W. P. S.

*Ore flotation; Molecular physics of* —. W. H. Coghill and C. O. Anderson. J. Phys. Chem., 1918, 22, 237—255.

THE principles involved in the flotation of solids in liquids are discussed. Methods of constructing liquid profiles are considered, and a formula is derived by means of which angles of contact may be calculated from cathetometric measurements of the rise or fall of liquids in contact with vertical plates. The connection between flotation and the angle of contact is indicated by curves. Measurements of the water profile in the case of floating steel and aluminium wires and of floating galena are recorded.—H. M. D.

*Composition of silica bricks after use in a Martin furnace.* Rengade. See VIII.

*Action of iron oxide on silica.* Le Chatelier and Bogitch. See VIII.

*Estimation of copper as copper oxide after precipitation as thiocyanate.* Fenner and Forschmann. See XXIII.

*Use of metallic silver as a reducing agent in the volumetric estimation of iron.* Edgar and Kemp. See XXIII.

#### PATENTS.

*Iron; Protecting surfaces composed of or containing — from chemical action.* D. Relehnstein, Zürich, Switzerland. Eng. Pat. 103,814, Nov. 14, 1916.

ESTERS of arsenious or antimonious acid, dissolved in oil, or in organic solvents, by means of which they are incorporated with oil or fat, are applied to the surface to be protected, or the esters may be added directly to the water which comes into contact with the metal surface, e.g., in a boiler.

—T. H. B.

*Iron or other magnetic metals; Process for obtaining coatings of — by the spraying process.* A. Wolf, Hamburg. Ger. Pat. 301,971, June 6, 1917.

OBJECTS to be coated with iron or the like are magnetised during the process, whereby the coating adheres more firmly.—F. C. Th.

*Phosphatic slags; Method of producing high-grade — in the manufacture of iron or steel from high-sulphur pig iron.* B. Queling, Saarbrücken. Ger. Pat. 302,283, Nov. 11, 1916. Additions to Ger. Pat. 301,839.

CAST iron from a mixer is, while being transferred to open-hearth furnaces, opposed by a blast of compressed air. This prevents the slag, high in silica and sulphur, and the light iron-sulphur alloy which is supposed to float on the surface of the iron in the mixer, from entering the second furnace, where a new, undiluted and highly phosphoric slag can be produced.—F. C. Th.

*Iron articles; Process for introducing nitrogen into* —. J. Pintsch A.-G., Berlin. Ger. Pat. 302,306, Sept. 24, 1916.

THE iron articles are carried on an endless chain into a chamber in which collects the hydrogen produced by the decomposition by heat of ammonia. The hot gas preheats the objects which are carried on into a hot zone of decomposing ammonia in which surface nitrogenation occurs. From here the chain carries the charge into colder nitrogen after which the articles are quenched in a water bath which acts as a gas seal.—F. C. Th.

*Charcoal iron; Method for preparing a substitute for* —. Rombacher Hüttenwerke, J. I. Bronn, and W. Schemmann, Rombach. Ger. Pat. 302,358, Oct. 5, 1915.

ORDINARY cast iron is refined in a basic-lined Bessemer converter or in a basic open-hearth furnace until the impurities are removed (e.g., carbon to about 0.1%). The liquid ingot iron is then brought into intimate contact with 6—7% of its weight of charcoal, when, if the temperature is kept sufficiently low and the duration of the process is not excessive, a satisfactory substitute for charcoal iron may be produced.—F. C. Th.

*Steel; Method for the oxidation of the phosphorus before the carbon in the manufacture of — by the basic Bessemer process.* W. Gontermann, Siegen. Ger. Pat. 302,768, Apr. 5, 1917.

By using a high-pressure blast in the basic lined Bessemer converter, the oxidation of the phosphorus is accelerated and that of the carbon delayed and with a certain pressure the order of oxidation of the two elements can be reversed. When the phosphorus has been removed the blast pressure can be lowered, when at the prevailing high temperature the carbon is rapidly oxidised. It is possible to decarburise to any desired extent.

—F. C. Th.

*Ferro-chrome; Process for manufacture of very low-carbon* —. Stahlwerke R. Lindenberg, Remscheid-Hasten. Ger. Pat. 302,675, Feb. 5, 1916.

A HIGH-PERCENTAGE ferro-silicon is melted in a reducing atmosphere in an electric furnace and covered with a mixture of commercially pure chromium oxide and lime. Ferro-chrome containing up to 80% Cr can easily be produced starting from a correspondingly high-percentage ferro-silicon, and a 98% ferro-chrome has been prepared.

—F. C. Th.

*Ferro-alloys; Method of producing low-carbon* —. Stahlwerke R. Lindenberg, Remscheid-Hasten. Ger. Pat. 302,862, Feb. 5, 1916. Addition to Ger. Pat. 302,675. (See preceding abstract.)

INSTEAD of chromium oxide technically pure oxides of tungsten, molybdenum, vanadium, titanium,



cobalt, and boron may be added (together with lime) to the molten ferro-silicon to produce the corresponding ferro-alloys.—F. C. Th.

*Galvanising articles of iron; Process for hot — after heating them in liquid lead.* F. Köster, Hamm. Ger. Pat. 302,268, July 30, 1916.

Iron wires are heated to 750° C. in a lead bath and transferred quickly to a bath of molten zinc.

—F. C. Th.

*Alloys; Manufacture of metal —.* Stabilimenti "Biak" A. Pouchain, Assignees of P. Peynetti, Turin, Italy. Eng. Pat. 111,288, Nov. 5, 1917. (Appl. No. 16,129 of 1917.) Under Int. Conv., Oct. 23, 1916.

Two or more of the constituents of an alloy are melted in a chamber wherein one or more of the other constituents exist in the state of vapour.

—T. H. B.

*Copper or other metals or compounds thereof; Extraction of — from ores.* J. S. Ross and G. L. Crump, London. Eng. Pat. 115,088, Apr. 25, 1917. (Appl. No. 5835 of 1917.)

Ore containing small quantities of the metal as carbonate (e.g., copper carbonate) is subjected, in lump form as mined, to the leaching action of a 5 to 10% solution of sulphuric acid, or of a substance containing sulphuric acid, e.g., nitre cake. The metal is then obtained from the solution by electrolysis or other known method.—C. A. K.

*Copper; Process for extracting — from its ores.* E. Anderson, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,263,727, Apr. 23, 1918. Date of appl., Aug. 15, 1916.

COPPER ore containing a carbonate is leached with a solution containing a ferrous salt, cupric chloride, and a chloride capable of retaining cuprous chloride in solution; copper is precipitated from a portion of the solution thus obtained and the solution then subjected to oxidising action sufficient to convert the cuprous salt to cupric, whilst leaving a substantial portion of the iron salt in the ferrous state.—T. H. B.

*Copper alloys; Method for the improvement of —.* P. Goldstein, Hannover. Ger. Pat. 302,962, Dec. 24, 1912. Addition to Ger. Pat. 208,657 (this J., 1914, 201).

Alloys of copper and zinc, tin, or aluminium containing small proportions of high melting-point metals such as titanium, vanadium, uranium, zirconium, tungsten, chromium, or molybdenum, are prepared as follows:—A briquette of very finely divided uranium or other oxide is reduced with molten sodium in a crucible and brass turnings are added, the melt stirred well, and cast at the highest possible temperature. The alloys are very ductile and have a high resistance to chemical action.

—F. C. Th.

*Furnaces; Electric — for melting or refining non-ferrous alloys.* V. C. Faulkner, Sheffield. Eng. Pat. 115,248, Feb. 2, 1917. (Appl. No. 1674 of 1917.)

The crucible of the furnace is surrounded by a mass of granular carbon, with adjustable electrodes projecting through the sides of the outer walls of the furnace, and a third electrode is fixed under the bottom of the crucible. An upstanding central hollow boss may be arranged on the floor of the furnace chamber for supporting a crucible smaller than the containing chamber, the cavity on the underside of the boss being filled with a heating medium, such as coke or broken electrodes.—B. N.

*Furnace; Combined tilting and reversible open-hearth melting — and converter.* A. Rollason, Nottingham. Eng. Pat. 115,323, June 16, 1917. (Appl. No. 8653 of 1917.)

The furnace has a cylindrical body which rests upon, and may be rotated in, a cradle provided with trunnions, the latter being mounted in bearings in hangers or pedestals. The body is lined with acid and basic material on opposite sides; has tuyères and ports for the admission of gas or oil and air (or, alternatively, electrodes); and is fitted with a circumferential toothed metal rack operated by spur and worm gear. One of the trunnions on the cradle is also provided with spur and worm gear for the purpose of tilting the furnace.—W. E. F. P.

*Crucible furnaces.* J. Connolly, Birmingham. Eng. Pat. 115,543, July 19, 1917 (Appl. No. 10,396 of 1917.)

THE furnace may comprise a single chamber with double walls, the air traversing the heated annular channel, and passing through slits in the inner wall into the combustion chamber; or, primary and secondary chambers may be connected by a flue, the secondary chamber being heated by the fume gases from the primary chamber, and serving as a pre-heating furnace. The space between the two chambers can be used as a regenerator, the air for the combustion of the fuel being conveyed through pipes which are contained in this space and which are heated by the waste gases from the secondary furnace. Alternatively, a combination of the two forms may be employed.—C. A. K.

*Zinc; Extraction of —.* The New Delaville Spelter Co., Ltd., Birmingham, and E. H. Shortman, jun., Bloxwich. Eng. Pat. 115,409, July 19, 1917. (Appl. No. 10,385 of 1917.)

A zinc retort is connected with a condenser of large capacity by a passage provided with a lead-intercepting and filtering device. The condenser is provided with an opening through which gases escape during the first stage of the process, but is closed by a plug while the zinc vapour is passing over.

—T. H. B.

*Zinc ores, calcined pyrites, and other metallurgical products; Method of dead-roasting low-grade sulphur-bearing, specially —.* W. Buddéus, Berlin. Ger. Pat. 302,880, Oct. 5, 1916.

The ore is roasted in a vertical shaft furnace with a blast chamber below the grate. The process is continuous, new material being introduced as the calcined ore is removed.—F. C. Th.

*Ores; Treatment of —.* J. B. Vogelsang, St. Louis, U.S.A. Eng. Pat. 115,697, Mar. 17, 1917. (Appl. No. 7078 of 1917.)

A Mixture of crushed ore, water, and oil is forced into a chamber by other than mechanical means, e.g., by a steam jet, the outlet of the chamber being somewhat restricted, so as to cause thorough emulsification. The outlet of this chamber is connected with a separating tank. An inner chamber having perforated walls may be provided to assist the emulsification. The fine particles of metal in the pulp are enmeshed and retained in suspension in the froth, whilst the heavier waste material settles out.—C. A. K.

*Acid resisting metal, and articles made thereof.* A. E. Gillespie, London, and E. E. Sheather, Teddington. Eng. Pat. 115,750, Aug. 1, 1917. (Appl. No. 11,102 of 1917.)

A bar or plate of chromium alloy steel of the kind known as "stainless" or "rustless" steel (a low-

carbon steel containing 10 to 15% Cr) is welded to a bar or plate of ordinary mild steel, by hammering or rolling under the action of heat. The composite plate is then heated and rolled to any required thickness for the manufacture of culinary utensils or cans for the storage of foodstuffs, the resulting sheet having one inoxidizable surface. The mild steel may be coated on both sides, and is then particularly suitable for the manufacture of dental plates.—C. A. K.

*Float metal; Process and apparatus for the recovery of — from water containing the same.* J. H. Reed, Philadelphia, Pa. U.S. Pat. 1,262,984, Apr. 16, 1918. Date of appl., July 25, 1917.

WATER containing float metal is caused to flow under a layer of oil, and by means of baffle-plates the float metal is caused to impinge on the under surface of the oil, thereby being entrapped.

—C. A. K.

*Metals from ores; Process of recovering —.* H. E. Wood, Denver, Colo. U.S. Pat. 1,263,503, Apr. 23, 1918. Date of appl., May 11, 1916.

THE concentrate obtained by subjecting dry-crushed, non-oxidised ore to a film (tension) flotation process is heated to oxidise undesirable constituents, then mixed with water and oil, and again treated by flotation to recover the oiled, metalliferous particles.—W. E. F. P.

*Tungsten-reducing [electric tube] furnace.* C. A. Pfauhlehl, Waukegan, Ill., Assignor to Pfauhlehl Co., Inc., North Chicago, Ill. U.S. Pat. 1,263,598, Apr. 23, 1918. Date of appl., Oct. 15, 1915.

THE tungsten trioxide to be reduced is contained in nickel trays arranged one above the other, with intervening spaces for the passage of the hydrogen used as reducing agent. This portion of the furnace tube is surrounded by a heating element by means of which a constant temperature is maintained throughout the length of the reducing zone of the furnace, whilst at the inlet end the tube is provided with another electric heating element directly in contact with the hydrogen, by means of which the latter is heated before coming in contact with the tungsten trioxide.—T. H. B.

*Tungsten or molybdenum; Process of coating or joining articles of —.* F. A. Fahrenwald, Cleveland, Ohio, dedicated to the Government and people of the United States. U.S. Pat. 1,263,656, April 23, 1918. Date of appl., May 31, 1916. (Dedicated to the public.)

ARTICLES of tungsten or molybdenum or their alloys are coated by heating them in contact with a dry flux containing a finely-divided precious metal, mixed with an alkali salt, to a temperature at least as high as the melting point of the precious metal. The coated articles may be soldered by means of a metal or alloy which will alloy with the precious metal.—T. H. B.

*Composition [alloy] for resisting heat.* M. R. Wolfard, Cambridge, Mass., Assignor to L. A. Ames, Spencer, and E. E. Kent, Newton, Mass. U.S. Pat. 1,263,831, Apr. 23, 1918. Date of appl., Aug. 8, 1916.

FOR withstanding the action of an oxidising flame at high temperature, an alloy composed of iron, 2 and nickel, 1 to 4 parts, and containing relatively little carbon, is employed in the cast condition.

—W. E. F. P.

*Metal-bearing material for sintering; Method of preparing finely-divided —.* F. W. Yost, Assignor to American Ore Reclamation Co., New York. U.S. Pat. 1,263,832, Apr. 23, 1918. Date of appl., Feb. 27, 1915.

FINELY-DIVIDED, dump, metalliferous material, containing an excess of fuel in lump form, is screened to separate the larger lumps of fuel (exceeding half an inch in diameter) and to form a fine product containing sufficient carbon for sintering.

—W. E. F. P.

*Metals; Process and apparatus for the separation of —.* [Recovery of silver from photographic solutions.] Ges. für angew. Photographie m.b.H., Freiburg. Ger. Pat. 302,279, June 1, 1916.

FOR the recovery of metals, particularly of silver from photographic solutions, a precipitating metal is used in the form of balls or rollers with smooth surfaces. These are kept in continual frictional motion in the solution, which may be contained in a rotating apparatus or shaking vessel.—C. A. M.

*Pyrites cinder; Process for the desulphurisation, dezincification, and agglomeration of —.* G. Hentschel, Duisberg-Meiderich. Ger. Pat. 302,281, Jan. 16, 1917.

PYRITES cinder is desulphurised by heating it strongly in a rotary tube furnace. It then passes into a second similar furnace where it is mixed with carbon and heated by an air blast, the zinc being distilled. The hot gases from this tube circulating around the desulphuriser heat it and are themselves cooled. The residue is mixed with fine iron ore or blast-furnace or other similar dust and sintered to form porous blocks in a third rotating tube furnace.—F. C. Th.

*Silicon coatings; Method for producing — by treating the object to be covered in the hot state with halogen or halogen-hydrogen compounds of silicon.* Böhmische Elektrizitäts A.-G., Vienna. Ger. Pat. 302,305, Apr. 4, 1915.

IRON and other metals are protected from acid and other corrosion by formation of a surface layer of silicon or a silicon alloy. Red hot iron reacts with silicon tetrachloride, forming ferric chloride and silicon. Other halogen compounds of silicon may be substituted. Substances which do not react with the silicon compounds are first coated with a reactive substance.—F. C. Th.

*Tools with sharp edges; Annealing of —.* A. Pfreitzschner, Munich. Ger. Pat. 302,359, Nov. 19, 1915.

THE hydrogen given off from an electrolytic cell may if the tension is sufficiently great be hot enough to cause the metallic cathode to glow. If the tool to be annealed is made the cathode it can be raised to any desired temperature. The edges must be protected, preferably by a carburising coating, from the direct action of the hydrogen which would otherwise blunt them. The insulating coating allows only the unprotected parts to act as cathode, the edges being heated by conduction.

F. C. Th.

*Iron, copper, and brass; Method of preparing coatings [of bismuth] on metals, especially on —.* Chem. Fabr. auf Actien, vorm. E. Schering. Ger. Pat. 302,816, June 23, 1916.

THE metals are treated by any ordinary process, such as dipping, rubbing, or spraying, with a solution of bismuth iodide or bromide in the alkali iodides or bromides and the corresponding acids. A layer of bismuth is deposited in the cold as



matt deposit which is firmly adherent. The iodide solution is more satisfactory than the bromide in the case of copper.—F. C. Th.

*Etching of printing-plates; Process for the electrolytic* —. H. Strecker, Munich. Ger. Pat. 302,902, Mar. 31, 1916.

THE electrodes are separated by such a short distance as almost to produce short-circuiting, in order to produce an etched surface of almost mirror-like brilliance. The nearness of anode and cathode causes the etching to be a lattice pattern. The plate is carried in a movable support in the bath so that the cathode may occupy any desired relative position. The process is applicable to the etching of plates with or without a coating on the surface.—F. C. Th.

*Metallic objects containing fine pores; Process for increasing the density of* —. Berliner Maschinenbau A.-G. Ger. Pat. 302,934, Sept. 2, 1916.

THE objects are galvanised electrolytically under high pressure—greater than that to which the article will later be subjected. Under this pressure the electrolyte penetrates the pores as it would not do at ordinary pressures.—F. C. Th.

*Ore concentrating tables.* E. Trestrail, Redruth. Eng. Pat. 115,494, May 10, 1917. (Appl. No. 6672 of 1917.)

*Distilling metallic ores and other materials containing metals; Process for* —. D. B. Jones, Chicago, Assignee of C. H. Fulton and T. M. Bains, jun., St. Louis, U.S.A. Eng. Pat. 112,924, Aug. 10, 1917. (Appl. No. 11,499 of 1917.) Under Int. Conv., Jan. 24, 1917.

SEE U.S. Pat. 1,242,339 of 1917; this J., 1917, 1240.

*Sintering fine ores, fluc-dust, purple ore, iron filings and the like; Process for* —. C. Giesecke, Harzburg, Germany. Eng. Pat. 115,441, Oct. 4, 1916. (Appl. No. 14,059 of 1916.)

SEE U.S. Pat. 1,205,944 of 1916; this J., 1917, 89.

*Process for the fractional separation of tarry substances such as pitch or steel works tar and tar oils from superheated crude gases.* Ger. Pat. 303,195. See IIa.

*Rust-proofing composition and process of making same.* U.S. Pat. 1,263,395. See XIII.

## XI.—ELECTRO-CHEMISTRY.

*Electrical endosmose.* II. T. R. Briggs, H. S. Bennett, and H. L. Pierson. J. Phys. Chem., 1918, 22, 256–272.

AN electrical endosmometer is described which has been used in the measurement of the rate of flow of solutions through diaphragms of carborundum, alundum, powdered glass, and gelatin and agar-agar jellies. Experiments made with dilute solutions of sodium hydroxide and hydrochloric acid show that the endosmotic flow is towards the cathode in alkali and towards the anode in acid solution. This reversal occurs with diaphragms of carborundum and alundum and also with jelly diaphragms, but in the case of powdered glass the flow is towards the cathode in both acid and alkaline solution. The effects obtained with various copper salts are described and interpreted in terms of the adsorption of the ions by the diaphragm.

Observations made on acid and alkaline solutions of Eosin, Crystal Ponceau, Safranin and Methylene Blue are also described and discussed in reference to the adsorption theory of dyeing. (Compare J. Chem. Soc., July, 1918.)—H. M. D.

*Galvanic batteries; Restoration of exhausted* —. E. Stern. Z. Elektrochem., 1918, 24, 91–93.

EXAMINATION of exhausted Leclanché cells used in telephonic work and for flash lamps showed that the zinc containing vessel was practically worn out and the carbon electrode was covered with ammonium chloride crystals and in many cases disintegrated. On washing the carbon electrodes and placing them in a 20% paste of ammonium chloride and sawdust contained in a fresh zinc container, it was found that the repaired cells fall into three groups, (1) those with an EMF of above 1 volt, (2) those with an EMF of 0.8–1.0 volt, and (3) those with EMF below 0.8 volt. The two first groups of cells are suitable for further use.—J. F. S.

## PATENTS.

*Electrolytic cells particularly adapted for the electrolysis of salt or similar material.* N. Statham, Hastings-on-Hudson, New York. Eng. Pat. 114,974, Sept. 26, 1917. (Appl. No. 13,883 of 1917.)

AN electrolytic cell is composed of a concrete cell body with metallic cathode members extending into it, and connected to cathode plates substantially free from direct contact with the cement; strengthening stay rods are arranged at the outside of the cell to hold the members together whilst minimising undesirable expansion strains. Carbon anode posts, with their exposed portions protected by an insulating covering of vulcanised rubber, are built into the cell, and movable carbon anode plates are connected with the anode posts by means of double-tapered graphite pins, so that the plates are supported in close co-operation with the cathode diaphragms at the sides of the cell.—B. N.

*Electrolysis; Apparatus for* —. P. A. Emanuel, Aiken, S.C. U.S. Pat. 1,233,314 Apr. 16, 1918. Date of appl., Dec. 24, 1917.

AN electrolytic vessel comprises a container formed of two hemispherical sections with flanges connected together but insulated from each other; there is a perforated metallic disc in each section and a partition separates the sections and discs. The partition does not interfere with electrolysis, but serves to prevent the commingling of the evolved gases. Two substantially hemispherical tanks are arranged above the container, and are connected by valved pipes to the respective sections, with means for placing the container and tanks under pressure. The electric circuit includes the tanks, container sections, perforated discs, and electrolyte.—B. N.

*Furnaces; Electric — and transformers for use therewith.* The British Electric Transformer Co., Ltd., and F. E. Berry, Hayes, Middlesex. Eng. Pat. 115,247, Jan. 31, 1917. (Appl. No. 1588 of 1917.)

A THREE-PHASE electric furnace is provided with upper electrodes and lower laterally-arranged electrodes, so connected to the associated transformer, that not only is the order of the electrodes in the upper set the reverse electrically of that in the lower lateral electrodes, but each upper electrode is directly opposite a lower lateral electrode as seen in plan. Means are provided whereby the lower lateral terminals can, when desired, be temporarily short-circuited either at the furnace or at

the transformer windings. By this arrangement good distribution and control of the heat in the furnace can be ensured, together with thorough mixing of the charge.—B. N.

*Electrodes in electric furnaces; Preventing the oxidation or wasting of the —.* W. B. Hamilton, Sheffield, and J. Holland, Grindelford, Derby. Eng. Pat. 115,705, May 25, 1917. (Appl. No. 7519 of 1917.)

The oxidation or wasting of electrodes in electric furnaces is prevented by building an elongated keystone brick or block, made in one or several pieces, into the roof or walls of the furnace. The block is provided with a central passage, of a bore only slightly larger than the diameter of the electrode passing through the same, and is of sufficient length to enclose the glowing portion of the electrode when the latter is withdrawn from the furnace.—B. N.

*Furnaces; Electric —.* O. Sahlin, London. Eng. Pat. 115,719, June 13, 1917. (Appl. No. 8478 of 1917.)

The electrodes, arranged in one or more pairs, enter through the side walls, approximately opposite the charging door, and are carried by water-cooled boxes secured to the furnace by cotters. The upper electrode of each pair is inclined downwards, while the lower electrode is substantially horizontal, and both are in the same vertical plane. The furnace is heated at the commencement by free-burning arcs formed between the upper and lower electrodes, the inclined electrode of each pair being then advanced further into the furnace so that the free-burning arcs are forced towards the hearth. The charge is introduced, and when some of it has been melted, the furnace is tilted and some or all of the lower horizontal electrodes withdrawn, so that direct arcs are formed between the corresponding upper electrodes and the bath. When two- or three-phase alternating currents are employed, the horizontal electrodes are connected to a conducting plate at the bottom of the furnace, so placed that when current is conveyed by this plate, the voltage between the bath and an inclined electrode is usually about half the normal voltage between two inclined electrodes.—B. N.

*Gases; Treatment of — in electric furnaces.* E. Edwin, Assignor to Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. U.S. Pats. (a) 1,263,389 and (n) 1,263,390, Apr. 23, 1918. Dates of appl., Aug. 29, 1916, and Feb. 14, 1918.

(a) The hot gases from an electric arc furnace are made to expand continuously whilst passing to an absorption device, thus cooling them. The mechanical energy which is developed is utilised in a hot air turbine to drive a rotary compressor for compressing the gases to the pressure at which they are used in the arc furnace, and producing the circulation of the gases in the system. The arc is thus stabilised, and velocity and energy production are maintained in accordance with the furnace operation. (b) The gases are circulated in a closed system between an electric arc furnace and an absorber, the latter being maintained under a pressure higher than that of the furnace. The pressure of the gases is diminished between the absorption plant and the furnace, thereby transforming their heat energy into mechanical work in a hot air turbine, the latter driving a rotary compressor working approximately at the same difference of pressure as the turbine, and supplying the circulating gases to the

absorption plant. The cooled gases, after leaving the absorption plant, are preheated by the hot furnace gases before being compressed.—B. N.

*Anode.* H. R. Swartley, jun., Manhasset, N.Y., Assignor to Davis-Bourmonville Co., Jersey City, N.J. U.S. Pat. 1,263,959, Apr. 23, 1918. Date of appl., Feb. 25, 1916.

The anode is provided with a permanent continuous surface of sprayed nickel.—B. N.

*Electrolytic apparatus for generation of oxygen and hydrogen.* I. H. Levin, Newark, N.J., U.S.A. Eng. Pat. 107,366, Jan. 3, 1917. (Appl. No. 117 of 1917.) Under Int. Conv., June 3, 1916.

SEE U.S. Pat. 1,199,472 of 1916; this J., 1916, 1224.

## XII.—FATS; OILS; WAXES.

*Para rubber seed; Oil-content, keeping qualities, and commercial possibilities of —.* F. G. Spring and F. W. F. Day. Agric. Bull. Fed. Malay States, 1918, 6, 231–244.

The yield of rubber seed on Malay plantations approximates to 300 lb. per acre per annum; about 37% of the seed consists of shell, as compared with 22% of shell in the English chestnut and 16% in the acorn, and to produce a ton of kernels 427,000 seeds are necessary; the seeds, either whole or decorticated, can be stored satisfactorily in bags after a preliminary sun-drying. On extraction with petroleum ether the dried meal from the kernels and the whole seeds respectively yield approximately 51% and 29% of oil. The oil possesses rather less drying power than linseed oil but should easily find industrial application. Samples of the oil hitherto examined have had sp. gr. at 15°/15° C. 0.925–0.930, acid value 10.7–40.9, saponification value 188.5–192.1, and iodine value 128.3–143.3. The residual cake after the removal of the oil by pressing, is a useful cattle food although care is required in its use for immature animals.—D. F. T.

*Spinacene and some of its derivatives.* A. C. Chapman. Chem. Soc. Trans., 1918, 113, 458–466.

When the hydrocarbon spinacene from shark-liver oil is distilled over sodium, especially if the pressure is allowed to rise to 45 mm., partial decomposition occurs and a cyclodihydroterpene,  $C_{10}H_{18}$ , b.pt. 170°–175° C. at atmospheric pressure, sp. gr. at 20°/20° C. 0.8125, and refractive index 1.4565 at 20° C., can be separated from the decomposition products. Spinacene is therefore more suitably purified by distillation under a maximum pressure of 10 mm., when it is found to have the composition  $C_{20}H_{34}$ , b.pt. 260° C. (corr.) at 9 mm., sp. gr. at 15°/15° C. 0.8610, and refractive index 1.4965 at 20° C. Its hexahydrochloride melts at 126° C. and its hexahydrobromide at 132° C.; the former when heated under reduced pressure undergoes loss of hydrogen chloride with formation of a hydrocarbon quite distinct from the original spinacene. The formula  $C_{20}H_{30}$  previously attributed to spinacene (Chapman, this J., 1917, 392) is withdrawn.

—D. F. T.

*Resins; Rapid determination of — in soap.* V. Fortini. Annali Chim. Appl., 1918, 9, 102–108.

TWITCHELL's method of determining rosin in soap has the drawbacks that it shows an apparent rosin content with pure soaps, and that it is not applicable to resins other than colophony. These drawbacks are obviated in the following method, which



is based on the fact that nitrated resins are insoluble in petroleum spirit. Two grms. of the mixed fatty and resin acids from 25 grms. of the soap is treated in a separating funnel with 50 c.c. of petroleum spirit (b.pt. 40° to 70° C.), the presence of resin acids being indicated by incomplete solution. Ten c.c. of nitric acid, prepared by mixing 25 c.c. of fuming nitric acid (sp. gr. 1.52) and 75 c.c. of ordinary nitric acid (sp. gr. 1.48) and adding a few crystals of urea to destroy nitrous acid, is then introduced in successive small portions. The mixture is shaken for 2 to 3 mins., then allowed to stand, the acid layer drawn off, and the petroleum spirit shaken with an additional 5 c.c. of the nitric acid, which is also drawn off. The petroleum spirit layer is then washed successively with ordinary nitric acid, and with water and filtered, the filtrate evaporated, and the residue of fatty acids dried and weighed. The quantity of resin acids is obtained by difference. The results thus obtained agree within about 1% of theory in the case of mixtures of 5 to 20% of rosin with saturated fatty acids or oleic acid.—C. A. M.

*Oxidation products of paraffin wax.* Bergmann.  
See IIA.

*Fat content of conifer resin.* Schwalbe and Schulz. See XIII.

*Saponins [from mourah seed].* Spiegel and Meyer.  
See XX.

#### PATENTS.

*Soap [for removing stains caused by nitro compounds].* F. Huntington, Lancaster, and W. H. Barnes, Denton. Eng. Pat. 115,110, May 12, 1917. (Appl. No. 6761 of 1917.)

Sodium sulphite or other sulphite is incorporated with soap in a proportion of not less than 10% by weight, to obtain a preparation which will remove stains made by organic nitro compounds on the skin, fabrics, etc.—C. A. M.

*Soap; Process for filling — with vegetable substances.* C. G. Schwalbe, Eberswalde. Ger. Pat. 304,093, May 24, 1916.

VEGETABLE substances such as wood, stalks, fibres, bran, sawdust, etc., are treated with acids or acid salts, and then disintegrated mechanically in the presence of water to obtain a mucilage which can be used as a filling for soap.—C. A. M.

*Detergents from synthetic aromatic tannins, etc.* Badische Anilin und Soda Fabrik. Ger. Pat. 304,024, May 18, 1916.

SYNTHETIC aromatic tannins, especially those claimed in Ger. Pats. 262,558, 280,233, and 290,965, and also crystalline compounds which contain sulpho or carboxyl groups or both, and are able to precipitate glue or gelatin from their solutions, may be used as detergents for removing stains of ink, fruit, rust, etc., and fat from paper, textile fabrics, the skin, etc.—C. A. M.

*Magnetic separator [for oils].* U.S. Pat. 1,263,406.  
See I.

*Conversion of oils, resins, waxes, etc., and hygroscopic substances into dry products.* Ger. Pat. 303,122. See I.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Conifer resin; Fat content of —.* C. G. Schwalbe and W. Schulz. Chem.-Zeit., 1918, 42, 229—230.

PINE wood yields about 11.4% of resin containing 27% of fatty acids as determined by the Twitchell esterification method; the heart wood gives 16% of crude resin containing 3.5% of fatty acids, and the sap wood 2.4% of resin containing 1.2% of fatty acids.—W. P. S.

*Oil varnishes; Examination of —.* H. Smit-Addens. Olien & Vetten, 1917, 2, 23, 58, 80. Z. angew. Chem., 1918, 31, Ref., 142.

For the estimation of the volatile thinners a weighed portion is steam-distilled in a current of carbon dioxide. The distillate is transferred to a burette containing 10 c.c. of water, and the volume of thinners is read off, whilst the aqueous layer is examined for smell and acid reaction. The specific gravity of the distillate can be determined in the case of turpentine by the method of floating a drop in dilute alcohol, while in the case of petroleum spirit a pycnometer is employed. A quantitative examination (see below) is necessary since all other substitutes have a greater "thinning" power than petroleum spirit. To estimate the non-volatile constituents the condensed water is removed from the distillation flask as far as possible, 96% alcohol and excess of N/1 alkali are added, and the mixture is boiled under a reflux condenser for 20 minutes. The alcohol is then partly evaporated off and the liquid transferred to a separating funnel, the flask being rinsed out with petroleum ether and alcohol. The soap solution is extracted twice with petroleum ether and the extract evaporated, dried at 105° C. and weighed as copal resin. The soaps of the softer resins, Manila, Congo, and Brazilian copals are not so easily hydrolysed and these resins are estimated along with the rosin acids. In the case of Dammar resin, a portion is easily saponifiable whilst another is unsaponifiable, the latter being partly soluble in petroleum ether whilst the former is soluble in ether only. Should Dammar be suspected, the soap is further extracted with ether and the extract evaporated when a light coloured residue indicates its presence; extraction with chloroform or carbon tetrachloride will serve to separate copal. The extracted soap residue is then acidified with dilute nitric acid and shaken twice with petroleum ether, nearly neutralised with alkali, and examined for metals in the usual way. The acid petroleum ether containing rosin acids and fatty acids is examined qualitatively by the Storch-Morawski test and quantitatively by Twitchell's method. The hydroxy fatty acids which cling to the walls of the separating funnel are dissolved in a mixture of equal parts of alcohol and carbon tetrachloride and calculated to oleic acid. Examination of the aqueous layer and of the water from the initial distillation will determine the presence of volatile and soluble substances (phenol). In the case of films of paints and varnishes, a separation into pigment and vehicle is obtained by means of suitable solvents, when these constituents can then be examined.

The specific gravity of the volatile "thinners" (see above) is determined by introducing a drop into alcohol of about 70% strength (sp. gr. 0.87 at 15° C.). If the drop floats or sinks rapidly, it is safe to assume that the distillate is not pure turpentine, but if the distinction is not so sharp, addition of water or alcohol will permit of the specific gravity being determined. A second drop serves for the determination of the refractive index and if the result therefrom agrees with that from

the specific gravity and the smell is found to be normal, no further examination is necessary. In other cases Herzfeld's method is used to obtain a separation into turpentine, petroleum spirit, and homologues of benzene.—A. de W.

#### PATENTS.

*Titanic acid pigments resistant to chemical and physical agencies; Preparation of —.* Det Norske Aktieselskab for Elektrokemisk Industri Norsk Industri-Hypotekbank, Christiania, Norway. Ger. Pat. 303,458, Dec. 1, 1916. Under Int. Conv., Dec. 10, 1915.

TITANIC oxide or hydroxide containing phosphoric acid in the free or combined condition is mixed with the usual painters' pigments.—A. de W.

*Condensation product and method of preparing same.* K. Brown, Montclair, and D. S. Kendall, East Orange, Assignors to Condensite Co. of America, Bloomfield, N.J. U.S. Pat. 1,263,031, Apr. 16, 1918. Date of appl., Mar. 30, 1917.

A MIXTURE of a phenolic substance and a substance containing an active methylene group, in suitable proportions to form an infusible insoluble condensation product, is moderately heated in a vessel having a large cooling surface in relation to the amount of material. As soon as the reaction begins the source of heat is withdrawn and the reaction allowed to proceed spontaneously until heat ceases to be evolved, and an intermediate product liquid at the ordinary temperature is obtained. Prior to the application of heat a condensing agent consisting of a salt of calcium and a phenolic substance which will not react with the other ingredients may be introduced into the liquid mass.

—C. A. M.

*Finely subdivided material [for printing ink, rubber mixings, etc.]; Method of treating —.* G. C. Lewis, New York. U.S. Pat. 1,263,082, Apr. 16, 1918. Date of appl., Jan. 25, 1917.

FLOCCULENT or other finely divided material is treated with an inert liquid so as to form an emulsion or paste in which the liquid replaces the air between the particles, and this liquid is then removed by evaporation. A composition suitable for use in printing ink, rubber mixings, etc., may thus be obtained by mixing a solution or emulsion of an oily substance in a volatile liquid with carbon black or lampblack, and completely evaporating the liquid.—C. A. M.

*Rust-proofing composition and process of making same.* W. R. Emig, Assignor to H. S. Bergen, Toledo, Ohio. U.S. Pat. 1,263,395, Apr. 23, 1918. Date of appl., July 3, 1916.

BURUNDY pitch is melted, boiled China wood oil is added, and the mixture heated to boiling point. A mixture of finely divided iron and phosphoric acid is then added, and the mixture stirred till effervescence ceases.—W. F. F.

*Varnishing metal objects; Process of —.* O. Ruff, Breslau. Ger. Pat. 304,985, June 30, 1917.

THE metal objects are treated with aqueous solutions of phenols or cresols, together with formaldehyde and alkali, the water evaporated off, and the objects then submitted to a temperature of at least 80° C. for about 6 hours, to render the film insoluble in water and other solvents.—A. de W.

*Conversion of oils, resins, waxes, etc., and hygroscopic substances into dry products.* Ger. Pat. 303,122. See 1.

*Method and plant for the distillation of tar, crude petroleum, resin and similar materials.* Ger. Pat. 302,323. See III.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Oil-content, keeping qualities, and commercial possibilities of Para rubber seed.* Spring and Day. See XII.

#### PATENTS.

*Vulcanised rubber goods; Process of separating fibrous materials from —.* W. Golombek, Spandau. Ger. Pat. 303,171, June 24, 1916.

THE material is finely divided and stirred for a short time with slightly warm water; the fibres are wetted more easily and sink, whilst the particles of rubber float, and are skimmed off.—A. de W.

*Method of treating finely-subdivided material [for rubber mixings, etc.].* U.S. Pat. 1,263,082. See XIII.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Leather; Determination of free sulphuric acid in —.* H. Strunk and O. Matthes. Z. angew. Chem., 1918, 31, 61—63.

THIRTY c.c. of magnesium nitrate solution (containing about 0.2 gm. MgO) is heated in a round-bottomed flask until an almost dry residue remains; 5 grms. of the leather and a few c.c. of 30% nitric acid are added and the mixture is heated on a water-bath until reaction ceases. Fifty c.c. of concentrated nitric acid is then added, the mixture boiled on a sand-bath for 2 hrs., heated over a flame until all the nitric acid has been expelled, cooled, again heated with concentrated nitric acid, and evaporated. The dry material is strongly heated to decompose nitrates and expel nitrous fumes from the flask, and the almost white residue is dissolved in 30 c.c. of N/2 hydrochloric acid, filtered if necessary, and titrated with N/10 sodium hydroxide solution, using phenolphthalein as indicator. Another portion of 30 c.c. of the magnesium nitrate solution is evaporated, heated to decompose the nitrate, the residue dissolved in N/2 hydrochloric acid, and titrated with sodium hydroxide solution. Less sodium hydroxide solution will be required in this titration than in the case of the residue from the mixture containing the leather if free sulphuric acid is present; the sulphuric acid combines with a portion of the magnesium oxide, and the difference in the two titrations is a measure of the quantity of free acid present.—W. P. S.

*Gelatin; Stoichiometrical character of the action of neutral salts on the swelling of —.* J. Loeb. J. Biol. Chem., 1918, 34, 77—95.

THE limiting concentrations of neutral salts which just inhibit the additional swelling of gelatin, sodium gelatinate, or gelatin chloride (compare this J., 1918, 274 a), are dependent on the valency but not on the nature of the ions. The limiting inhibiting concentration of salts with univalent ions is twice as high as that of salts with divalent ions. Antagonistic salt action occurs when gelatin salts with univalent ions are transformed into salts with divalent ions. (See further J. Chem. Soc., July, 1918.)—H. W. B.



*Gelatin solutions; Influence of neutral salts on the viscosity of —.* J. Loeb. J. Biol. Chem., 1918, 34, 395—413.

THE viscosity of solutions of gelatin which has been previously treated with neutral salts is affected in a manner similar to that observed in the case of the swelling effect previously recorded (see preceding abstract). In the experiments with gelatin and sodium gelatinate, the viscosity is increased by cations, whilst with gelatin chloride only the anion has any effect. (See further J. Chem. Soc., July, 1918.)—H. W. B.

*Gelatin; Effects of electrolytes on — and their biological significance. IV. Precipitation of gelatin by mixtures of salts.* W. O. Fenn. J. Biol. Chem., 1918, 34, 415—428. (Compare this J., 1918, 216 A, 274 A.)

THE amount of sodium chloride or sulphate required to precipitate gelatin from a solution containing an acid, alkaline, or neutral salt besides gelatin, depends on the nature of the ions which are present. Acids promote precipitation of the gelatin whilst alkalis usually have the reverse effect. (See further J. Chem. Soc., July, 1918.)—H. W. B.

#### PATENTS.

*Impregnating material for leather, leather substitute, fabrics, etc.; Manufacture of —.* F. Nathö, Hamburg. Ger. Pat. 302,158, Apr. 14, 1915.

BURGUNDY, coumarone or similar resins are heated with animal fats, and when the mass is brought to a certain consistency and temperature, sulphur is added for the purpose of vulcanisation, and then the mixture is allowed to cool and suitably diluted. This filling material is especially good for leather, which it impregnates quickly and renders permanently waterproof and flexible. Equally good products are obtained with leather that is not fully tanned, and poor leathers can be improved so as to be equal in value for many purposes to the best grades.—J. C. W.

*Waterproof [fibrous] composition and process for making such composition [from spent bark].* U.S. Pat. 1,263,823. See V.

### XVI.—SOILS; FERTILISERS.

*Soils; Mechanical and physical analysis of —.* G. Richter. Bied. Zentr., 1918, 47, 49—52.

IN the mechanical examination of soils, preliminary treatment, such as ignition, oxidation with bromine, etc., should be avoided as it destroys the colloids and alters the character of the soil. Kopecky's titration method appears to be the best of the mechanical methods. As regards methods for determining the hygroscopic character of soils, it is found that Breitenbach's method gives the highest results, those obtained by Mitscherlich's method (this J., 1910, 1217) are lower, whilst Atterberg's method gives still lower results. In the author's opinion, the chief value of the results obtained in the determination of the hygroscopic character of soils lies in the indications they give of the nature and size of the soil particles. (See also J. Chem. Soc., July, 1918.)—W. P. S.

*Aluminium as a factor influencing the effect of acid soils on different crops.* B. L. Hartwell and F. R. Pember. J. Amer. Soc. Agron., 1918, 10, 45—47. Bull. Agric. Intell., 1918, 9, 420—421.

THE toxicity of so-called acid soils is not due to the acid alone, as is shown by the different effects in

seedlings of barley and rye. Further, whilst the addition of acid to an ordinary nutrient solution caused similar effects with rye and barley seedlings, the aqueous extract of an acid soil affected the two kinds of seedlings very differently. This soil extract thus contained a substance not present in the ordinary nutrient solutions, and aluminium being present in large quantities in the soil extract a study was made of the effects of this element either alone or in combination with other substances on plant growth. The results indicate that aluminium must be considered as an important factor in the injurious effects of acid soils. The determination of the "active" aluminium of a soil may, therefore, prove as useful as the determination of its acidity, and the lime requirements of a soil may be due to the need of lime to precipitate the toxic aluminium just as much as to the need of lime to neutralise its acidity.—W. G.

*Soil acidity; Action of neutral salts on humus and other experiments on —.* L. J. Gillespie and L. E. Wise. J. Amer. Chem. Soc., 1918, 40, 796—812.

TRULY acid soils are common and widely distributed in the United States. The hydrogen electrometer provides the most trustworthy method for the measurement of the hydrogen ion concentration of soils; litmus paper used without suitable precautions can yield seriously inaccurate results. It is possible that the relatively high hydrogen ion concentration of some soils may account for their comparative freedom from potato scab disease. (See also J. Chem. Soc., July, 1918.)—D. F. T.

*Phosphoric acid; Use of "mine run" phosphates in the manufacture of soluble —.* W. H. Waggaman and C. L. Wagner. J. Ind. Eng. Chem., 1918, 10, 353—355.

PHOSPHATE rock as mined can be successfully treated by the electric furnace method (Ross and others, this J., 1916, 1154; 1917, 134), and the necessity of preparing and washing the rock is thus obviated, and the waste due to the discarding of low-grade material avoided. Experiments were made with a 500-lb. charge, mixed with coke, and with sand when silica was deficient. This was smelted in the furnace for 3 hours, and a yield of about 98% of the phosphorus was obtained. The cost of producing phosphorus pentoxide from "mine run" rock is estimated at from \$3.75 to \$6.50 less than from high-grade phosphates. Results are also given showing that by using the phosphorus pentoxide thus obtained to treat another batch of phosphate rock to form "double superphosphate," the cost of production compares favourably with that of the production of ordinary superphosphate by the sulphuric acid process under the present shortage of sulphuric acid.—W. F. F.

*Thomas slag; Determination of citric-soluble phosphoric acid in —.* R. Hartleb. Z. angew. Chem., 1918, 31, 61.

A DILUTE solution of nitric acid may be used in place of the citric acid solution usually employed for the determination of "citric-soluble phosphoric acid." A solution of which 1 c.c. is equivalent to 0.164 c.c. of N/1 nitric acid gives results identical with those obtained by the use of 2% citric acid solution.

*Fertilising value of activated [sewage] sludge.* G. G. Nasmith and G. P. McKay. J. Ind. Eng. Chem., 1918, 10, 339—344.

TO determine the fertilising value of activated sewage sludge and to compare it with other ferti-

lisers, experiments were made with a number of vegetable crops. Ordinary manure, activated sludge, sludge from an old bed, "Brush filter" humus, and tank sludge, air dried in each case, were used on similar plots, and comparative results are given showing the variation of the yield with different manures and with different vegetables. The increase in yield due to activated sludge over farmyard manure varied from 40% with radishes to 554% with Weatherfield onions, beets and tomatoes also giving a very high yield. The plants also reached maturity earlier than those grown with other fertilisers.—W. F. F.

*Treatment of washing water in the production of artificial silk.* De Chardounet. See V.

#### PATENTS.

*Nitrate of ammonia; Treatment of* —. E. Bergve and J. Sundby, Assignors to Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. U.S. Pat. 1,263,363, Apr. 23, 1918. Date of appl., May 8, 1917.

AMMONIUM nitrate, alone or mixed with plaster of Paris, is molstened with a liquid containing inorganic material which becomes viscous on the addition of water; and the mixture is agitated while being dried at 90°–110° C.—W. E. F. F.

*Fertiliser; Process of preparing a* — which remains permanently in a state suitable for distribution. Farbw. vorm. Meister, Lucius, u. Brünning. Ger. Pat. 301,184, Oct. 6, 1916.

IMPURE urea obtained from crude calcium cyanamide is mixed with calcium carbonate, in particular with the residue of calcium carbonate left in the preparation of urea from cyanamide. In this way a fertiliser containing about 20% of nitrogen is obtained in the form of a powder which does not cake on exposure to air.—C. A. M.

### XVII.—SUGARS; STARCHES; GUMS.

*Molasses; Water content of true final* —. H. C. Prinsen Geerligs. Intern. Sugar J., 1918, 20, 214–218.

As a corollary of the author's theory of the formation of final molasses (see this J., 1895, 667; 1918, 316 A), it follows that there is an optimum water content to which the massecuite must be reduced in order to attain the maximum exhaustion in practice. If more water is left in the massecuite than corresponds with the real water content of the hydrated combination of sucrose, reducing sugars, and salts (of which, according to the theory, molasses is composed), the excess water retains free sucrose in solution, and the final molasses is not fully exhausted. If, on the other hand, the concentration has been conducted beyond the optimum point, the viscosity of the product may be so greatly increased as to retard crystallisation and render the work of separation very laborious. To determine the optimum water content, molasses is diluted with water and divided into a number of portions, one of which is left untouched, whilst the others are evaporated each to gradually increasing stages of concentration, so as to obtain a series of molasses solutions, the first of which contains a higher and the last a lower water content than that corresponding to the optimum value. These solutions are heated in a water-bath to 100° C., and to each is added 20% of sucrose crystals (sifted free from "dust," and heated to the same temperature to pre-

vent sudden cooling of the mixture). After gradually lowering the temperature of the bath until after 3 days it reaches 45° C., the crystals are separated from the molasses, and determinations of sucrose, water content, and purity made in each case. When the dilution has been excessive and free sucrose has been dissolved, the purity of the molasses will be higher than at first; whilst when the water content is below the optimum the purity of the molasses will be lower. In the samples in which the water content corresponds with the optimum value, the purity of the molasses will have remained constant, and the true water content of the hydrated combination for the particular molasses under examination may then be calculated. From preliminary experiments using this method and from practical tests carried out in the sugar-house, it is concluded that the concentration to which molasses should be boiled to obtain a fully exhausted molasses is about 16% of water (i.e., 84% dry substance as indicated by the refractometer). A final molasses at this density may contain 160–174 parts of sucrose per 100 of water, depending upon the purity of the particular product.—J. P. O.

*[Beet] molasses; Polarisation of acidified solutions of* —. Andriik. Böhm. Zuckerind., 1918, 42, 347. Chem.-Zeit., 1918, 42, Rep., 55.

NUMEROUS experiments showed that whilst some acids, such as hydrochloric and sulphuric acids, give rise to hydrolysis and also (apart from this) raise the polarisation, others, such as boric and acetic acids, have little or no action in either of these respects, and others, including phosphoric and hydrofluosilicic acids, give rise to hydrolysis but do not otherwise affect the polarisation; acids of the last group often exert a considerable decolorising influence. In inverted solutions acids diminish the levorotation by their influence on the amino-acids; in the case of neutral or faintly acid molasses there occurs also a transformation of saccharin into saccharinic acid, which gradually increases the levorotation to a certain extent. On these results the author bases two methods of clarification, applicable to very dark molasses before and after inversion. After a preliminary defecation with basic lead acetate, a mixture of phosphoric and phosphotungstic acids is used in one case, and in the other acetic acid, sodium sulphide, and zinc dust (to precipitate the lead).—J. H. L.

*Molasses; Determination of sucrose in cane* — by Walker's method, using dry basic lead acetate for the preliminary clarification. H. S. Walker. Intern. Sugar J., 1918, 20, 239–240.

IN the determination of sucrose by double polarisation 0.5–1.0% less sucrose is found when the preliminary clarification is effected by Horne's method of using dry basic lead acetate than when a solution of basic lead acetate is used. Experiments carried out with solutions containing known amounts of sucrose, reducing sugars, and impurities, corresponding in composition to ordinary molasses, showed that when the dry reagent is used the results approximate closely to the truth, being only about 0.09% too low; whereas when a solution of the reagent is used the value obtained may be about 0.7% too high owing to the error due to the volume of the lead precipitate. The following procedure involving dry clarification is recommended: Twice the normal sugar weight of the sample is dissolved in water, made up to 300 c.c., clarified with 15–20 grms. of dry basic lead acetate, and filtered; 75 c.c. of the filtrate is transferred to a 100 c.c. flask, 20 c.c. of phosphoric acid (containing 100 grms. per litre) added, the volume made up to 100 c.c., and the liquid filtered. A pale yellow liquid is obtained,



the direct reading of which can be made in a 400 mm. tube. Another 75 c.c. is placed in a 100 c.c. flask, together with 2 c.c. of hydrochloric acid (1:1) to neutralise the alkalinity due to dissolved basic lead acetate, and the inversion carried out by Walker's method (this J., 1917, 153). After heating to 65°–70° C., 10 c.c. of hydrochloric acid (1:1) is added, and after at least 15 minutes the liquid is cooled, made up to bulk, filtered, and the inversion reading taken in a 400 mm. tube, the sucrose being calculated according to the formula:

$$\frac{D-I}{142.0-0.5T}$$

The results are accurate to within 0.07%, and are independent within reasonable limits of the amount of lead reagent used. Pellet's sulphurous acid method (this J., 1911, 64; 1913, 155) was found to yield results about 0.44% too high, and is uncertain owing to the danger of inversion while taking the direct reading. In the author's method the excess of phosphoric acid remaining in solution is the maximum possible without inversion occurring while taking the direct reading, and although its influence upon the levorotation of the reducing sugars is not so pronounced as that of 5 c.c. of hydrochloric acid, the difference is insufficient to introduce any appreciable error.—J. P. O.

*Molasses; Gravimetric determination of glucose (reducing sugars) in —.* R. S. Norris and A. Brodie. Intern. Sugar J., 1918, 20, 238–239.

ALTHOUGH in some samples of molasses non-sugar reducing substances precipitable by normal lead acetate may be absent, or only present in a negligible amount, clarification by means of this reagent, previous to the determination of the reducing sugars by means of Fehling's solution, is generally required. In order to remove the excess of the lead reagent disodium phosphate is recommended in preference to other salts, as it effects complete precipitation, and the excess left in solution has no action upon the copper reduction. Potassium oxalate (*cf.* Meade and Harris, J. Ind. Eng. Chem., 1917, 8, 504) increases the reduction; the use of sodium carbonate leads to low results, owing apparently to precipitation of some of the reducing sugars by the basic lead acetate formed on its addition; phosphoric acid, on the other hand, gives high results on account of its hydrolysing action on the sucrose. Varying the amount of sodium hydroxide in the Fehling's solution was not found appreciably to influence the amount of cuprous oxide precipitated.—J. P. O.

*Lime in products of [beet] sugar factories; Determination of —.* A. Vermehren. Z. Ver. deut. Zuckerind., 1917, 42, 610. Z. angew. Chem., 1918, 31, Ref., 81.

BLACHER's potassium palmitate solution (see this J., 1912, 555; 1913, 158), applied to the determination of the lime-content of beet sugars, massecuites, and molasses, gave results accurate enough for practical purposes. 25 grms. of the material is dissolved and made up to 100 c.c. with water containing phenolphthalein. An aliquot part of this solution, depending on the colour, is diluted to 100 c.c., neutralised by titration with N/10 hydrochloric acid (thus furnishing the "alkalinity"), then heated to boiling, and after cooling titrated with the palmitate solution.—J. H. L.

#### PATENTS.

*Sugar and the like; Apparatus for evaporating —.* J. T. Davis, Alameda, Cal. U.S. Pat. 1,263,747, Apr. 23, 1918. Date of appl., July 11, 1916.

THE evaporator comprises a vapour chamber, a conical plate therein, a steam chamber immediately

beneath it, of which the conical plate forms the upper side, an annular chamber situated at the top of the conical plate for containing the liquid to be evaporated and having an outlet through which the liquid is discharged on to the plate, a trough into which the liquid is discharged from the conical plate, a conveyor in the trough and a steam chamber on the under side, a vertical tube containing a conveyor which receives the material delivered from the first conveyor, a steam chamber around the tube, and means for operating the conveyors and supplying steam to the steam chambers simultaneously.—J. F. B.

*Adhesive composition [gum] and process of making same.* W. Alexander, Assignor to National Gum and Mica Co., New York. U.S. Pats. (A) 1,263,634 and (B) 1,263,635, Apr. 23, 1918. Dates of appl., Feb. 8 and Sept. 18, 1917.

(A) A SOLUTION of a gum of the gum arabic type is mixed with borax or a soluble boron compound and the mixture is dried and powdered. (B) The gum and boron compound are powdered or granulated and intimately mixed in the dry state.

—J. F. B.

#### XVIII.—FERMENTATION INDUSTRIES.

*Wine; Detection of cider in —.* P. Medinger and F. Michel. Chem.-Zeit., 1918, 42, 230.

FIFTEEN c.c. of the wine is shaken with a few c.c. of concentrated sodium nitrite solution; pure wines give a bright yellow or yellowish-brown coloration, whilst cider or perry is coloured dark brown or brownish-black and a brownish-black precipitate separates. This precipitate is insoluble in water, alcohol, ether, etc., but dissolves in alkali solution giving a red solution. Wines when treated with both sodium nitrite and potassium hydroxide give a yellow coloration, whilst cider and perry and mixtures of the same with wine yield a pure red coloration.—W. P. S.

*Enzyme formation.* VI. M. Jacoby. Biochem. Zeits., 1918, 86, 329–336.

WHILST leucine promotes the formation of urease in the case of *B. coli* it inhibits the sugar-fermenting action. (See also J. Chem. Soc., July, 1918.)

—S. B. S.

*Phosphoric acid esters of carbohydrates (zymophosphate); Preparation of — by means of living yeasts.* H. Euler. Biochem. Zeits., 1918, 86, 337–342.

NEUBERG has found that yeasts in the presence of toluene could only bring about esterification of 7–8% of phosphate, whereas the author, working under the same conditions, found complete esterification. The difference in results may be due to two causes, viz., excess of toluene inhibits the action, and not all bottom beer yeasts are active in this respect. The author also confirms the statement of Harden and Young that only a small amount of zymophosphate is produced by the action of the hydroxyacetone on phosphates. (See also J. Chem. Soc., July, 1918.)—S. B. S.

*Use of Sorghum vulgare (millet) in the cellulose industry.* Haas. See V.

*Some constituents of the American grape-fruit (Citrus decumana).* Zoller. See XIXa.

## PATENT.

*Diastatic product; Process for producing* —. J. Takamine, New York. U.S. Pat. 1,263,817, Apr. 23, 1918. Date of appl., Aug. 11, 1916.

A CULTURE medium is agitated in a tumbling drum and, during agitation, the medium is sown with a liquid containing in suspension the spores of a fungus capable of producing diastatic enzymes, and the mixture is agitated under incubating conditions so as to secure the contact of air with the medium. The culture medium contains an antiseptic composed of 10 parts of sodium fluoride and 17 parts of salicylic acid, added in the proportion of 1 part of antiseptic to 500–1000 of medium.—J. F. B.

## XIXA.—FOODS.

*Wheat; Substitutes for — in war bread.* Balland. Comptes rend., 1918, 166, 846–849.

FURTHER experiments (compare this J., 1917, 608) have been performed testing the use of various flours as substitutes for wheat flour in making war bread. The results indicate that the various substitutes act differently on the wheat gluten. Manioc flour used to the extent of 10 or 20% lowers to 28 or 24% the gluten of wheat flour containing 33%. Under the same conditions the flour from maize, barley, or rice gives approximately 29 and 26%. With soya flour panification is good, the crumb being more developed. Bread made with these substitutes retains more water than wheaten bread.—W. G.

*Beef; Estimation of various forms of nitrogen in raw —, including the products of hydrolysis of some of the proteins. I. Hexone bases of some proteins of beef. II. Bromination of the hydrolysates of some proteins of beef.* W. E. Thrun and P. F. Trowbridge. J. Biol. Chem., 1918, 34, 343–353, 355–362.

THE results showing the distribution of nitrogen in the proteins of beef indicate that the proteins which are soluble in water are chemically distinct from those which are insoluble. The amount of bromine absorbed by a hydrolysed protein can be regarded as a measure of the amount of histidine which it contained. After sundry corrections for the amount of bromine absorbed by other amino-acids, etc., the proposed method gives results which approximate to those yielded by Van Slyke's method. (See also J. Chem. Soc., July, 1918.)

—H. W. B.

*Grape-fruit (Citrus decumana) Some constituents of the American —.* H. F. Zoller. J. Ind. Eng. Chem., 1918, 10, 364–374.

ANALYSES of grape-fruit (weight 430–770 grms.) showed that the peel contained 2.5 to 6.0 grms. per fruit of essential oils, consisting principally of limonene (90–92%) with small quantities of citral,  $\alpha$ -pinene, geraniol, and linalool; also about 0.2 to 1.6 gm. of the glucoside naringin per fruit, and about 4 grms. of pectin. The pulp contains about 1.9 to 6.0 grms. of citric acid, 6.2 to 15 grms. of sucrose, and 8.6 to 19.6 grms. of dextrose per fruit. It is estimated that 10 to 15 galls. of proof spirit could be obtained from the sugars extracted from 1 ton of grape-fruit. (See also J. Chem. Soc., July, 1918.)—W. F. F.

*Litchi nut (Litchi chinensis); The edible —.* B. E. Read. J. Amer. Chem. Soc., 1918, 40, 817–822.

THE so-called Chinese hazel nut, *Litchi chinensis*, is known in several varieties, but those from Canton are almost spherical with a dull brick red

pericarp enclosing a sweet brown fleshy arillus which surrounds a glossy chestnut brown globular seed; It has long been used by the Chinese in medicine for reducing swellings, and is said to produce feverishness and bleeding at the nose if eaten excessively, but no iodine or saponin is present. It is practically fat free; the nitrogen-free extractive matter is composed mainly of simple sugars in which invert sugar predominates. Examination of the ash indicates a considerable content of the mineral salts needed in a well-balanced diet. A full bibliography on the grape fruit is included in the paper.—D. F. T.

*Potatoes; Digestibility and food value of — in their different forms as fed, of potato-vinasses and of potato-haulms with farm animals.* W. Völtz. Z. Spiritus-Ind., 1918, 41, 1–2. Z. angew. Chem., 1918, 31, Ref., 162.

DETAILS are given as to the food value of potato-vinasses and the preservation of raw and steamed potatoes by "souring." Metabolism experiments with sheep, milch cows, and a horse, feeding potato-haulms (in a dry condition and as silage) and potato "apples" are described.

—W. G.

*Manufacture of paper pulp, etc., from dead leaves.* Bramson. See V.

*Oil-content, keeping qualities, and commercial possibilities of Para rubber seed.* Spring and Day. See XII.

*Determination of chlorine in presence of organic matters (gastric juice, blood, milk, etc.).* Sirot and Joret. See XXIII.

## PATENT.

*Nucleo-albumins in preserved eggs; Method for making soluble the —.* N. Bendixen, London. Eng. Pat. 115,261, Apr. 30, 1917. (Appl. No. 6086 of 1917.)

THE nucleo-albumins of dried eggs are again rendered soluble in water by conversion into salts. When the eggs have been dried whole, a sample of the product is titrated with standard alkali, and the requisite quantity of alkali hydroxide, either in the dry state or in solution, is added to the bulk of the product. When added in solution, the product is again dried. When the whites have been dried and the yolks preserved in the liquid condition by means of boric acid, water is added to the dried whites and the preserved yolks mixed with the solution, which is then neutralised with alkali, and hydrogen peroxide added as a preservative.—L. A. C.

## XIXB.—WATER PURIFICATION; SANITATION.

*Sand filters; Theory of action of —.* L. Minder. J. Gasbeleucht., 1918, 61, 56–57.

KISSKALT's view (J. Gasbeleucht., 1917, [9]) that the action of the sand filter in water purification is biological and due to the destruction of bacteria by other organisms is contested on the basis of experience at Zürich waterworks where the lake water is filtered in two stages through sand. The first retains most of the fresh-water planktons but allows some of the bacteria to pass. The bacteria are retained by the second filter and at the surface, so that at a depth of 10 cm. the number per c.c. has already fallen to one-tenth. Furthermore the retention of bacteria is satisfactory even when



there are considerable fluctuations in the number of bacteria in the water, and it is concluded that the process is mechanical rather than biological.

—H. J. H.

*Snow and rain water; The sulphuric acid content of —.* E. Küppers. Z. angew. Chem., 1918, 31, 74–76.

SULPHUR dioxide is so rapidly oxidised in the atmosphere that only sulphuric acid is found in snow and rain water. Sulphuric acid is rapidly deposited from the atmosphere on the exposed surfaces of trees and roofs. The sulphuric acid content of freshly fallen snow and rain depends on the proximity of industrial centres and the prevailing direction of the wind. An average figure for fresh snow in an industrial district is 15–20 mgrms.  $\text{SO}_3$  per kilo. Fresh rain water collected in an open space in an industrial district contained 17–18 mgrms.  $\text{SO}_3$  per litre; this is very much less than has been recorded by certain agricultural stations. Distilled water exposed to the air in an industrial district absorbed about 1.2 mgrm.  $\text{SO}_3$  per 100 sq. m. in 24 hours.—J. F. B.

*Antiseptic value of some essential oils.* L. Cavel. Comptes rend., 1918, 166, 827–829.

FORTY-FIVE essential oils were examined in regard to the limit dose (in parts per 1000) necessary to be added to gelatin bouillon to prevent all microbial vegetation, even after seven months, when the plates were inoculated with a sewage effluent containing 9–11 million germs per c.c. The amounts required varied from 0.7 part per 1000 in the case of thyme oil up to over 15.0 parts per 1000 in the case of patchouli oil, the value, under similar conditions, for phenol being 5–6 parts per 1000.—W. G.

*Fertilising value of activated [sewage] sludge.* Nasmith and McKay. See XVI.

#### PATENTS.

*Water; Process of softening —.* H. Reiser. Ges.m.b.H., Cologne - Braunsfeld, Germany. Eng. Pat. 112,127, Nov. 28, 1917. (Appl. No. 17,607 of 1917.) Under Int. Conv., Dec. 16, 1916.

THE water is boiled to remove free and half-bound carbon dioxide before being treated with base-exchanging reagents, the formation of slimy deposits being thus prevented. Apparatus for this process includes a cooling apparatus between the boiling vessel and the tank containing the reagents, the untreated and the boiled water traversing this cooler in opposite directions. The cooling apparatus and the reagent tank are preferably sealed against admission of air.—C. A. M.

*Waste waters [containing organic matter in solution]; Method of treating —.* C. E. Cummings, Assignor to Eastern Tanners Glue Co., Gowanda, N.Y. U.S. Pat. 1,263,532, Apr. 23, 1918. Date of appl., Oct. 27, 1917.

CARBON dioxide is passed through the waste liquid to precipitate the organic matter, and the precipitate is removed.—L. A. C.

*Sewage; Biological filter with oxidation for the destruction of bacteria, etc., in town and industrial —.* Claros G.m.b.H. für Reinigung städtischer u. gewerblicher Abwässer, Dresden. Ger. Pat. 302,642, Nov. 9, 1915.

THE filter is constructed of non-porous bodies of natural or artificial material, in the shape of crosses, built up in a regular order and separated by bars of square cross-section, so as to allow free access of air to the finely divided liquor which trickles through the filter.—H. J. H.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Arbutin; Detection of —.* H. Salomon. Ber. Dent. pharm. Ges., 1918, 28, 138–139.

THE ferric chloride test for bearberry extract and the similar test to be applied to the urine excreted after the consumption of bearberry leaf tea are not specific to arbutin, nor are the reducing tests with Fehling's solution or Nylander's reagent. In all these cases the reaction is probably due to other substances. (See also J. Chem. Soc., July, 1918.)

—D. F. T.

*Saponins [from mowrah seed].* L. Spiegel and A. Meyer. Ber. Dent. pharm. Ges., 1918, 28, 100–126.

THE saponin mowrin obtained from the seeds of *Bassia longifolia* (mowrah seeds) is a mixture of two substances (compare Moore, Sowton, Baker-Young and Webster, this J., 1909, 1061), the chief of which is more soluble in alcohol. This main ingredient has the formula  $\text{C}_{42}\text{H}_{68}\text{O}_{23}$  and on hydrolysis yields levulose, arabinose, and mowric acid, the last of which is a mixture of a crystalline mowragenic acid,  $\text{C}_{19}\text{H}_{28}\text{O}_8$ , and an amorphous mowrageninic acid,  $\text{C}_{19}\text{H}_{26}\text{O}_8$ . By careful hydrolysis with dilute acetic acid an intermediate pentoside  $\text{C}_{29}\text{H}_{50}\text{O}_{16}$  can be obtained. (See also J. Chem. Soc., July, 1918.)—D. F. T.

*Lecithin. II. Preparation of pure lecithin; composition and stability of lecithin cadmium chloride.* P. A. Levene and C. J. West. J. Biol. Chem., 1918, 34, 175–186.

LECITHIN may be freed from accompanying traces of other phosphatides by transformation into its cadmium chloride compound, which is then recrystallised repeatedly from ethyl acetate and alcohol until free from amino-nitrogen. Subsequent treatment with ammonium carbonate liberates the pure lecithin. (See further J. Chem. Soc., 1918, i., 288.)—H. W. B.

*Essential oils; Constituents of —.* Betulol. F. W. Semmler, K. G. Jonas, and W. Richter. Ber., 1918, 51, 417–424.

BETULOL,  $\text{C}_{15}\text{H}_{24}\text{O}$ , and its acetate were found by von Soden and Elze (this J., 1905, 634) in the oil of birch buds. From the results of a systematic investigation, the conclusion is drawn that betulol is a bicyclic, sesquiterpene alcohol of the terpene type. When treated with phosphorus pentachloride, part of the oil suffers ring closure under the influence of the hydrogen chloride liberated, the product being a mixture of bi- and tri-cyclic chlorides. If this is warmed with lime, a mixture of bicyclic dehydrobetulene,  $\text{C}_{15}\text{H}_{22}$ , and tricyclic betulol,  $\text{C}_{15}\text{H}_{24}\text{O}$ , is obtained, the latter crystallising in silky needles, m. pt. 147°–148° C. (See further J. Chem. Soc., 1918, i., 301.)—J. C. W.

*Chloretone (trichloro-tert-butyl alcohol); Method for detecting small quantities of — in aqueous solutions.* T. B. Aldrich. J. Biol. Chem., 1918, 34, 263–267.

VERY small quantities of chloretone can be recognised by distilling with steam and boiling the distillate in a flask with reflux condenser, when needle crystals of chloretone form in the cooler part of the condenser. The presence of other organic solvents prevents the crystallisation. (See also J. Chem. Soc., July, 1918.)—H. W. B.

*Formamide; Preparation of* —. A. Brauna. J. Amer. Chem. Soc., 1918, 40, 793—796.

WORKING details are given for the preparation of formamide by the distillation of ammonium formate in a current of ammonia gas. By fractional redistillation under reduced pressure pure formamide can be obtained as a neutral viscous hygroscopic liquid which freezes at 2-25° C. (See also J. Chem. Soc., July, 1918.)—D. F. T.

*Dehydrogenation. A new method. Synthesis of decacyclene, fluorocyclene, and chlorene, a green hydrocarbon.* K. Dziewonski and S. Suknarowski. Ber., 1918, 51, 457—465.

WHEN acenaphthene is heated with lead oxide in a sealed tube, decacyclene,  $C_{30}H_{18}$ , fluorocyclene,  $C_{30}H_{12}$ , and small quantities of chlorene,  $C_{30}H_{12}$ , crystallising in dark green scales and giving pure green solutions, are formed. By modifying the conditions slightly, either of the chief products can be obtained in better yields than hitherto experienced. (See further J. Chem. Soc., 1918, i, 296.)—J. C. W.

*Toluol [and cumene] from spruce turpentine.* Wheeler. See III.

*Some constituents of the American grape-fruit (Citrus decumana).* Zoller. See XIXA.

*The edible Litchi nut (Litchi chinensis).* Read. See XIXA.

*Antiseptic value of some essential oils.* Cavel. See XIXB.

*Graphic methods of analysis.* Gradenwitz. See XXIII.

#### PATENTS.

*"Zingiberone" (methyl 3-methoxy-4-hydroxyphenylethyl ketone); Method of preparing* —. H. Nomura, Sendai, Japan. U.S. Pat. 1,263,796, April 23, 1918. Date of appl., June 6, 1917.

ZINGIBERONE, a pungent principle of ginger root (*Zingiber officinale*) is prepared by condensing vanillin with acetone in the presence of aqueous caustic soda with formation of vanillylideneacetone, and reducing this by means of hydrogen in the presence of platinum black. (See also this J., 1917, 1062.)—D. F. T.

*Ketones of the thiophen series; Manufacture of* —. W. Steinkopf, Berlin-Hatensee. Ger. Pat. 302,838, Oct. 8, 1916. Addition to Ger. Pat. 297,203 (this J., 1917, 638).

ORGANIC acid anhydrides may be used instead of their chlorides.—J. C. W.

*Lecithin from plant substances; Process for obtaining* —. C. F. Hildebrandt, Hamburg. Ger. Pat. 304,889, June 25, 1914.

LECITHIN is obtained by the usual extraction processes from the powdery substance occurring on the underside of the shells of leguminous or oleaginous fruits or of the husks of corn. This substance is much richer in lecithin than the whole fruit; e.g., peas contain about 1% lecithin, maize only  $\frac{1}{4}$ %, the meal from the shells containing about 3% in each case.—B. V. S.

*Alkaloids of cinchona bark; Preparation of hydrogenated products of the total* —. Chem. Werke Grenzach A.-G., Grenzach. Ger. Pat. 304,910, Aug. 12, 1913.

THE impure drug obtained by the usual technical methods, or the ordinary extract of cinchona bark is treated with hydrogen in the presence of palladium. From the resulting tarry extract easily soluble crystallisable substances are obtained.

—B. V. S.

*1 - Phenyl - 2,3 - dimethyl - 4 - diallylamino - 5 - pyrazolone; Preparation of* —. Ges. für Chem. Ind. in Basel. Ger. Pat. 304,983, Apr. 14, 1916.

1-PHENYL-2,3-DIMETHYL-4-AMINOPYRAZOLONE is treated in the warm with alkyl halides, conveniently in the presence of a solvent or diluent and of a substance capable of neutralising the resulting acid. 1-Phenyl-2,3-dimethyl-4-diallylamino-5-pyrazolone obtained by this method is a stronger antipyretic than its well known dimethylamino-analogue, and possesses a more prolonged action although without increased toxicity; it also has a marked narcotic effect which is not shared to any appreciable extent by the dimethylamino-compound.

—D. F. T.

*Extracts of drugs; Preservation of* —. Knoll und Co., Ludwigshafen. Ger. Pat. 305,328, Dec. 14, 1915.

THE extracts or solutions of such extracts are treated with glycerol and camphor or a camphor substitute such as synthetic camphor. Solutions which have received this treatment, digitalis extracts in particular, resist the development of moulds and bacteria and therefore remain of use for injection purposes even after having been exposed to the air.—D. F. T.

*Alphylselenylureas; Preparation of* —. Chem. Fabr. von Heyden, A.-G. Ger. Pats. (A) 305,262, and (B) 305,263, May 27, 1915.

(A) THE products obtained by the action of hydrogen selenide on an alkyl cyanamide, e.g., allylcyanamide, in contrast to phenylselenylurea possess marked therapeutic effect, especially towards cancerous diseases; they are particularly valuable as intermediate products to their more stable and similarly active additive compounds with the alkyl halides. (B) The additive compounds of the alkylselenylureas with the alkyl halides probably possess the constitution  $NH_2C(NHR):SeRI$  where R represents an alkyl radicle; the selenium is more firmly attached than in the corresponding selenylureas and as the compounds are more stable towards light and more soluble in water they are more convenient for injection.—D. F. T.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### PATENTS.

*Photographic emulsions; Preparation of* —. W. Scheffer, Berlin-Wilmersdorf. Ger. Pat. 304,737, Dec. 17, 1916.

THE light-sensitive salts are produced as very fine-grained precipitates by allowing the reacting solutions to come into contact at a dialysing membrane. The solutions may be at different temperatures and pressures, and two or more dialysing membranes may be used.—B. V. S.

*Process and apparatus for the separation of metals.* Ger. Pat. 302,279. See X.



## XXII.—EXPLOSIVES; MATCHES.

*Silver acetylide.* J. Eggert. Chem.-Zeit. 1918, 42, 199—200. (See also Ber., 1918, 51, 454.)

THE noise accompanying explosive decomposition is due to sudden alterations in the gaseous pressure occurring with a velocity of the order of that of sound. The acetylides of the heavy metals, e.g., silver acetylide, in decomposing, might be expected to give rise to carbon and metal only and therefore the explosion should be quiet except for the sudden disturbance in the surrounding air. However silver acetylide, especially if prepared from a solution of silver nitrate in aqueous nitric acid, explodes with great violence and noise even in a vacuum, whereas with a product precipitated from an ammoniacal solution of silver nitrate the explosion is much milder. The explanation of the vigour of the explosion and of the difference between the behaviour of the two varieties is to be found in the fact that the explosion does give rise to gaseous products, the "acid" variety yielding 17.2 c.c. of gas (N.T.P.) per 0.1 grm. of substance, whilst the "ammonia" variety yields only approximately one-tenth of this amount. The gaseous products consist of water vapour, hydrogen, oxides of carbon, and methane, together with oxides of nitrogen in the case of the "acid variety"; these must be due to the presence of occluded silver nitrate and silver oxide respectively in the "acid" and "ammonia" products. The latter variety is obtained purer, the lower the concentration of the silver and the greater the proportion of the ammonia. The disruptive power of "acid" silver acetylide is greater than that of silver fulminate but less than that of silver azide, the difference being attributed to difference in the velocities of detonation.—D. F. T.

## PATENTS.

*Explosives.* A. Segay. Ware, Herts. Eng. Pat. 113,083, Mar. 15, 1917. (Appl. No. 3812 of 1917.) Under Int. Conv., Aug. 17, 1916.

IN explosives of the type composed of ammonium nitrate, trinitrotoluene, and calcium silicide, the proportion of the last-named ingredient is increased up to an amount such that, once the metal is oxidised, the whole of the carbon is burnt only to carbon monoxide, and only half the hydrogen to water. For example, with a mixture of ammonium nitrate 66, tetranitromethylaniline, or any explosive which may be detonated by itself, 7, and calcium silicide 27%, the excess of calcium silicide results in a more powerful explosive, and one which can be advantageously compressed to a greater degree, whereby it is made less hygroscopic, and becomes hard enough to handle easily. When the proportion of ammonium nitrate is high enough to cause the complete oxidation of the calcium silicide and to burn all the carbon and hydrogen to carbon dioxide and water respectively, the mixture will not explode satisfactorily if compressed so that the specific gravity is above 1.25, but an explosive of the composition given above may, on the other hand, be compressed to a specific gravity of 1.45 or even higher.—T. St.

*Soap [for removing stains caused by nitro compounds].* Eng. Pat. 115,110. See XII.

## XXIII.—ANALYSIS.

*Ultra-filters; New and simple* —. Ostwald. Kolloid Zeits., 1918, 22, 72—76.

SIMPLE forms of apparatus for the filtration of colloidal solutions are described. An ordinary funnel or a Büchner funnel may be employed if the filter paper is treated *in situ* with a 2%

collodion solution and certain precautions are observed in the fitting up of the apparatus. The best results, considered with reference to the speed of filtration, were obtained with an ultra-filter prepared by similar treatment of a Schleicher-Schüll Filtrierhut No. 577. A number of colloidal solutions of widely divergent character have been subjected to ultra-filtration in the apparatus described and the results obtained in each case are recorded.—H. M. D.

*Gravimetric analysis; Contributions to* —. L. W. Winkler. Z. angew. Chem., 1918, 31, 101—103.

V. *Determination of chlorides.* 100 c.c. of the cold solution is treated with 5 c.c. of N/1 nitric acid (or in presence of ferric salts, 10—20 c.c.) and a moderate excess of N/1 silver nitrate solution. The mixture is allowed to stand for one hour and then heated to boiling. After 24 hours the clear liquid is decanted through a plug of cotton wool in a "Kelch funnel." The clotted precipitate in the beaker is broken up with a very small quill in the first portion of the wash water and finally transferred to the filter. It is washed with 50 c.c. of cold water containing 2—3 drops of nitric acid and then with 50 c.c. containing a few drops of acetic acid. Pure water gives a cloudy filtrate. The precipitate is sucked dry with the pump and dried at 132° C. Small corrections depending on the weight of the precipitate are applied, e.g., -0.8 mgrm. for 1 grm., -0.1 mgrm. for 0.4 grm., and +0.2 mgrm. for 0.2 grm. and less. The results are accurate in presence of ammonium, alkali, alkaline-earth, magnesium, zinc, copper, manganese, aluminium, and ferric salts, but not in presence of mercury salts.

VI. *Determination of bromides.* The details are similar to those given for chlorides; the correction values are slightly smaller.

VII. *Determination of iodides.* The nitric acid is added half an hour after the silver nitrate; the correction values are -0.2 mgrm. for 1 grm., +0.3 mgrm. for 0.4 grm., and +0.5 mgrm. for 0.2 grm. of precipitate. Iodides may also be determined as palladium iodide. The procedure is varied according to the amount of chloride present. With iodides in excess, the neutral solution is diluted so that 100 c.c. will give about 0.1 grm. of precipitate; 1.0 grm. of sodium chloride is added and 10 c.c. of palladium chloride solution (0.5 grm. of palladium converted into chloride and dissolved in 10 c.c. of 10% hydrochloric acid, mixed with 1 c.c. of alcohol, and diluted to 100 c.c.). The mixture is gently boiled until the precipitate becomes granular, filtered off the next day, washed with 100 c.c. of cold water, and dried at 132° C. If the quantity of iodide is small, 100 c.c. of the solution acidified with hydrochloric acid is treated with 1 c.c. of palladium chloride and left with occasional shaking for 1—2 days. The flocculent precipitate is collected as before. The presence of bromides interferes with the accuracy, and, if considerable, the method cannot be used.—J. F. B.

*Metals of the group soluble in ammonia; Separation of the* —. A. Carnot. Ann. Chim. Analyt., 1918, 23, 93—97.

*Copper and zinc.* The metals are weighed together as oxides and the latter are then heated at bright redness for about 45 mins. in a current of hydrogen; the zinc is reduced and volatilises completely, leaving a residue of metallic copper which is weighed. *Copper, zinc, and nickel.* The dilute hydrochloric acid solution of the three metals is heated to boiling and treated with sodium thio-sulphate; copper is thus precipitated as sulphide and is separated by filtration. The zinc and nickel in the filtrate are precipitated as carbonates, ignited, and the zinc volatilised by heating in a



current of hydrogen. **Zinc and cadmium.** The metals are dissolved in nitric acid, the solution evaporated, the residue dissolved in dilute nitric acid, the solution is diluted and rendered alkaline with sodium carbonate, and sufficient ammonium carbonate is added to dissolve the zinc carbonate. The mixture is boiled until all free ammonia has been expelled, the cadmium carbonate is collected on a filter, and the zinc is determined in the filtrate as sulphide. To remove all the zinc, the cadmium carbonate should be dissolved and re-precipitated. **Copper, zinc, cadmium, nickel, and cobalt.** Copper is separated as sulphide by treating the hydrochloric acid solution of the metals with sodium thiosulphate (see above). The solution is then nearly neutralised, a large excess of ammonium oxalate is added, and the zinc is precipitated by treating the hot solution with hydrogen sulphide. Cadmium, if present, is also precipitated, and may be separated from the zinc as described. Nickel and cobalt are precipitated together as sulphides by rendering the solution slightly ammoniacal, heating it at  $100^{\circ}\text{C}$ ., and adding sodium sulphide; they are then separated from one another by known methods.—W. P. S.

**Copper; Estimation of** — as copper oxide after previous precipitation as thiocyanate. G. Fenner and J. Forschmann. Chem.-Zeit., 1918, 42, 205—206.

The advantage of the thiocyanate precipitation of copper over the hydrogen sulphide method may be increased by the direct conversion of the cuprous thiocyanate into cupric oxide. The copper thiocyanate precipitation is not interfered with by the presence of iron, manganese, zinc, cobalt, nickel, arsenic, or bismuth; antimony and tin are previously removed together with lead when the latter is converted into lead sulphate. The accuracy of the thiocyanate precipitation is not affected by a twenty-fold excess of reagent, but the proportion of sulphurous acid or of ammonium sulphite should not be too great. By heating in a muffle at  $800^{\circ}\text{C}$ . the cuprous thiocyanate precipitate can be quantitatively converted into cupric oxide but, if desired, the alternative procedure may be adopted of dissolving the precipitate in a mixture of nitric and sulphuric acids and separating the copper electrolytically; with small quantities of copper this alternative solution method may be modified by applying a colorimetric method of estimation. (See also J. Chem. Soc., July, 1918.)—D. F. T.

**Strontium; Determination of** —. L. W. Winkler. Z. angew. Chem., 1918, 31, 80 and 83—84.

For the determination of strontium as sulphate, 100 c.c. of a neutral solution containing 0.5 gm. of strontium salt is acidified with 1 c.c. of acetic acid and heated to boiling, when 10 c.c. of a 10% solution of sodium sulphate is added. Heating is continued until the precipitate has become powdery, when it is allowed to stand overnight. The precipitate is transferred to a Gooch crucible, washed with 50 c.c. of saturated strontium sulphate solution, and weighed after drying at  $132^{\circ}\text{C}$ . If the filtrate is required further, alcohol is used for washing. Low results are obtained in the presence of many salts and of nitric and hydrochloric acids. For determination as oxalate, precipitation is effected as above but with 10% potassium oxalate solution. After standing overnight the precipitate is washed with saturated strontium oxalate solution. It may be dried at  $100^{\circ}\text{C}$ . for 2 hours and weighed as  $\text{SrC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ , or at  $132^{\circ}\text{C}$ . for 6 hours and weighed as  $\text{SrC}_2\text{O}_4$ . Other salts, especially magnesium chloride interfere. Strontium may be determined as carbonate by adding 1.0 gm. of potassium nitrate and 10 c.c. of 10% sodium carbonate solu-

tion to a boiling solution of not more than 0.5 gm. of strontium salt in 100 c.c. of solution. Next day the precipitate is washed with 50 c.c. of saturated strontium carbonate solution, dried at  $132^{\circ}\text{C}$ ., and weighed. Owing to incomplete loss of carbon dioxide the precipitate cannot be weighed as oxide after ignition. It is most convenient and accurate to estimate strontium as oxalate.—H. J. H.

**Iron; Use of metallic silver as a reducing agent in the volumetric estimation of** —. G. Edgar and A. R. Kemp. J. Amer. Chem. Soc., 1918, 40, 777—784.

Ferric sulphate in sulphuric acid solution is quantitatively reduced to ferrous sulphate by the action of metallic silver if a soluble thiocyanate is present. The solution is treated with 3–5 grms. of metallic silver and with ammonium thiocyanate in excess of that necessary to precipitate the silver dissolved in the reaction. The dark red solution is shaken in the cold until it becomes quite colourless, is then filtered and the filtrate treated with silver nitrate and titrated with standard permanganate. If the reacting mixture is boiled, reduction is effected more quickly. An alternative method consists in adding measured quantities of standard ammonium thiocyanate and silver nitrate solutions and after titrating with permanganate, determining the excess of silver nitrate in the solution by means of the standard ammonium thiocyanate. Test measurements show that the methods give accurate results. The use of silver and thiocyanate has certain advantages over other methods of estimation in that the thiocyanate shows when the ferric iron is completely reduced, that metallic silver is ordinarily quite free from iron, making blank determinations unnecessary, and that silver has no reducing action on titanium, whilst vanadium is reduced sharply to the quadrivalent state and may consequently be easily corrected for.—H. M. D.

**Chlorine; Determination of** — in the presence of organic matters (gastric juice, blood, milk, etc.). Sirot and Joret. Ann. Chim. Analyt., 1918, 23, 109—113.

To determine total chlorine in gastric juice, 20 c.c. of the liquid is treated with 20 c.c. of a reagent containing 10 grms. of picric acid and 25 grms. of acetic acid per litre; the mixture is diluted to 100 c.c., filtered, and the chlorine titrated in the filtrate by Volhard's method. Combined chlorine (organic and inorganic) is determined in the same way after the liquid has been evaporated to expel free hydrochloric acid and then diluted with water. Fixed chlorine (inorganic) is determined in the ash of the sample. Sodium metaphosphate is a better clarifier than picric acid in the case of blood; 20 c.c. of the sample is treated successively with 75 c.c. of water, 10 drops of nitric acid, 20 c.c. of 5% sodium metaphosphate solution, and 1.5 c.c. of acetic acid, the mixture is diluted to 200 c.c., filtered, and the chlorine titrated in an aliquot portion of the filtrate. The picric acid reagent mentioned, sodium metaphosphate, or basic lead acetate may be used for clarifying milk previous to the determination of the chlorine present. (See also J. Chem. Soc., July, 1918.)—W. P. S.

**Fluorine; Gravimetric and volumetric determination of** — as thorium fluoride. F. A. Gooch and M. Kobayashi. Amer. J. Sci., 1918, 45, 370—376.

In the method described by Pisanì (this J., 1916, 709) the fluoride solution should contain from 0.12 to 1.2 gm. of free acetic acid per 100 c.c., and the quantity of standardised thorium nitrate solution added must be such that the excess of thorium does not exceed by more than 50% the quantity required



for precipitation. The precipitated thorium fluoride is collected after a few hours, washed by decantation with very dilute acetic acid, and ignited to thorium oxide; the weight of the latter is a measure of the thorium fluoride,  $\text{ThF}_4 \cdot \text{H}_2\text{O}$ . Alternatively, the excess of thorium nitrate in the filtrate may be determined by precipitation as oxalate and titration of the latter with permanganate. In dealing with unknown quantities of fluorine, a small quantity of the thorium nitrate solution is treated with the fluoride solution until a distinct turbidity is produced; twice as much thorium nitrate is required, in proportion, to produce this turbidity as is necessary to precipitate the fluoride and leave an excess of about 50% of thorium in the solution. (See also J. Chem. Soc., July, 1918.)—W. P. S.

*Combustions of substances containing nitrogen; Rapid organic* —. H. L. Fisher and A. H. Wright. J. Amer. Chem. Soc., 1918, 40, 868–869.

In effecting rapid organic combustions of nitrogenous substances, using cerium oxide as a catalyst, with subsequent layers of copper oxide and a mixture of lead peroxide and red lead, trouble may be encountered owing to the formation of copper nitrate especially as the neighbouring lead oxide layer has to be maintained at  $300^\circ$ – $320^\circ \text{C}$ .; when a tube is used several times it may even become choked with basic copper nitrate. If copper oxide is used and if it is desired to make several combustions with a tube it is necessary either to renew the copper oxide each time or so to modify the procedure as to ensure the complete decomposition of any copper nitrate after each combustion. It is also found advantageous to use the cerium oxide deposited on pumice instead of on asbestos.—D. F. T.

*Analysis; Graphic methods of* —. H. Gradenwitz. Chem.-Zeit., 1918, 42, 221.

GRAPHS are given by means of which the composition of ternary mixtures may be ascertained. In the case of a mixture of formaldehyde, methyl alcohol, and water, the formaldehyde is determined in the usual way and the sp. gr. of the mixture is taken; these data being known, the percentage of methyl alcohol is found from the graph. With a mixture of ethyl acetate, alcohol, and water, the sp. gr. and the volume of ethyl acetate which separates when 100 c.c. of the mixture is shaken with 100 c.c. of water are determined; the percentage quantities of the three constituents are then read on the graph. (See also J. Chem. Soc., July, 1918.)—W. P. S.

*Calculation of the excess of air used in combustion.* Hassreidter. See IIa.

*Nature of the cellulose of cereal straw.* Heuser and Haug. See V.

*Colour reaction of mechanical wood pulp or the incrusting substances of wood with phenylhydrazine hydrochloride.* Jentsch. See V.

*Apparatus for the determination of nitric acid by the Schulze-Tiemann method.* Leuchs. See VII.

*Determination of carbon dioxide in carbonates by Dittrich's method.* Von Horvath. See VII.

*Adsorption of sodium gold chloride by charcoal and the estimation of gold in sea water.* Koch. See VII.

*Use of Brinell ball in testing [hardness of] constructional materials.* Le Chatelier and Bogitch. See VIII.

*Rapid determination of total carbon in steel and cast iron.* Sernagiotto. See X.

*Estimation of oxygen in copper.* Oberhoffer. See X.

*Analysis of white metals.* Drawe. See X.

*Rapid determination of resins in soap.* Fortini. See XII.

*Examination of oil varnishes.* Smit-Addens. See XIII.

*Determination of free sulphuric acid in leather.* Strunk and Matthes. See XV.

*Mechanical and physical analysis of soils.* Richter. See XVI.

*Determination of citric-soluble phosphoric acid in Thomas slag.* Hartleb. See XVI.

*Water content of true final molasses.* Prinsen Geerligs. See XVII.

*Polarisation of acidified solutions of beet molasses.* Andrlík. See XVII.

*Determination of sucrose in cane molasses by Walker's method, using dry basic lead acetate for the preliminary clarification.* Walker. See XVII.

*Gravimetric determination of glucose (reducing sugars) in molasses.* Norris and Brodie. See XVII.

*Determination of lime in products of [beet] sugar factories.* Vermehren. See XVII.

*Detection of cider in wine.* Medlinger and Michel. See XVIII.

*Estimation of various forms of nitrogen in raw beef, including the products of hydrolysis of some of the proteins.* Thrum and Trowbridge. See XIXa.

*Detection of arbutin.* Salomon. See XX.

*Method for detecting small quantities of chloretone (trichloro-tert.-butyl alcohol).* Baldrich. See XX.

#### PATENTS.

*Oil-tester [for determining the flash-point].* C. J. Tagliabue, Brooklyn, N.Y., Assignor to C. J. Tagliabue Manufacturing Co. U.S. Pat. 1,263,145, Apr. 16, 1918. Date of appl., Aug. 14, 1915.

An open oil cup is mounted in a water bath, and a bracket having a vertical bearing is attached to the outside of the bath. A gas tube is mounted to rotate

in the bearing and the upper end of the tube is bent horizontally and carries a burner tip which is thus movable over the top of the oil cup in a horizontal plane. Means are provided on the tube and bracket to maintain the burner at a constant predetermined height, and a stop-cock is provided for adjusting the size of the flame.—W. F. F.

*Drying apparatus.* Eng. Pat. 114,620. See I.

*Process for testing the wearing quality of wool.*  
Ger. Pat. 302,808. See V.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Allen. Centrifugal mixers, washers, etc. 10,279 and 10,280. June 21.

Barbet et Fils et Cie. 10,116. See XX.

Beeton, and Trufood, Ltd. Drying organic substances. 10,057. June 18.

Birkedal and Nielson. Apparatus for mixing liquid or gaseous substances. 10,685. June 28. (Denmark, Apr. 24, 1917.)

Brooks. Regenerative oven or furnace. 10,659. June 28.

Brownlow. Centrifugal separators. 10,231. June 21.

Drysdale, Drysdale and Co., and Young. Straining or screening apparatus for liquids. 10,244. June 21.

Drysdale, Drysdale and Co., and Young. Strainers or screens for liquids. 10,245. June 21.

Elektro-Osmose A.-G. 10,453—10,460. See XI.

Fullager, Reavell, and Kestner. Evaporator and Engineering Co. Evaporators. 10,061. June 18.

Furness. Filter-press plate for filtering acid, etc. 9921. June 17.

Grant. Raising liquids of low viscosity. 10,333 and 10,334. June 22.

Grant. Raising liquids, semi-liquids, and slimes. 10,335. June 22.

Morson and Son, and Roy. Means for carrying out catalytic or contact reactions between gases and/or vapours. 10,536. June 26.

Rosborg. Furnaces. 9953. June 17.

Shakespeare. Means for heating or melting solids, semi-solids, or liquids. 10,091. June 19.

Walker. Pulverising-mills. 10,474. June 25.

Wallwin. Gas-heated furnaces or muffles. 10,589. June 27.

Walpole. Means for regulating humidity of drying-kilns, hot-houses, etc. 10,281. June 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

8616 (1917). Allen. Roller grinding-mill. (107,201.) July 3.

8686 (1917). Adam. Method of separating crystals from solutions. (116,749.) July 3.

9465 (1917). Barbet et Fils et Cie. See XVIII.

10,865 (1917). Persch. Recovery by condensation of volatilised solvents. (116,590.) June 26.

10,908 (1917). British Dyes, Ltd., Turner, and Eastwood. Stopper for carboys or the like. (116,798.) July 3.

11,795 (1917). Bouillon. Drying-apparatus for use in drying fruit, herbs, vegetables, seeds, chemicals, fibre, and the like. (116,606.) June 26.

15,082 (1917). Soc. l'Air Liquide. See VII.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Adam. Purification of coal gas. 10,287. June 21.

Barbet et Fils et Cie. Process for dissociation of heavy oils for production of light spirits. 9961. June 17. (France, June 22, 1917.)

Barbet et Fils et Cie. Continuous rectification of petrols, petroleum, benzoles, etc. 10,341. June 22. (France, July 3, 1917.)

Holmes. Treatment of liquor from plants for purifying gases from producers, etc. 10,208. June 20.

O'Brien. Treatment of liquid hydrocarbon and production of valuable by-product. 10,704. June 28.

Ramage. Extracting bitumen, petroleum, etc., from certain materials. 10,437. June 25.

Silberrad. 10,563. See XX.

Somerville, and South Metropolitan Gas Co. Process for removing carbon monoxide, and manufacture of material therefor. 10,767. June 29.

Spicer (Wells). Production of gas from liquid bodies. 10,055. June 18.

Strache. Gasification of carboniferous substances. 10,352. June 22. (Austria, June 28, 1916.)

Wallwin. 10,589. See I.

Willner. Manufacture of gas mantles. 10,068. June 18.

Zwillinger. Converting beehive coke-ovens into by-product ovens. 9977. June 17.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9099 (1917). Bogaerts. See XII.

10,059 (1917). Smith, and Perry and Co. Twin-generator gas plants for manufacture of water-gas containing varying proportions of the component gases. (116,582.) June 26.

15,662 (1917). Toogood, and Dempster and Sons. Manufacture of gas in vertical retorts. (116,831.) July 3.

19,065 (1917). Thompson. See XI.

3417 (1918). De La Devesa. Compounds for increasing the calorific value of coal and like fuel. (116,676.) June 26.

4525 (1918). Easton. Construction of retorts, coke-ovens, and the like. (116,873.) July 3.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Barbet et Fils et Cie. 9961 and 10,341. See II.

Barbet et Fils et Cie. Process for continuous sulphonation of bodies having a benzenic function, such as naphthalene and impure benzenes. 10,117. June 19. (France, June 26, 1917.)

Brewster. Process of nitrating benzol, and apparatus therefor. 10,518. June 26. (U.S.A., Nov. 20, 1917.)

Nitrogen Products and Carbide Co., and Perkin. Treatment of pitch. 10,759. June 29.



O'Brien. 10,704. *See* II.  
 Pohlmann. 10,027. *See* XXII.  
 Ransford (Cassella and Co.). Manufacture of anthraquinone derivatives. 10,285. June 21.

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

Imray (Soc. of Chem. Industry in Basle). Manufacture of disazo-dyestuffs. 10,461. June 25.  
 Kay and Robinson. Manufacture of intermediate products for preparation of dyestuffs. 10,503. June 26.  
 Newton (Bayer und Co.). Manufacture and production of new azo-dyestuffs. 10,682. June 28.  
 Ransford (Cassella und Co.). Manufacture of a colour of the anthraquinone series. 10,545. June 26.

##### COMPLETE SPECIFICATIONS ACCEPTED.

183 (1917). Oman. Method of preparing azo colouring matters from lignine-sulphonic acid or its salts. (103,479.) July 3.  
 8329 (1917). Imray (Soc. of Chem. Industry in Basle). Manufacture of acid wool dyestuffs indifferent to copper. (116,530.) June 26.  
 8420 (1917). Imray (Soc. of Chem. Industry in Basle). Manufacture of chromium compounds of azo-dyestuffs. (116,535.) June 26.  
 8802 (1917). Imray (Soc. of Chem. Industry in Basle). Manufacture of condensation products of gallocyanine dyestuffs with amines. (116,754.) July 3.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Bawtree and Hadfield. Composition for rendering walls of cardboard containers impervious to fluid or otherwise preserving their contents. 10,177. June 20.  
 Clayton, Huebner, Williams, and Manchester Oxide Co. Process of treating celluloses. 10,567. June 27.  
 Clayton, Huebner, Williams, and Manchester Oxide Co. Manufacture of parchmentised paper, vulcanised fibre, etc. 10,568. June 27.  
 Danin. Solution for treating aeroplane canvas, etc. 10,258. June 21.  
 Dreyfus. Manufacture of cellulose esters, etc. 10,174. June 20.  
 Dreyfus. Manufacture of cellulose esters and further transformation products thereof. 10,667. June 28.  
 Dreyfus. Manufacture of cellulose derivatives. 10,755. June 29.  
 Thompson (Underwood and Illston). Treatment of waste liquors from wool scouring, etc. 10,513. June 26.  
 Van Wessen. Manufacture of fibrous material from sawdust and wood waste. 10,616. June 27. (Holland, Dec. 23, 1915.)  
 Van Wessen. Manufacture of fibrous material from wood, etc. 10,681. June 28. (Holland, Jan. 29, 1918.)  
 Wheatley. 9912. *See* XV.

##### COMPLETE SPECIFICATION ACCEPTED.

9034 (1917). Hey. Method of cleaning, carbonising, and sterilising raw wool. (116,763.) July 3.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATIONS.

Calico Printers' Assoc., and Fourneaux. Production of black upon vegetable textile fibres, silk fibres, or mixtures of same. 10,298. June 22.  
 Calico Printers' Assoc., Fothergill, and Wilson. Treatment of insoluble natural gums for production of thickenings for calico, etc., printing and finishing, etc. 10,299. June 22.

##### COMPLETE SPECIFICATIONS ACCEPTED.

9022 (1917). Calico Printers' Assoc., and Fourneaux. Production of black upon vegetable fibres. (116,562.) June 26.  
 18,123 (1917). Crayton. Dyeing of slubbing yarn and the like, and apparatus therefor. (116,652.) June 26.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

Beedle and Bradford. Process for obtaining pure zirconium oxide. 10,314. June 22.  
 Calvert. Manufacture of cyanide and cyanogen compounds. 10,181. June 20.  
 Chance and Hunt, Gidden, and Ragg. Manufacture of arsenic chloride. 10,288. June 21.  
 Davis. Apparatus for concentration of sulphuric, etc., acids. 10,127. June 19.  
 Deutsche Gold- und Silber-Scheideanstalt vorm. Rössler. Manufacture of sodium percarbonate. 10,282. June 21. (Germany, May 18, 1917.)  
 Deutsche Gold- und Silber-Scheideanstalt vorm. Rössler. Manufacture of alkali percarbonates. 10,401. June 24. (Germany, June 25, 1917.)  
 Dutt and Dutt. Process for manufacture and production of manganese oxide. 10,478. June 25.  
 Field, and Metals Extraction Corporation. Purification of metallic solutions. 10,708. June 28.  
 Furness. 9921. *See* I.  
 Hodgkinson. 9952. *See* XXII.  
 Matheson. Manufacture of acetic acid. 10,071. June 18.  
 Matheson. 10,073. *See* X.  
 Wilton. Apparatus for preparation of sulphate of ammonia. 10,452. June 25.  
 Wyld. Apparatus for distillation or treatment of ammoniacal liquors, etc. 10,162. June 20.

##### COMPLETE SPECIFICATIONS ACCEPTED.

7015 (1917). Levin. Manufacture of a boron derivative of pyrophosphoric acid. (116,735.) July 3.  
 15,082 (1917). Soc. l'Air Liquide (Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude). Process for the separation by liquefaction and rectification of the elements of the air. (114,817.) July 3.  
 1403 (1918). Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank. Process for the manufacture of aluminium compounds poor in iron. (112,948.) June 26.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Anderson. Means for coating pottery or earthenware with glazing-mixture. 10,652. June 28.

Baber, Baber, and Jones. Fast-firing furnace for manufacture of bricks. 10,656. June 28.

Rolrant. Glass-melting furnaces. 10,572. June 27. (France, July 12, 1917.)

## IX.—BUILDING MATERIALS.

### APPLICATIONS.

Baber, Baber, and Jones. 10,656. *See* VIII.

Bottomley. 10,213. *See* XIII.

Pennington and Pennington. Cement. 10,148. June 20.

Williams. Manufacture of concrete. 10,377. June 24.

### COMPLETE SPECIFICATION ACCEPTED.

8602 (1917). Dieker (Lambert Process Co.). Method of burning bricks and the like. (116,745.) July 3.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

### APPLICATIONS.

Bennett. Steel-heating furnace. 10,026. June 18. (U.S.A., Aug. 16, 1917.)

Clegg, and Greenwood and Batley. Annealing-furnaces. 10,379. June 24.

Davies and Thomas. Pickling iron and steel plates or sheets. 10,155. June 20.

Harris and Wilkes. Reverberatory furnaces for use in recovery of zinc from dross. 10,553. June 26.

Herdman. Manufacture of mineral wool. 10,283. June 21.

Herdman. Manufacture of slag wool. 10,538. June 26.

Isherwood. Recovery of tin from waste of tin-plate. 10,684. June 28.

Knowles. Recovering scrap steel and dolomite discharged with slag from Siemens open-hearth, etc., furnaces, and disposing of slag. 10,469. June 25.

Lord. Metal-melting furnaces. 10,216. June 20.

Lowenthal. Electrodeposition of zinc and nickel. 10,491. June 26.

Matheson. Process of recovering mercury. 10,072. June 18.

Matheson. Process of oxidising mercury. 10,073. June 18.

Morfeet. Furnaces for melting lead, etc. 10,763. June 29.

Stabilimenti Biak-Ing. Pouchain. Metal alloy containing manganese. 10,209. June 20. (Italy, Mar. 9.)

Stabilimenti Biak-Ing. Pouchain. Metal alloy. 10,210. June 20. (Italy, Mar. 16.)

Sumitomo Chukosho, Ltd. Magnet steel. 10,531. June 26. (Japan, June 15, 1917.)

### COMPLETE SPECIFICATIONS ACCEPTED.

16,309 (1916). Relchinstein. Rust-preventing process. (103,474.) July 3.

15,116 (1917). Bagliardi. Process of annealing high-speed steel. (116,829.) July 3.

15,254 (1917). Kelth and Kelth. Crucible furnaces. (116,636.) June 26.

16,131 (1917). Stabilimenti Biak-Ing. Pouchain. Manufacture of metal alloys containing nickel. (111,290.) June 26.

747 (1918). Bagley. Process and apparatus for removing molten slag from open-hearth steel furnaces by means of air or steam blast. (116,663.) June 26.

## XI.—ELECTRO-CHEMISTRY.

### APPLICATIONS.

C. I. Syndicate, and Jenkins. Electrolytic cells. 10,658. June 28.

East Stratford Works, and Jenkins. Electrolytic cells. 10,205. June 20.

Elektro-Osmose A.-G. (Graf Schwerin Ges.). Charging material to electro-osmotic apparatus for removing liquids from substances. 10,453. June 25. (Germany, Oct. 20, 1917.)

Elektro-Osmose A.-G. Electrodes for electro-osmotic processes. 10,454. June 25. (Germany, Oct. 20, 1917.)

Elektro-Osmose A.-G. Electro-osmotic process for removing water from materials. 10,455. June 25. (Germany, Oct. 20, 1917.)

Elektro-Osmose A.-G. Apparatus for electro-osmotically removing water. 10,456 and 10,459. June 25. (Germany, Oct. 20, 1917.)

Elektro-Osmose A.-G. Apparatus for electro-osmotically removing liquids from materials. 10,457. June 25. (Germany, Oct. 20, 1917.)

Elektro-Osmose A.-G. Apparatus for electro-osmotically removing water from materials. 10,458. June 25. (Germany, Oct. 20, 1917.)

Elektro Osmose A.-G. Electro-osmotically removing water from materials. 10,460. June 25. (Germany, Dec. 12, 1917.)

Lowenthal. 10,491. *See* X.

Peto. Electric storage batteries or accumulators. 10,031. June 18.

White (Industrial Electric Furnace Co.). Electric furnaces. 10,183. June 20.

### COMPLETE SPECIFICATIONS ACCEPTED.

8343 (1917). Muller. Electric insulating materials. (116,532.) June 26.

15,541 (1917). Cotte. Electrical accumulators. (11,107.) July 3.

19,065 (1917). Thompson. Manufacture of carbon electrodes. (116,661.) June 26.

551 (1918). Kilburn (Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank). Manufacture of carbon electrodes. (116,853.) July 3.

## XII.—FATS; OILS; WAXES.

### APPLICATIONS.

Paulin. Manufacture of cleansing material. 10,537. June 26. (France, Oct. 23, 1917.)

Powling and Powling. Apparatus for extracting fat from bones, etc. 10,129. June 19.

Sharp. Manufacture of soap. 10,361. June 24.

Witter. 10,424. *See* XIII.



Wynne. Refining and treatment of oils, fats, greases, etc. 10,302. June 22.

COMPLETE SPECIFICATION ACCEPTED.

9099 (1917). Bogaerts. Method of treating oils. (116,765.) July 3.

XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

APPLICATIONS.

Bottomley. Priming or paint for concrete or cement surfaces, etc. 10,213. June 20.

Bromley. Destructible printing-ink pigment. 10,525. June 26.

Grozier. Reclaiming used printers' roller composition. 10,668. June 28. (Australia, Jan. 3.)

Witter. Process for treating linseed oil residual fatty acids, and utilising same as oil for paint or oxidation for manufacture of linoleum, etc. 10,424. June 25.

XIV.—INDIA-RUBBER; GUTTA-PERCHA.

APPLICATION.

Wheatley. Process for heat-vulcanising rubber-proofed fabric. 9913. June 17.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

La Chevardière de la Grandville, and Roy. Glue. 10,396. June 24. (France, Dec., 10, 1917.)

Wheatley. Process for proofing or impregnating leather, textile material, etc. 9912. June 17.

COMPLETE SPECIFICATION ACCEPTED.

12,800 (1917). Tunnell. Vegetable glue or adhesive. (116,620.) June 26.

XVI.—SOILS; FERTILISERS.

APPLICATION.

Parker and Partington. Fertilisers. 10,528. June 26.

COMPLETE SPECIFICATION ACCEPTED.

8858 and 18,802 (1917). Robinson. Manufacture of fertilisers. (116,758.) July 3.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATIONS.

Calico Printers' Assoc., and others. 10,299. *See* VI.

McLaurin. Process of making dextrine. 10,532. June 26.

McLaurin. Dextrine. 10,533. June 26.

COMPLETE SPECIFICATION ACCEPTED.

6824 (1917). McNeill. Process of and apparatus for macerating crushed sugar cane. (116,521.) June 26.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Desborough, Henley, Reilly, and Thaysen. Manufacture of fermentation products. 10,219 and 10,220. June 20.

COMPLETE SPECIFICATION ACCEPTED.

9465 (1917). Barbet et Fils et Cie. Process and apparatus for distilling in multiple effect alcoholic liquors and the like. (107,599.) June 26.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Adenauer. Preservation of foodstuffs such as sausages, etc. 10,511. June 26.

Beeton, and Trufood, Ltd. 10,057. *See* I.

Bradley, Bradley, Bradley, and Bradley Bros. Bread-improver. 9919. June 17.

Ellis and Gardner. Drying milk, etc., or farinaceous substances in form of liquids or pastes for production of powders, etc. 10,765. June 29.

Monhaupt. Process for purifying casein with recovery of butter fat and lecithin. 10,463. June 25.

Müller. Apparatus for atomising and drying or evaporating milk, etc. 10,062. June 18. (Germany, June 18, 1917.)

Turney. Process for making a new food product of milk. 10,414. June 24.

COMPLETE SPECIFICATION ACCEPTED.

11,397 (1917). Rydberg. Apparatus for the manufacture of margarine. (116,603.) June 26.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Barbet et Fils et Cie. Recovering waste ether and alcohol vapours from atmosphere in factories, etc. 10,116. June 19. (France, June 20, 1917.)

Haworth. Preparation and use of derivatives of arylamines. 10,743. June 29.

Matheson. Process of manufacturing acetaldhyde. 10,070. June 18.

Napp (Hoffman-La Roche & Co.). Process for manufacture of easily-soluble compounds of the CC-dialkylbarbituric acids. 10,180. June 20.

Pegram. Manufacture of preparations of addition compounds of iodine and hexamethylenetetramine. 10,211. June 20. (U.S.A., June 23, 1917.)

Ramy. Manufacture of carbon tetrachloride. 10,034. June 18.

Silberrad. Manufacture of ethylene. 10,563. June 27.

Soc. Chim. Usines du Rhône anc. Gilliard, P. Monnet, et Cartier. Production of acetic anhydride and acetaldhyde. 10,395. June 24. (France, June 22, 1917.)

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Hodgkinson. Production of azides. 9952. June 17.

Pohlmann. Manufacture of picric acid. 10,027. June 18.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Chemical apparatus; Use of wood in —.* A. W. Schorger. Met. and Chem. Eng., 1918, 18, 525—531.

THE results of an inquiry by the U.S. Forest Products Laboratory show that wood is attacked by most of the inorganic acids, a strong solution of the acid dissolving the cellulose, while more dilute acids have slight action on the cellulose but attack the lignin, causing disintegration of the wood. For pickling tanks in which hydrochloric acid or sulphuric acid (up to 5% concentration) is used, cypress wood appears to be the best. Pine, fir, or California redwood is satisfactory for the storage of hydrochloric acid, and particularly if coated with pitch or asphalt, but no wood will withstand for any length of time the action of nitric or sulphuric acids. For chemical processes generally cypress has the longest life, followed by yellow pine. Paraffined hard maple is mentioned as suitable for filter-press plates where acid liquors have to be filtered.

—C. A. K.

*Filter-presses; Washing in —.* D. R. Sperry. Met. and Chem. Eng., 1918, 18, 520—522.

THERE are certain cases in which it is desirable to have the wash-water connection on the filter-press separate from the feed connection. It would be possible to put in a duplicate feed channel composed of a separate set of grommets, but this is too expensive. The use of frames is necessary in the recessed type, and a channel is formed by providing an eye in the upper corner opposite the one occupied by the grommet. In the flush type of plate without grommet feed a separate inlet for wash water is provided by a second similar feed channel in the opposite corner, and where the washings are required separate from the filtrate, they are collected and run off by a channel at the base of the plates, opposite to the outlet cocks, which are turned off during the washing process. One of the disadvantages of simple washing is that a hollow space must be left in the cakes so that the wash water can penetrate the cake thoroughly and this presupposes an even deposit of cake on the plate. In practice this is difficult to attain.

—C. A. K.

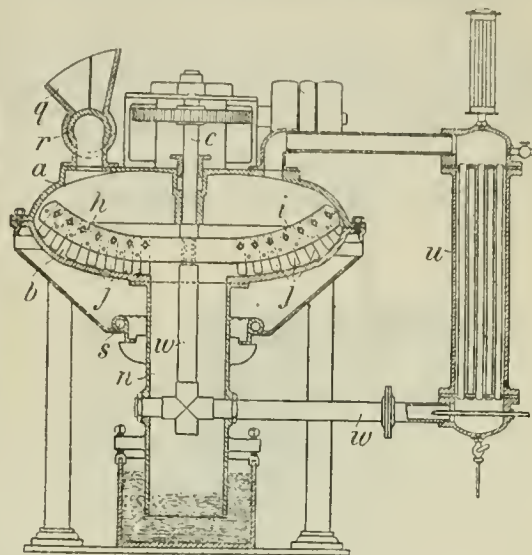
*Corrosion of gas and water pipes.* Medinger. See X.

**PATENTS.**

*Catalytic material employed for the promotion of synthetic chemical reactions; Preparation of —.* H. Lane, Ashford, Middlesex. Eng. Pat. 115,924, June 11, 1917. (Appl. No. S295 of 1917.)

A METALLIC circular air-tight pan, *a*, *b*, is provided with a vertical shaft, *c*, carrying two arms, *h*, *i*, which are curved helically in a horizontal plane. The arms carry sliding teeth, *j*, which rest on the bottom of the pan, and the shaft, *c*, may be rotated in either direction so that the material in the pan may be forced outwards or inwards as desired. The material is fed from a hopper, *q*, provided with an oscillating valve, *r*, and finally discharged through the column, *n*. The pan is heated by an annular burner ring, *s*. Oil or other coating substance for the catalyst is first fed into the apparatus to seal the lower outlet of the column, *n*, and a reducing gas is injected through the pipe, *w*. The raw material is then fed from the hopper, *q*, and heating is continued till water ceases to be condensed in the condenser, *u*. The rotation of the

shaft, *c*, is then reversed, and the material thereby collected towards the centre of the pan and discharged into the coating liquid. The apparatus is



applicable for the manufacture of finely divided reduced nickel for the production of stearin by hydrogenation of oils.—W. F. F.

*Catalytic or contact reactions; Means for carrying out — between gases and/or vapours.* C. S. Roy, Chingford, and T. Morson & Son, Ltd., London. Eng. Pat. 116,151, June 2, 1917. (Appl. No. 7939 of 1917.)

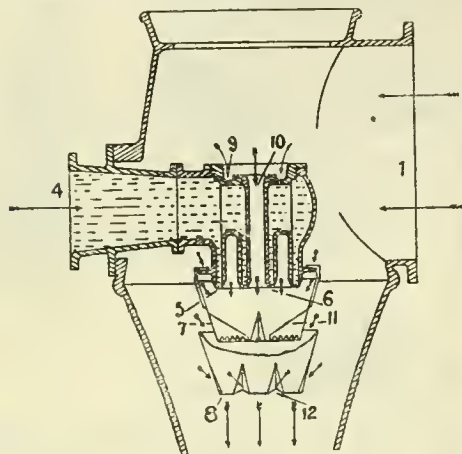
WITHIN a metal casing fitted with a dome-shaped cover with a flue at the top is a series of horizontal tubes of suitable material, *e.g.*, copper, porcelain, for containing the catalyst. The tubes are open at both ends, being fitted into the sides of the casing. If the catalyst is in the form of pumice, pieces of brick, and the like, with or without the addition of other substances, it is disposed partly in shallow boats of half cylindrical form and partly on the floor of the tube itself. The boats are each connected to a header adapted to move in a chamber enclosing the inlet ends of the tubes, the header being controlled by means of a rack and pinion or other suitable means. By moving the header it is possible to adjust the amount of surface of catalyst exposed and also to expose fresh surface to the action of the gases. If the catalyst is in the form of metallic gauze, discs or coils of the gauze are employed, each fitting into a short copper cylinder. The cylinders rest on boats in the tubes and by means of an arrangement of pins on the cylinders fitting into slots of different lengths in the boats—increasing in length from the supply ends of the tubes—the spacing of the cylinders in the tubes can be adjusted from the outside. The tubes may be heated before starting the reaction by means of gas burners at the bottom of the casing, and during the reaction the temperature of the catalyst can be controlled by a supply of steam admitted into the casing. On leaving the tubes, the reaction products are cooled rapidly, *e.g.*, by cooled baffle plates, or by passing through tubes surrounded by water or steam. Means are provided for sampling the reaction products on leaving the tubes and an explosion door may be fitted to the apparatus, and a preheater at the inlet end.

—L. A. C.



*Condensing exhaust steam and other vapours, and setting up an induced current of air or gases to obtain a vacuum; Apparatus for —.* J. Tinsley, Beaufort, Brecon. Eng. Pat. 116,769, June 26, 1917. (Appl. No. 9133 of 1917.)

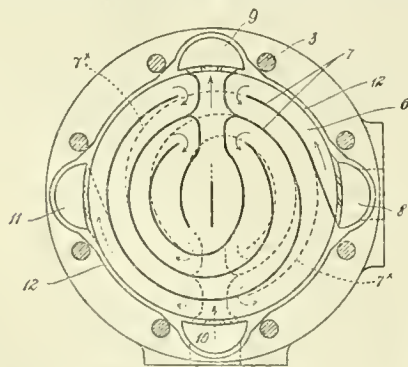
Cold water supplied to the pipe, 4, is discharged through two annular passages, 5, 6, into the conical guides, 7, 8, open at the ends. Exhaust steam or other vapour enters by the pipe, 1, and passes around the water nozzle and also through the central passage, 10, and annular passage, 9. The



water and steam jets thus surround each other alternately. V-shaped pieces are cut out of the lower edges of the guides, 7, 8, and V-shaped blades, 11, 12, extending nearly to the axis, are inserted as shown. The lower edges of the guides are also serrated so that the water and steam are divided into a number of streams and intimately mixed. The steam is thus effectively condensed and a high vacuum is obtained.—W. F. F.

*Transmission or exchange of heat between fluids; Apparatus for —.* A. D. Harrison, Marple, Cheshire. Eng. Pat. 116,786, July 17, 1917. (Appl. No. 10,334 of 1917.)

In a heat-exchanging apparatus, particularly applicable for cooling oil, shown in plan in the figure, a horizontal plate, 6, is provided on its two sides with ribs, 7 and 7<sup>a</sup>, respectively, and with four apertures, 8, 9, 10, 11, formed by a flange, 12. The cooler is built up of a number of such elements superposed, with a flat plate between each pair,



having apertures corresponding to 8, 9, 10, 11, the whole being clamped together by bolts, 3. Each rib, 7, thus forms a circuitous zigzag passage as shown, the two ends of which communicate with the vertical conduits, 8, 9, which form the inlet

and outlet for oil. Similarly each rib, 7<sup>a</sup>, forms a circuitous passage communicating with the common inlet, 10, and outlet, 11, for water, the two liquids flowing in opposite directions on the two sides of the plate, 6. In a modification, the passages for the oil and water may be continuous spirals and the oil inlet and water outlet may be central instead of at the sides. In another modification, the zigzag passage may be rectilinear instead of curved, the common inlets and outlets being at the four corners of the column. In a further modification the passages may be so arranged that the adjacent elements are connected in series instead of in parallel. A method of constructing the circulating elements is also described.—W. F. F.

*Volatile constituents; Method of separating —.*

E. O. Barstow and T. Griswold, jun., Midland, Mich. U.S. Pat. 1,261,005, Apr. 2, 1918. Date of appl., Dec. 21, 1914.

In a method of removing the residual solvent from an extracted material, the material is moved in contact with a counter-current of steam so that a mixture of steam and vaporised solvent is obtained in varying proportions at different points. The sensitive element of a thermostat is placed at a selected point in the current and controls the steam inlet valve so that a constant temperature is maintained at that point.—W. F. F.

*Extracting apparatus.* D. C. Reynolds, Philadelphia, Pa. U.S. Pat. 1,253,470, Apr. 23, 1918. Date of appl., Mar. 27, 1917. Renewed Feb. 27, 1918.

The apparatus comprises a series of extracting tanks, a series of storage tanks, an evaporator tank, and a circulating pump. Each extracting tank has a horizontal partition a short distance above the bottom, with central aperture which is partially obstructed by a trap or seal. The trap permits liquid to flow through the aperture only in one direction. A series of tubes extend through the partition to a point near the top of the tank, and below the partition is a steam coil to heat the contents of the tank. The extracting tanks are filled with the material to be extracted, and water is introduced. The water is heated to boiling by the steam coil and circulates up through the tubes and down over the material in the tank. It may be passed from the lower part of the tank to the storage tanks and thence again introduced into the upper part of the extraction tank by means of the pump. When the extract is sufficiently concentrated, it is passed to the evaporator tank, which is of similar construction to the extraction tank.

—J. H. P.

*Volatile liquids; Process for recovering the vapours of —.* K. E. Barth, Charlottenburg, Assignor to Berlin-Anhaltische Maschinenbau A.-G., Berlin, Germany. U.S. Pat. 1,264,479, Apr. 30, 1918. Date of appl., Apr. 7, 1914.

A GASEOUS mixture containing inflammable vapours is cooled to condense the vapours, the condensed product is separated, and the remainder of the mixture diluted with a suitable gas to insure non-explosiveness.—J. H. P.

*Fire extinguishing compound.* C. J. Strosacker, Assignor to The Dow Chemical Co., Midland, Mich. U.S. Pat. 1,261,067, Apr. 2, 1918. Date of appl., June 6, 1916.

A MIXTURE of carbon tetrachloride and a relatively small amount, e.g., 5 to 10%, of a monohalogen derivative of methane, e.g., monochloro- or monobromomethane or both.—J. H. P.

*Fireproofing solution.* G. E. Ferguson, New York, Assignor to Pyrene Manufacturing Co., D. U.S. Pat. 1,261,736, Apr. 2, 1918. Date of appl., Sep. 30, 1915.

The solution comprises a solvent containing a volatile substance and zinc phosphate and zinc hydroxide. For example zinc phosphate and zinc chloride are treated with sufficient ammonia to convert the chloride into hydroxide and dissolve the hydroxide thus formed.—J. H. P.

*Distillation apparatus; Sectional* —. G. B. Furman, East Orange, N.J. U.S. Pat. 1,265,318, May 7, 1918. Date of appl., Aug. 26, 1917.

The apparatus comprises a still and a number of separators in series. The first separator is connected to the still, and a return connection is provided between this separator and the still, as well as another connecting pipe to return any condensate from the pipe joining two separators to the above-mentioned return connection.—J. H. P.

*Dryer.* F. J. Bowman, Cleveland, Ohio. U.S. Pat. 1,265,298, May 7, 1918. Date of appl., Feb. 24, 1914.

The apparatus consists of a vertical tower containing a series of distributing devices comprising parallel spaced angle plates having the apex of the angles projecting upwards. Some of the plates terminate in inclined end plates resting against the walls of the tower. Hot air is passed upwards through the tower, and a blower discharges the air from the top of the tower downwards through a conduit into a settling tank for the fine material.—J. H. P.

*Liquid separator; Centrifugal* —. C. B. Dalzell, Assignor to D. H. Burrell & Co., Little Falls, N.Y. U.S. Pat. 1,265,061, May 7, 1918. Date of appl., Mar. 27, 1917.

The space between the rotating separator bowl and the stationary case is free from obstructions which would prevent the air from rotating with the bowl. Blades fixed on the lower portion of the bowl cause an upward flow of air between the bowl and the case.—J. H. P.

*Solutions; Method of separating crystals from* —. W. G. Adam, London. Eng. Pat. 116,749, June 16, 1917. (Appl. No. S686 of 1917.)

The hot saturated solution is supplied to a number of transverse troughs at the top of a tower, and overflows on to transverse horizontal plates and then splashes on to a series of transverse bars of glass arranged "in staggered form" and extending down the tower. The solution is cooled by an upward current of air, which is induced by the heating of the air in the tower by the hot solution, and large crystals are formed, which collect in a vessel below. The temperature in the tower may be controlled by regulating the air exit, or by having different amounts of water of crystallisation.—W. F. F.

*Cooling, humidifying, and filtering air or the like; Apparatus for* —. H. F. Brown, Sheffield. Eng. Pat. 116,839, July 14, 1917. (Appl. No. 16,640 of 1917.) Addition to Eng. Pat. 112,653, Jan. 1, 1917 (this J., 1918, 114 A).

In apparatus of the type described in the principal patent, in which a perforated sprinkler arm rotates close to the upper ends of a number of parallel vertical plates, the rotating arm is provided on its trailing side with a flange which covers a number

of spaces between the plates to which water has just been supplied. The passage of air through these spaces is thus prevented until all the free moisture has fallen through the spaces and drained from the plates.—W. F. F.

*Drying [and disintegrating] machine.* T. C. Kelly, Cincinnati, Ohio. U.S. Pat. 1,261,132, Apr. 2, 1918. Date of appl., Nov. 22, 1916.

The machine comprises a long closed conduit through which the material is passed by means of intermeshing screw conveyors. The conveyors are driven separately and a drying fluid is passed through the conduit, which may be heated.

—J. H. P.

*Grinding ores and other materials; Mill for* —. W. H. Janney, Hurley, N. Mex. U.S. Pat. 1,261,398, Apr. 2, 1918. Date of appl., May 1, 1917.

A BARREL-SHAPED container mounted to rotate on horizontal trunnions is provided with a transverse central vertical partition which is double walled and provided internally with partitions forming spiral passages. External scoops are provided at the periphery of the partition, so that as the container rotates, material is taken from a receptacle below and fed into the spiral passages. The material is discharged alternately into the two compartments of the container near the axis, at about the level of the material in the container. Transverse passages are provided in the partition to equalise the material in the two compartments and the ground material is discharged through the hollow trunnions.—W. F. F.

*Roller grinding mill.* H. Allen, Gainesville, Fla., U.S.A. Eng. Pat. 107,204, June 15, 1917. (Appl. No. S616 of 1917.) Under int. Conv., June 15, 1916.

The material is ground between a horizontal cylindrical roller and a stationary surrounding egg-shaped member, the grinding surfaces of both being serrated. The hollow member is supported on two horizontal pivots parallel to the axis of rotation, one on each side at the bottom, the roller being in its lower enlarged portion. One of the pivots is fixed and the other is adjustable by means of a set screw acting on a lever carrying the pivot, so that the hollow member may be adjusted eccentrically to the roller to vary the distance between the grinding surfaces. The hollow member and roller are both made in flat transverse sections which are then built up, and an adjustable baffle-plate is interposed between two sections of the hollow member to vary the rate of feed longitudinally through the mill. The material is fed and delivered at opposite ends of the roller. Radial passages for air may be provided between the sections of the hollow member.

—W. F. F.

*Crushing machine; Rotating-ring* —. E. B. Symons, Chicago, Ill. U.S. Pat. 1,264,347, Apr. 30, 1918. Date of appl., Nov. 9, 1914.

In a crushing machine having a rotating roller within and supporting a ring, the material is fed to the inner surface of the ring near its middle line. The ring is hollowed internally and the ring and roller are given a reciprocating movement to and from one another to crush the material. The material is fed transversely across the face of the ring by a series of separate steps of progressively diminishing length and velocity from the centre outwards.—W. F. F.



*Gas-dryer.* H. A. Brassert, Chicago, Ill. U.S. Pat. 1,264,263, Apr. 30, 1918. Date of appl., Dec. 8, 1915.

Gas enters tangentially at the top of a vertical casing and passes down outside a concentric vertical pipe. A number of rows of baffles, arranged circularly, project downwards from the bottom of the pipe and are supported from the wall of the casing so as to divert the gas into the inner pipe through which it passes upwards.—W. F. F.

*Dust catcher for cleaning gases.* C. Heine, Düsseldorf. Ger. Pat. 302,973, Nov. 21, 1916. Addition to Ger. Pat. 230,182 (see Fr. Pat. 419,933; this J., 1911, 120).

THE gas enters at the bottom of the annular space between two concentric vertical cylinders, flows upwards in a spiral path, and leaves through the inner cylinder which it enters at the top. In its passage, the gas beats against vertical radial baffles fixed alternately to the inner and outer cylinder, whereby dust particles are arrested and fall into the conical base of the apparatus.—H. J. H.

*Gases; Apparatus for the electrical separation of suspended particles from —.* G. A. Krause, München. Ger. Pat. 303,078, June 1, 1913.

THE gas is passed through a casing closed below by a metal plate on which a series of glass jars is placed. Plate electrodes connected to the positive pole of a source of current pass through the top of the casing, but insulated therefrom, and project into the jars. The metal plate below the jars is connected to the negative pole of the source of current, so that the jars act as condensers of the Leyden jar type. Suspended particles in the gas become charged and are deposited in the jars.

*Filtering apparatus and system.* W. C. Graham, Loveland, Colo. U.S. Pat. 1,264,635, Apr. 30, 1918. Date of appl., July 26, 1915. Renewed Sep. 25, 1917.

SEVERAL tubular shafts extend through a casing and carry rotary filter elements, the interiors of which communicate with the interiors of the shafts. The filter elements on each shaft pass between and close to those on the adjacent shaft, so that material adhering to the filters is displaced during rotation.

—J. H. P.

*Engine for low temperature expansion.* E. A. W. Jefferies, Assignor to Jefferies-Norton Corporation, Worcester, Mass. U.S. Pat. 1,264,871, Apr. 30, 1918. Date of appl., Dec. 1, 1915. Renewed Jan. 19, 1918.

THE cylinder of an engine in which compressed or liquefied gases are made to expand at a low temperature for the production of extreme cold, consists of a metal casing provided with a lining having a low heat capacity, so as to prevent material absorption of heat by the surfaces with which the expanding gases come in contact. This lining is mainly composed of fibrous material and a binding agent, with or without the addition of graphite, and may be compounded by the application of heat and pressure.—C. A. M.

*Centrifugal machine.* H. Delvenne, Grevenbroich. Ger. Pat. 302,664, Oct. 3, 1916.

THE upper end of the vertical shaft of the centrifuge carries a hub which is connected to the shaft of the motor above by a ball and socket joint; the upper end of the motor shaft works in a collar bearing. The motion is transmitted by means of a flanged coupling disc secured to the lower end of the motor shaft and provided with rollers which fit in grooves in the hub on the centrifuge shaft.

*Liquids; Machine for extracting — from various materials.* C. D. Jenkins, Assignor to Peat Coal Ltd., Boston, Mass. U.S. Pat. 1,265,206, May 7, 1918. Date of appl., May 1, 1915.

A HOLLOW cylinder is formed of sections of wood having the grain perpendicular to the cylindrical surface. An eccentric rotating member is arranged within the cylinder so as to press the material against the wall and force the liquid through the pores.—W. F. F.

*Settling tank.* W. E. Saxe, Inglewood, Cal. U.S. Pat. 1,265,256, May 7, 1918. Date of appl., Oct. 11, 1915.

THE material to be treated is fed through an automatically controlled discharge outlet in a receiving trough into the conical settling tank, which is provided with means for spreading or retarding the movement of the material. The tank is supported by pivoted levers with counterweights, and is provided with an outlet valve which may be locked or unlocked automatically. The liquid outlet is arranged under the spreader, near its centre, so that the outlet flow of liquid is accelerated and the lighter material held in suspension after the heavy matter has been deposited. Converging partitions lead to the liquid outlet and prevent a whirling action.—J. H. P.

*Furnace.* A. W. Moyer, Belmar, N.J. U.S. Pat. 1,264,961, May 7, 1918. Date of appl., Nov. 5, 1917.

A FURNACE is provided with a removable arched top, and is heated by burners in combustion chambers outside the side walls of the heating chamber and communicating with it by lateral passages through the walls. The hot gases are discharged from the lower part of the heating chamber. The furnace is supported in a cradle comprising transverse girders beneath the hearth, vertical stays to prevent spreading of the side walls, and inclined bracing members attached to the stays and girders. This arrangement does not prevent the removal of the furnace top.—W. F. F.

*Bleaching earth [fullers' earth]; Process for increasing the decolorising power of —.* L. Kern, Hamburg. Ger. Pat. 305,896, Feb. 2, 1917. Addition to Ger. Pat. 304,076.

THE dried ground earth is treated with gaseous acid (e.g. sulphur dioxide) instead of with liquid acids as described in the original patent. After the treatment the earth may be freed from the sulphur dioxide, etc., by means of a current of warm air.

—C. A. M.

*Filling material for reaction towers, washing towers, and the like.* H. Petersen, Berlin-Steglitz. Ger. Pat. 302,753, Oct. 22, 1913.

THE filling material consists of hollow rings having the shape of a regular polygon with an odd number of sides, and provided with undulating grooves or channels.

*Gases or vapours; Process of separating — from gaseous mixtures.* P. Beck, Nordhausen. Ger. Pat. 304,603, June 12, 1917. Addition to Ger. Pat. 302,692 (this J., 1918, 328 A).

GASEOUS mixtures are treated at the ordinary or in some cases at higher temperatures with lignite or lignite products which have been freed from gas. Gases such as sulphur dioxide, hydrogen sulphide, ammonia, chlorine, and cyanogen, are absorbed by lignite which has been freed from gas, and may be recovered by steaming, exhaustion with a pump, or by the passage of an inert medium through the material.—C. A. M.

*Solid substances; Apparatus for the continuous solution of —.* H. Eberhardt, Wolfenbüttel. Ger. Pat. 304,802, Dec. 16, 1916. Addition to Ger. Pat. 302,641 (this J., 1918, 360 A).

The vessel containing the solvent is enlarged at the top, and the enlargement carries a ring-shaped channel. The heating device is separated from the surrounding wall of the cylinder by a protective jacket, thus forming an annular space in which the sludge can subside. This sludge is removed together with the undissolved residue from the cylinder by means of a screw conveyor at the base of the apparatus.—C. A. M.

*Powdery or granular material; Process and apparatus for treating — in a shaking vessel.* H. Stegmeyer. Ger. Pat. 305,237, Dec. 8, 1915. Addition to Ger. Pat. 303,475 (see page 427 A).

The shaking vessel contains a vertical partition extending nearly to the end of the chamber, which is also provided at one end with two pipes communicating with a deeper receiver. One of these pipes, which has a valve to prevent the liquid passing back, conducts the liquid from the shaking chamber, whilst the other conveys it back again. The whole apparatus or the shaking chamber only may be heated by means of a heating jacket or other device.—C. A. M.

*Salt solutions or other liquids; Reflux condensing apparatus for cooling —.* Maschinenfabrik Thyssen und Co., A.-G., Mülheim. Ger. Pat. 305,240, Dec. 29, 1916. Addition to Ger. Pat. 302,887 (this J., 1918, 360 A).

The distribution channels of a reflux cooling apparatus of the type described in the original patent are directly heated by the vapours within the apparatus. These vapours may receive their preliminary heating from a heating device below or near the distribution channels, or may be heated by the introduction of hot gases.—C. A. M.

*Refrigerating machine; Rotary —.* H. Lanz, Mannheim. Ger. Pat. 305,413, Apr. 2, 1915.

The machine consists of two horizontal cylindrical vessels on the same axis, the liquefier revolving in a vat of cooling water and the vaporiser in a vat of brine. The compressor is situated radially inside the liquefier, and the compressor pistons are reciprocated by an auxiliary shaft parallel to the axis of rotation and operated from the main shaft at double the speed by means of gear-wheels. The liquefier is charged with lubricant and a liquid refrigerating medium, e.g. liquefied sulphur dioxide, and the machine revolved. The casing of the compressor being freely suspended from bearings on the main shaft does not revolve; the pistons are operated by the auxiliary shaft and lubricated in a bath of oil. The liquefied gas is forced through an axial passage and regulating valve into the vaporising vessel and cools the surface of the latter, while gas is withdrawn by the pistons from the vaporising vessel through another axial passage and reliquefied in the compressor.—J. F. B.

*Refrigeration; Process and apparatus for —.* J. C. Bertsch, Edgewood Park, Pa. U.S. Pat. 1,245,037, May 7, 1918. Date of appl., Jan. 26, 1917.

The apparatus comprises an evaporator connected to a condenser, and a jet absorber receiving vapour from the evaporator and weak liquor at high pressure. A jet compressor receives the solution from the absorber and delivers it into a separator.

The weak liquor is cooled and returned from the separator to the absorber, and the gas passing from the separator to the condenser is compressed.—J. H. P.

*Gas-compression plants for refrigerating and other purposes.* A. Heane, Blayney, N.S.W. Eng. Pat. 111,081, May 4, 1917. (Appl. No. 6373 of 1917.) Under Int. Conv., Jan. 11, 1917.

*Centrifugal drying machines.* W. P. Thompson, London. From G. H. Elmore, Philadelphia, U.S.A. Eng. Pat. 115,909, May 26, 1917. (Appl. No. 7612 of 1917.)

SEE U.S. Pat. 1,235,193 of 1917; this J., 1917, 996.

*Elevating or lifting acids or the like; Apparatus for —.* T. Bradbear, Birmingham. U.S. Pat. 1,261,351, Apr. 2, 1918. Date of appl., June 18, 1917.

SEE Eng. Pat. 109,522 of 1916; this J., 1917, 1170.

*Refrigeration; Process of —.* E. A. W. Jefferies, Worcester, Mass., Assignor to Jefferies-Norton Corporation. U.S. Pat. 1,264,807, Apr. 30, 1918. Date of appl., Jan. 26, 1915.

SEE Eng. Pat. 106,675 of 1916; this J., 1917, 858.

*Refrigeration; Process of —.* F. E. Norton, Worcester, Mass., Assignor to Jefferies-Norton Corporation. U.S. Pat. 1,264,845, Apr. 30, 1918. Date of appl., Mar. 3, 1915.

SEE Eng. Pats. 106,676 and 106,677 of 1916; this J., 1917, 859.

*Spraying electrode for the electrical purification of air or gases containing dust or fog.* Ger. Pat. 305,363. See XI.

*Densimetric methods and appliances.* Eng. Pat. 115,662. See XXIII.

## II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coke breeze; Combustion of — on chain grates with forced draught.* O. Wirmer. Z. Ver. deuts. Ing., 1917, 61, 818–824. Z. angew. Chem., 1918, 31, Ref., 134.

At the Badische Anilin und Soda Fabrik it has been found possible to admix 25% of coke breeze to the fuel burnt in chain grates without further modification. Above this proportion forced draught is necessary, the pressure being limited to that necessary to overcome the resistance of the fuel bed, thereby avoiding the passage of ash into the flues. Under such conditions coke breeze alone has been successfully used. The best results were obtained with a mixture of 80% of coke breeze with 20% of nut coal, when a boiler efficiency of 80% was attained. To secure the high temperature on the hearth required for the ignition of the breeze a long low furnace arch is necessary.—H. J. H.

*Coke ovens; By-product — and town's gas supply.* E. Rees, Wales and Monmouth Dist. Inst. of Gas Eng., May 29, 1918. Gas J., 1918, 142, 427–429.

The use of coke-oven gas for town supply where possible is advocated on national and local grounds.



By taking such supplies in bulk a local gas undertaking can meet increased demands with but small outlay of capital. Where the ovens are fitted with duplex mains it is preferable to take the richer portion of the coke-oven gas only. Otherwise it is advocated that the gas should be taken unstripped from a point just before the benzol scrubbers. The principal objection to coke-oven gas is the high nitrogen content, but, even with this, the newer requirements in respect of calorific value can be met.—H. J. H.

*Effluents from ammonia-recovery plant of coke ovens.* M. Marsson and R. Weidert. Mitt. Kgl. Landesanstalt f. Wasserhyg., 1917, 237—284. J. Gasbeleucht., 1918, 61, 200.

THERE are three effluents to consider, that from the stills, the liquid condensed from the gases leaving the saturator, and the effluent from the Claus sulphur-recovery kilns, of which the first is by far the most important. It amounts to 40 to 60 litres per ton of coal in direct processes and 200 to 450 litres per ton in indirect processes. In experiments on the disposal of still effluents from gas works made in Berlin during the period 1907—1910, it was found that the bulk of the suspended matter could be separated by settling for 1 hour and 97—99% after 24 hours. This left the dissolved matter unchanged. The yellow colour of the effluent darkened with time according to the intensity of the light and the alkalinity of the effluent. Treatment with flue gases was effective in precipitating dissolved lime. Attempts to remove other injurious constituents by chemical precipitants had no practical success. Filtration through lignite cinders produced an effluent which was non-poisonous to fish life. When applied to a road the effluent from stills had some beneficial effect in suppressing dust.

—H. J. H.

*[Gas] purification; Notes on oxide* —. H. Tindale. Gas J., 1918, 142, 554—555.

LABORATORY tests showed that the "oxides" available at Sydney, N.S.W., contain varying amounts of iron oxide in a form unsuitable for the purification of coal gas, since the reaction with hydrogen sulphide is slow, and the products of subsequent revivification include an appreciable amount of sulphur permanently combined with the oxide. Revivification is also slow, and, when effected *in situ*, requires an excess of oxygen—necessitating the admission of air, and consequently increasing the proportion of nitrogen in the gas. For efficient sulphiding of the oxide a temperature of not less than 80° F. (about 27° C.) is required; but, owing to the slow reaction, this temperature is not maintained by the heat of formation of iron sulphide, and in practice is supplemented by the heat of formation of ammonium salts. The latter reduce the purifying efficiency of the oxide and also cause excessive back pressure in the purifying system.

—W. E. F. P.

*Gasoline; Determination of unsaturated hydrocarbons in* —. E. W. Dean and H. H. Hill. U.S. Bureau of Mines, Tech. Paper 181. 22 pages.

THE proportion of unsaturated hydrocarbons in motor fuels appears not to affect the commercial value, but its determination is often useful as a means of determining the source and method of production of a given sample. Tests made by the authors show that the best methods are the determination of the iodine value, and absorption by strong sulphuric acid. Although the chemical mechanism of these two reactions is different, yet, in the case of cracked gasolines, the ratio of iodine value to percentage acid absorption lies between

fairly close limits. The average figure is 6.5, and most of the values fall between the limits of 6 and 7. The authors found the Hanus iodine method very satisfactory and accurate when the necessary precautions were observed. For such volatile liquids as gasoline it is necessary to deliver the sample below the surface of the chloroform or carbon tetrachloride by means of a capillary pipette. The sulphuric acid absorption method is rapid, reliable, and of fair accuracy, and is highly recommended as satisfactory for general use. The sample is run into a clean, dry, 6-inch Babcock cream bottle, cooled for a minute or two in ice-water, and then 200% by vol. of concentrated sulphuric acid is poured in, down the side of the bottle. This is then closed by a rubber stopper and the contents shaken, first slowly, then vigorously with a rotary motion for several minutes. Sulphuric acid is now added until the surface of the liquid is about level with the upper graduation mark, and the mixture allowed to stand overnight. A more speedy separation is obtained by revolving the stoppered bottle at once in a centrifuge for two or three minutes at a speed of 500 to 1000 r.p.m. Sufficient acid is then added to bring the level up to the lower graduation mark, and the contents are again centrifuged to complete the separation. More acid is added, and the residual volume of gasoline is read. The results have a possible error of 1 to 2%. Under the conditions of test, aromatic hydrocarbons are substantially unaffected. The "acid heat test" (determination of Maumené numbers) proved unreliable, especially for highly unsaturated mixtures. The bromine absorption method proved less satisfactory than the iodine method, although giving approximately equivalent results. There was more difficulty in obtaining concordant duplicate results, more time was required, and there appeared to be no compensating advantages.—T. St.

*Emulsions; Water-in-[hydrocarbon] oil* —. A. U. M. Schiaepfer. Chem. Soc. Trans., 1918, 113, 522—526.

PICKERING (this J., 1908, 88) showed that it is possible to prepare emulsions of hydrocarbon oils in water by means of precipitates which are insoluble in both liquids and are moistened more by water than by oil. To ascertain if solid particles which are more readily wetted by oils than by water would bring about the emulsification of water in oils, experiments were made with American carbon black (gas black, soot), thoroughly extracted with hot benzene, and such emulsions have been prepared. The emulsion obtained with 30 c.c. of water, 70 c.c. of kerosene, and 1 gm. of carbon was found to mix freely with kerosene but not with water. Under the microscope it was seen that the droplets of water were covered with a dark-brown skin of adsorbed particles of carbon: those particles which were not adsorbed formed aggregates in the kerosene phase. The emulsion was liquid and excess of kerosene separated after standing for one day, leaving a viscid emulsion which was stable. An emulsion made with 70 c.c. of water, 30 c.c. of kerosene, and 1 gm. of carbon also mixed freely with kerosene but not with water: this emulsion would not flow out of an inverted test-tube, and was stable for over a week. The order in which the ingredients are added makes no difference to the final result. The viscosity of these emulsions rises as the proportion of water is increased; when prepared by Pickering's method by alternately filling and emptying a garden syringe, the viscosity of the mixture rises as the number of strokes of the syringe, indicating the degree of mixing, is increased. In no case was it found possible to prepare an emulsion of oil in water by the use of carbon. "Carron oil," which

is prepared with lime water and linseed or rape oil, is another example of water-in-oil emulsions, whereas embrocations are of the oil-in-water type.

—J. F. B.

*Artificial asphalts; Chemical composition of* —. J. Marcusson. Z. angew. Chem., 1918, 31, 113—115, 119—123.

The most important artificial asphalts are composed of petroleum residues, lignite pitch, fat pitch, and coal tar and coal tar pitch, and these may be differentiated by the methods devised for natural asphalts (this J., 1916, 1099). *Petroleum residues*:—The residues from the distillation of petroleum with superheated steam resemble natural asphalts most closely both in composition and applicability. Whilst natural asphalts frequently show a relatively high proportion of mineral constituents (e.g. Trinidad asphalt, 33 to 55%), petroleum asphalts usually contain less than 1%. Free asphaltogenic acids are not present in petroleum asphalts, since they have been converted by the heat into anhydrides. For this reason the acid value of petroleum asphalts is low (e.g. less than 1 as against 2.8 to 15.4 for natural asphalts). Samples of petroleum asphalt of various origin contained from 2.7 to 3.4% of asphaltogenic anhydrides, whilst the saponification value was usually about 10, as compared with 29 to 36 in the case of natural asphalts. The proportion and character of the oily constituents depends upon the extent to which the distillation was carried, but even hard, friable petroleum residues invariably contain a considerable proportion of oil. For example, a hard sample of Californian origin contained 48%, whilst soft petroleum asphalts contained over 80% of oil. This oil resembles heavy cylinder oils in its composition and behaviour, has a similar iodine value (about

*Petroleum asphalt from acid resins*:—On diluting the sulphuric acid which has been used for refining petroleum products a resinous oily mass separates, which was first used as an asphalt substitute by Jenny (Ger. Pat. 3577; Dingler's Polyt. J., 233, 285). A sample of a product purified by washing and neutralisation with lime contained 15% of substances, mainly calcium compounds, insoluble in benzene. The soluble constituents, which resemble soft asphalt, had a saponification value of 18, and yielded 7% of solid black acids which were nearly insoluble in petroleum spirit. The unsaponifiable matter was 17.3%, asphaltic substances insoluble in petroleum spirit, 8.7%, and petroleum resins and oil, 52%. The oil in acid resin asphalts is of the type of machine oils, and is free from paraffin wax. These artificial asphalts are characterised by the presence of sulphonated addition compounds, which may be separated by treating the bitumen with petroleum spirit. The insoluble portion is not decomposed by boiling with potassium hydroxide solution, but when boiled with dilute hydrochloric acid is decomposed with liberation of the sulphuric acid. Acid asphalts are mainly used in the varnish industry; owing to their slight elasticity they are much less suited for building purposes. *Lignite tar pitch*:—The crude tar obtained in the dry distillation of lignite is not used directly in the asphalt industry. It contains relatively little asphaltic substances, and consists in the main of paraffin wax and oil, which must be separated by a second distillation. Frequently the distillation is carried only far enough to obtain the softer "goudron." Another lignite product consists of the acid resin pitch, and is obtained by distilling the acid resins formed during the purification of the oils and paraffin wax with sulphuric acid. Typical samples of these products had the following characters:—

	M. pt.	Acid value.	Saponif. value.	Insol. in hot benzene.	Saponifiable substances	Asphaltenes.	Resin melting below 100° C.	Oily substances.	
								%	Iodine value.
Lignite goudron ..	C 31	0.5	2.8	% 11.0	% 1.8	% 24.0	% 11.0	48.0	8.4
Acid resin pitch ...	88	4.4	13.4	20.0	6.0	36.0	8.1	26.0	26.1

18), and contains as a characteristic constituent a considerable amount of a high-melting paraffin wax, whereas the oil from natural asphalts contains only a small amount (usually less than 1%). The quantity of oil in natural asphalts is much less than in the case of petroleum asphalts, and after removal of resinous substances and unsaturated hydrocarbons by treatment with sulphuric acid, is fluid, whereas the oil from petroleum asphalts remains semi-solid after such treatment. The following table shows the comparative composition of typical natural and petroleum asphalts:—

The phenols of lignite tar pitch, unlike those of lignite tar oils, are partially insoluble in petroleum spirit, and this characteristic affords a means of distinguishing between lignite pitch asphalt and natural asphalt. *Fat-distillation residues*:—These are characterised by the presence of fatty compounds. They may contain variable proportions of free fatty acids, hydroxy-fatty acids, lactones, hydrocarbon compounds, asphaltic compounds, ketones, traces of neutral fat, sulphur compounds, and copper soaps. *Coal tar and coal tar pitch* are composed of insoluble carbonaceous substances (a

BITUMEN FROM	Free asphaltogenic acids.	Inner anhydrides of asphaltogenic acids.	Asphaltenes.	Petroleum resins.	Oily substances.
	%	%	%	%	%
<i>Natural Asphalts</i> —					
Trinidad asphalt ... ..	6.4	3.5	37.0	23.0	31.0
Refined Berinda asphalt ... ..	3.9	2.0	35.3	14.4	39.6
<i>Petroleum Asphalts</i> —					
Kansas petroleum asphalt (medium) ...	absent	3.0	24.0	11.0	62.0
German petroleum residue (soft) ... ..	"	4.0	4.4	8.6	83.0
Russian petroleum pitch ... ..	"	2.0	15.5	16.1	66.0



mixture of carbon with coal dust or coal-like resins) and of soluble tar bitumen, which contains, in addition to phenols, bases, and hydrocarbons, three distinct tar resins, respectively soluble in benzene, chloroform, and pyridine. These resins are aromatic compounds of high molecular weight. The resin soluble in benzene absorbs oxygen from the air, and is transformed into the other two resins. On this property mainly depend the drying power and resinification capacity of coal tar. (See also this J., 1911, 480; 1913, 646; 1914, 739.)—C. A. M.

*Analysis of spent oxide.* Hottenroth and others. See VII.

*Corrosion of gas- and water-pipes.* Medinger. See X.

*Determination of albuminoid ammonia in liquids containing gas liquor.* Stephenson. See XIX.

*Gas analysis with very small quantities.* Hamburger and Koopman. See XXIII.

#### PATENTS.

*Firing-chamber for powdered fuel.* W. D. Wood, New York, Assignor to Fuel Savings Co., Allentown, Pa. U.S. Pat. 1,266,088, May 14, 1918. Date of appl., Mar. 23, 1914.

THE fire-box of the furnace has a depressed auxiliary chamber the sides of which slope downwards and inwards to the bottom, the latter being inclined and having an opening at the lower end for removing slag and other accumulations. Overheating at the junction of the "mud ring" and side walls of the fire-box is prevented by means of an air space between the part in question and the refractory lining of the auxiliary chamber, a perforated plate being provided below the air space.

—W. E. F. F.

*Coal; Apparatus for burning pulverised* —. V. Z. Caracristi, Bronxville, and J. E. Muhlfeld, Scarsdale, N.Y. U.S. Pat. 1,265,172, May 7, 1918. Date of appl., Sept. 1, 1916.

PULVERISED fuel is supplied tangentially to a horizontal cylindrical mixing chamber so as to pass through it in a helical path, and air for combustion is also supplied. The burning mixture emerging from the cylinder meets a rising column of air which has been supplied through a horizontal chamber below the mixing chamber, having its outlet end curved upwards in front of the mixing chamber so as to form a combustion chamber. Combustion is completed in this chamber by means of the additional supply of air.—W. F. F.

*Gas; Manufacture of — in vertical retorts.* H. J. Toogood, and R. Dempster and Sons, Ltd., Elland. Eng. Pat. 116,831, Oct. 27, 1917. (Appl. No. 15,662 of 1917.)

IN a vertical gas retort, in which steam is injected into the lower part, the steam is superheated by the surplus heat in the setting. Producer gas flues and waste gas or carbon dioxide flues are arranged vertically in the wall of the retort close to the vertical conduit in the wall through which the steam passes to the bottom of the retort. The steam is injected through a small pipe in the top of the conduit, and induces a flow of producer gas and waste gas through small valved pipes opening close to the steam outlet, and connected to the respective flues.—W. F. F.

*Retorts, coke ovens, and the like; Construction of* —. R. W. Easton, London. Eng. Pat. 116,873, Mar. 14, 1918. (Appl. No. 4525 of 1918.)

A HORIZONTAL retort, coke oven, or the like for the continuous distillation of coal, shale, peat, or other carbonaceous or nitrogenous substance, consists of a casing of metal, fireclay, or firebrick, or part metal and part fireclay or firebrick, built into a suitable furnace setting. Two or more intermeshing helical screws constructed as described in Eng. Pat. 109,663 are arranged within the casing and rotated by gearing at one or both ends. The material is fed from a hopper at one end and discharged from a hopper at the other end, and gases are drawn off from a pipe at the top at the middle of the retort. In a modification the screws are mounted in a second casing at the entrance to the retort, the casing and operating gearing being outside the furnace setting.

—W. F. F.

*Water-gas; Apparatus for the production of* —. Soc. d'Exploit. des Appareils Rateau, Paris. Eng. Pat. 112,942, Jan. 19, 1918. (Appl. No. 1114 of 1918.) Under Int. Conv., Jan. 24, 1917.

IN apparatus for producing water-gas, of the type in which the fuel in the generator is submitted alternately to blasts of air and steam, the air is injected by a fan operated by a steam turbine. The exhaust steam from the turbine passes to a steam accumulator so that it may be used for the steam blast when the turbine and fan are stopped. An auxiliary pipe for the direct supply of steam from the boiler may also be provided.—W. F. F.

*Coal gas; Manufacture of* —. Goodall, Clayton, and Co., Ltd., and T. Settle, Leeds. Eng. Pat. 115,997, Sept. 7, 1917. (Appl. No. 12,834 of 1917.)

A SERIES of vertical retorts is connected through valves at the bottom to a common highly heated horizontal passage, and through valves at the top to a common hydraulic main. When the fuel in the first retort is highly incandescent, superheated steam is admitted to the top and passes down through the retort to the passage below. The resulting water-gas passes upwards through the second retort which is at a distillation temperature, so that the distillation products are diluted and a large volume of gas of high calorific power is obtained. The process is then repeated with the second and third retorts and so on.—W. F. F.

*Gaseous fuel; Process of manufacturing* —. J. R. Rose, Edgeworth, Pa., U.S.A. Eng. Pat. 116,406, Aug. 3, 1917. (Appl. No. 11,238 of 1917.)

SEE U.S. Pat. 1,252,033 of 1918; this J. 1918, 144 A. Hydrogen may be added to the mixture of steam and fluid hydrocarbon, and the final mixture is subjected to a temperature not materially lower than 1609° F. (870° C.).

*Hydrocarbon gas and method of producing the same. Hydrocarbon gas cartridge.* L. Wolf, Assignor to The Thermalene Co., Chicago Heights, Ill. U.S. Pats. (A) 1,265,573 and (B) 1,265,574, May 7, 1918. Dates of appl., June 16 and Sept. 13, 1915.

(A) ACETYLENE is generated from a carbide and the heat evolved is used to gasify a hydrocarbon oil. The gases are mixed and then purified by cooling. (B) In the method described in (A) the gas is produced from a cartridge composed of a suitable carbide and a vehicle carrying the oil to be gasified.

—W. F. F.

*Valves for controlling, reversing, and by-passing the flow of gas through purifiers, condensers, and the like.* R. Dempster & Sons, Ltd., J. E. Horsfall, and H. Lumb, Elland. Eng. Pat. 115,936, June 21, 1917. (Appl. No. 8914 of 1917.)

VALVES are arranged in a casing outside each end of the purifier in such a manner that the flow of gas in the purifier may be from top to bottom, from bottom to top, from the middle to top and bottom respectively, or from both top and bottom to the middle.

*Ammonia or its salts; Manufacture of* — [by direct recovery process]. E. Oré, Caen, France. Eng. Pat. 115,449, Mar. 7, 1917. (Appl. No. 3399 of 1917.)

In a process for direct, total recovery (as sulphate) of ammonia generated by carbonising coal, etc., the hot gas passes through a condensing column which is operated under such conditions that the whole of the free ammonia is carried forward by the gas. The condensed aqueous liquor containing ammonium salts is treated with lime in a column without the circuit traversed by the gaseous products of carbonisation, and the gaseous ammonia set free is returned to the condensing column and carried forward by the gas. After leaving the condensing column the gas passes through surface condensers and a tar separator, and thence into a saturator to recover the ammonia as sulphate.—W. E. F. P.

*Tar from producer gases, such as brown coal producer gas; Process and plant for recovering — in two portions particularly suitable for dehydration.* Berlin-Anhaltische Maschinenbau A.-G. Ger. Pat. 301,459, Apr. 4, 1915.

Hot producer gas is passed through a washer tar-extractor, followed by a spraying fan. The tar from the extractor is heated by a steam coil to about 70° C. until homogeneous, then the lower portion is allowed to cool slowly to 45°–50°, while the temperature of the upper portion is maintained at 70°, whereby the heavy tar is caused to sink, and the water to rise; on further cooling less than 5% of water remains in the tar. The tar from the spraying fans is similarly dehydrated, but being lighter than water, owing to the presence in it of small bubbles of gas, the tar rises to the top in the dehydrator. The gas may alternatively be expelled from the light tar by heating it under vacuum, whereby it is rendered heavier than water, and responds to treatment in the dehydrator in the same way as the tar from the extractor.

*Petroleum hydrocarbons; Art of cracking* —. J. S. Cosden and J. W. Coast, jun., Tulsa, Okla., Assignors to The Process Co. U.S. Pat. 1,261,215, Apr. 2, 1918. Date of appl., Dec. 26, 1916.

PETROLEUM hydrocarbons are cracked and vaporised and the vapours confined at a pressure greater than 50 lb. per sq. in. Natural gas, heated to a temperature higher than that of the confined vapours, is forced into the vapours, and a relatively cool high-boiling hydrocarbon liquid is forced into the mixture. The vapours are then led into a condenser to obtain a substitute for gasoline.—L. A. C.

*Petroleum; Process for the distillation of* —. C. E. Lepley, Bayonne, N.J. U.S. Pat. 1,261,410, Apr. 2, 1918. Date of appl., July 5, 1916.

PETROLEUM is distilled until gasoline and lighter products have been driven off and the temperature is then raised to approximately that of the boiling point of kerosene. A stream of highly heated

petroleum vapour is then injected under pressure into the delivery region of the still. When the lighter products formed at this stage have been driven off, the contents of the still are subjected to a temperature suitable for the distillation of kerosene or other heavy products.—L. A. C.

*[Petroleum] distillation; Process of — and apparatus therefor.* G. L. Prichard, Port Arthur, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,264,435, Apr. 30, 1918. Date of appl., June 28, 1916.

A CONTINUOUS current of petroleum oil is passed through a series of stills, each still being provided with a vapour outlet and dephlegmating column. The oil, as it is fed into each still, passes down the corresponding dephlegmating column, meeting the ascending vapours. In each still are means whereby the oil is caused to flow in a tortuous passage.

—L. A. C.

*Mineral oils; Apparatus for refining* —. H. D. Lorch, Louisville, Assignor to Lorch Construction Co., Tulsa, Okla., and S. Z. Lorch, Louisville, Ky. U.S. Pat. 1,264,668, Apr. 30, 1918. Date of appl., Sept. 7, 1916. Renewed Nov. 24, 1917.

MINERAL oils are vaporised and the vapours led into a vapour separator comprising a casing having compartments for receiving respectively the heavier and lighter fractions. Each compartment has a separate outlet pipe, connected with a condenser, and means are provided by producing varying degrees of vacuum in the several outlet pipes, to cause separation of the vapours.—L. A. C.

*Oils; Method of treating — [with liquid acids].* J. Bogaerts, Sutton, Surrey. Eng. Pat. 116,765, June 25, 1917. (Appl. No. 9099 of 1917.)

CRUDE and other oils are treated with liquid acids in a cylindrical metallic casing which is caused to rotate about a horizontal axis at right angles to the axis of the cylinder, the contents thereby being projected from one end to the other of the casing.

—L. A. C.

*Bituminous binder and process for making the same.* G. A. Henderson, St. Albans, W. Va. U.S. Pat. 1,264,932, May 7, 1918. Date of appl., Feb. 15, 1916.

THE bituminous residue of petroleum having an asphaltic base is mixed with sulphur and heated so that the sulphur is absorbed and the greater part of the oily constituent is driven off. The product contains 5–10% of sulphur.—W. F. F.

*Coke ovens; Doors for —.* R. Peel and T. F. Shevels, New Braucepeth Colliery, Durham. Eng. Pat. 116,393, July 18, 1917. (Appl. No. 10,368 of 1917.)

*Coke oven doors.* W. Cunningham, Moresby. Eng. Pat. 116,478, Feb. 23, 1918. (Appl. No. 3258 of 1918.)

*Drains for hydraulic mains used in coal gas manufacture.* R. Dempster & Sons, Ltd., and R. M. Brooke, Elland. Eng. Pat. 116,068, Apr. 16, 1918. (Appl. No. 6422 of 1918.)

*Gas purifiers; Purifier grids and means for supporting same in* —. J. A. Spencer, Southall. Eng. Pat. 116,443, October 22, 1917. (Appl. No. 15,297 of 1917.)



*Fuel.* E. R. Sutcliffe, Leigh, Assignor to Pure Coal Briquettes, Ltd., Cardiff. U.S. Pat. 1,261,645, Apr. 2, 1918. Date of appl., Mar. 27, 1915.

SEE Eng. Pat. 5018 of 1915; this J., 1916, 826.

*Gas washer-scrubber.* J. A. P. Crisfield, Assignor to the United Gas Improvement Co., Philadelphia, Pa. U.S. Pat. 1,261,106, Apr. 2, 1918. Date of appl., Sept. 29, 1916.

SEE Eng. Pat. 113,937 of 1917; this J., 1918, 233 A.

*Cracking heavy oils; Process and apparatus for* —. U. S. Jenkins, Chicago, U.S.A. Eng. Pat. 116,119, May 17, 1917. (Appl. No. 7079 of 1917.)

SEE U.S. Pat. 1,226,526 of 1917; this J., 1917, 704.

*Gas-dryer.* U.S. Pat. 1,264,263. See I.

*Process for increasing the decolorising power of bleaching earth [fullers' earth].* Ger. Pat. 305,896. See I.

*Dust catcher for cleaning gases.* Ger. Pat. 302,973. See I.

*Process of separating gases or vapours from gaseous mixtures.* Ger. Pat. 304,603. See I.

*Manufacture of carbon or lampblack [from natural gas].* U.S. Pat. 1,264,796. See XIII.

*Apparatus for analysing gas.* Eng. Pats. 103,812 and 104,160. See XXIII.

*Gas calorimeters.* Eng. Pat. 115,915. See XXIII.

## II.B.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Shale [and coal]; Process for obtaining a distillate from — free from sulphur.* G. E. Heyl, London. Eng. Pat. 115,897, Oct. 11, 1917. (Appl. No. 5081 of 1917.)

Two or more rotary drums are connected in series by a hollow shaft, each drum being surrounded by a hood into which hot gases are passed. The first drum is charged with shale or coal to which has been added 10 to 20% of coal-tar or other mineral oil in order that the distillation may be carried out at a lower temperature. A certain amount of scrap metal may also be added. The remaining drums contain scrap metal such as borings, turnings, and the like. The vapours generated in the first drum pass through a dust separator into the hollow shaft, and out through holes into the next drum in which they come in contact with the scrap metal which is heated to a temperature sufficiently high to promote the formation of metallic sulphides. Attrition of the metallic particles causes removal of the sulphide and exposure of fresh surfaces of metal. The vapours are removed from the apparatus by means of a vacuum pump, purified, and condensed.—L. A. C.

*Pyroligneous acid; Method of distilling* —. H. O. V. Bergström, Stockholm. U.S. Pat. 1,261,344, Apr. 2, 1918. Date of appl., June 19, 1917.

Wood spirit is separated from pyroligneous acid in a column apparatus and the acid passed through a column into a boiler, in which it is distilled under pressure. The vapours are led back through the column under pressure in an opposite direction to that of the acid, and then into an alkaline absorbent (lime). Unabsorbed vapours are led through the heating coil of a second boiler for pyroligneous acid, thus effecting a transfer of heat from the vapours to the acid.—L. A. C.

*[Electric] lamp; Metallic-filament — and process of making it.* H. J. Jaeger, Weehawken, N.J. U.S. Pat. 1,265,204, May 7, 1918. Date of appl., July 27, 1917.

A "CONCENTRATED" metallic filament is coated with a chloride of a metal of the chromium group (e.g. uranium), and the lamp is filled with an inert gas, exhausted, and sealed. The filament is then heated so that the chloride is decomposed and uranium deposited on the filament.—W. F. F.

*Construction of retorts, coke ovens, and the like.* Eng. Pat. 116,870. See IIA.

## III.—TAR AND TAR PRODUCTS.

*Chemical composition of artificial asphalts.* Marcussou. See IIA.

### PATENTS.

*Naphthalene salts and similar substances; Apparatus for the extraction of — from the distillates of coal tar distillation.* Brotherton & Co., Ltd., J. Porteous, and E. D. Whitelock, Leeds. Eng. Pat. 115,906, May 24, 1917. (Appl. No. 7485 of 1917.)

EACH of a series of horizontal cylindrical vessels is provided with a steel inner tube in which is a rotating shaft fitted with a worm conveyor. The inner tubes are connected alternately at opposite ends of the apparatus by bends fitted with inspection doors; the outer vessels are similarly connected by vertical pipes. The tar oil is pumped into the top inner tube, passes down the series, and is cooled by ammonia or other refrigerant flowing through the annular spaces, preferably from the lower end. Naphthalene and other solids which separate are prevented from adhering to the walls of the tubes by the rotating worms, and are carried forward by the oil, being subsequently separated in presses. In the event of any cylinder being choked, it may be cut out by removing a connecting bend, the remaining sections being temporarily connected.—L. A. C.

*Phthalic anhydride; Making* —. T. D. Greenley, Glen Ridge, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,261,022, Apr. 2, 1918. Date of appl., Mar. 13, 1917.

NAPHTHALENE is heated with an insufficient quantity of fuming sulphuric acid to 250° C. in the presence of a catalyst, e.g., mercury sulphate. Two additional quantities of fuming sulphuric acid are added, the temperature being again raised to 250° C. after each addition, and then finally to 300° C. to expel the phthalic anhydride from the reaction mass.—L. A. C.

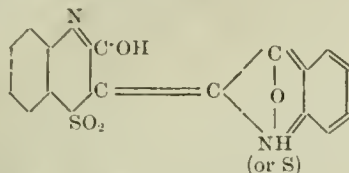
*Process and plant for recovering tar from producer gases, such as brown coal producer gases, in two portions, particularly suitable for dehydration.* Ger. Pat. 304,459. See 11A.

*Manufacture of resins.* U.S. Pat. 1,263,843. See XIII.

#### IV.—COLOURING MATTERS AND DYES.

*Dyes derived from sulphazone. A contribution to Claasz's new theory of the indigo chromophore.* W. Herzog. Ber., 1918, 51, 516—521.

SULPHAZONE condenses with  $\alpha$ -isatinanilide and thionaphthenequinone-anilide to form vat dyes of the indigoid type, without, however, any great affinity for fibres. These do not contain the usually-accepted indigo chromophore  $\text{—CO—C=C—CO—}$ , but their tinctorial properties can be ascribed to the presence in them of Claasz's indigo chromophores (this J., 1916, 1150). The dyes are represented therefore by the formulæ,



Condensation with  $\beta$ -isatinanilide yields a compound which is devoid of tinctorial properties, being related to the above as indirubin is to indigotin. (See further J. Chem. Soc., 1918, 1, 310.) —J. C. W.

*Anthocyanins and anthocyanidins; Production of —.* III. A. E. Everest. Proc. Roy. Soc., 1918, B 90, 251—265. (See also this J., 1914, 1199.)

From the petals of a purple-black viola the author has isolated an anthocyan pigment, which he has shown to be a delphinidin glucoside, identical with the violanin obtained by Willstätter and Weil from the blue-black pansy (see this J., 1916, 1213). The yellow sap-pigment from the same viola petals contains a myricetin glucoside and another yellow sap-pigment which does not give a green coloration with dilute alkalis. The so-called alum reaction described by Willstätter and his collaborators is shown to be due to the presence of iron as an impurity in the alum, no colour being obtained with the pure reagent.—W. G.

*Sorghum; Utilisation of the red dye obtained from the leaf sheaths and stalks of certain species of —.* G. C. Dudgeon. Agric. J. of Egypt, 1917, 7, 150—153. Bull. Agric. Intell., 1918, 9, 506.

THE red pigment developed at maturity in parts of the leaves and stems of certain varieties of sorghum (e.g., *S. technicum*) is used in Africa for dyeing goat leather and staining grasses used in basket making. The red pigmentation has been observed in varieties of broom sorghum, and even in *S. vulgare* in the form of red spots. The origin of the colouring matter is still unknown; it contains two pigments: one brick-red to orange-red in shade which is extracted by boiling water, the other crimson or purplish-red, extracted by boiling the residue with sodium bicarbonate. An infusion of the pigment in water containing a little sodium carbonate dyes tanned leather to shades ranging from red to brown, and unprepared wool brown. Wool previously boiled with alum is dyed by an infusion in

pure water to shades of rose and orange-pink, but the colour fades on exposure. In the case of unprepared wool, after-treatment with sodium "hyposulphide" fixes the dye. The addition of sodium "hyposulphide" to the water in which the sorghum is boiling gives a browner shade which appears to be fast to light. With zinc nitrate and tartaric acid as fixing agents, very bright dark orange to rose-pink shades are produced on silk and are apparently fast. The collection of the pigment does not interfere with the ordinary utilisation of the sorghum.—J. F. B.

*Adsorption compounds [of dyes].* Haller. See VI.

#### PATENTS.

*Sulphur dyestuffs; Process for manufacture of yellow —.* Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 302,792, Aug. 22, 1916. Addition to Ger. Pats. 292,148, 293,187, and 293,558 (this J., 1916, 832, 922, 1056).

INSTEAD of the simple diamines of the diphenyl series methylated in the nucleus, or their previously specified derivatives, the intermediate products of a primuline nature obtained by melting the specified diamino-compounds with *o*-, *m*-, or *p*-toluidine, in equimolecular proportions, and sulphur, or else mixtures of the methylated diamines or their derivatives and toluidine are melted with benzidine and sulphur at high temperatures.—J. F. B.

*Colouring matter soluble in ammonia and water; Process for the manufacture of a —.* O. Bauer. Heilbronn. Ger. Pat. 303,599, Apr. 25, 1917.

A COLOURING matter particularly suitable for wood staining is prepared by dissolving tanned leather waste in a boiling aqueous solution of a caustic agent. This colouring matter possesses the advantage of not roughening the pores; a large variety of shades is obtainable by the addition of more or less ammonia or water.—J. F. B.

*Gallocyanine dyestuffs with amines; Manufacture of condensation products of —.* O. Imray, London. From Soc. Chem. Industry in Basle, Switzerland. Eng. Pat. 116,754, June 19, 1917. (Appl. No. 8802 of 1917.)

IN the condensation of two mols. of a gallocyanine dyestuff with an aromatic amine, one mol. of the gallocyanine is converted into arylaminogallocyanine whilst the other is reduced to its leuco-compound, which escapes condensation. This reduction is prevented by conducting the reaction in presence of hydrogen peroxide or a derivative thereof, such as a persulphate or perborate, and completing the condensation by heating for several hours on the water-bath; a suitable diluent, such as alcohol, may be used. Condensation products may be made with aromatic amines or with amines containing, besides the free amino group, an acylamino group or an imino-azole residue. The condensation products may be converted into leuco-compounds by dissolving in formic acid and reducing with zinc dust, or by other known methods, and then applied in the same manner as chrome-printing dyestuffs. —J. F. B.

*Ligninsulphonic acid or its salts; Method of preparing azo colouring matters from —.* E. Oman, Stockholm. Eng. Pat. 103,479, Jan. 4, 1917. (Appl. No. 183 of 1917.) Under Int. Conv., Jan. 14, 1916.

LIGNINSULPHONIC acid or one of its salts (see this J., 1918, 121 A) is coupled with a diazotised aromatic



amine, and the azo dyestuff produced is precipitated either directly or after the addition of salt. *Example*: 6 grms. of aniline diazotised in the ordinary way is added while stirring to a solution of 5 grms. of sodium ligninsulphonate of 60% purity in 50 grms. of water; a brick-red colouring matter is precipitated. Brown and red colouring matters are obtained with benzidine and  $\beta$ -naphthylamine-disulphonic acid respectively. If preferred, the crude waste sulphite lye may be used instead of the sodium ligninsulphonate, but the colouring matters are then contaminated with impurities.—J. F. B.

[*Anthracene*] *vat dyestuffs and process for their formation*. M. Iijinsky, Assignor to R. Wedekind und Co., Uerdingen, Germany. U.S. Pat. 1,261,354, Apr. 2, 1918. Date of appl., Sept. 15, 1913.

SEE Eng. Pat. 19,435 of 1913; this J., 1914, 415.

*Chromium compounds of azo dyes*. R. Bohn, Mannheim, and P. Nawiasky, Assignors to Badische Anilin u. Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,264,604, Apr. 30, 1918. Date of appl., July 10, 1913.

SEE Eng. Pat. 26,460 of 1912; this J., 1913, 905.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Cotton; The testing of — by steaming*. M. Freiburger. *Färber-Zeit.*, 1917, 28, 221—224, 235—236, 249—252. *Z. angew. Chem.*, 1918, 31, Ref., 146.

CUTTINGS of cotton cloth were treated with solutions of various alkaline salts, dried, and steamed, then compared with a scale of standard whites prepared by staining a good white cloth with known quantities of diluted scouring liquor. Fats play no appreciable part in the yellowing of the goods during steaming. Treatment with sodium ricinoleate causes a slight lowering of the colour. As regards the behaviour of the "gums," both dry heating and steaming cause a yellowing of cotton at all stages of the scouring and bleaching process, the discoloration due to steaming increasing with the time. The presence of alkali intensifies the yellowing. Fully scoured cotton is discoloured only slightly less than the raw goods; in this respect the non-cellulose impurities differ but little from the cellulose. Chemicked goods show the strongest discoloration, those bleached cold show it more strongly than those bleached warm and acid; goods bleached warm with an excess of soda in sodium hypochlorite are relatively less subject to yellowing. Mercerised goods keep their colour well in spite of their greater attraction for water. Oxycellulose becomes quite brown on steaming, about 100 times darker than cellulose; hydrocellulose is ultimately discoloured less than cellulose. By steaming cotton treated with sodium ricinoleate comparatively with the untreated sample and observing its discoloration, a relative measure of the proportion of oxycellulose present is obtained. The main cause of the yellowing of cotton is the presence of  $\beta$ -oxycellulose.—J. F. B.

*Paper yarns and textiles; Effect of cold on —*. M. Lummerzheim. *Monatsschr. Textil-Ind.*, 1917, 32, 144. *Z. angew. Chem.*, 1918, 31, Ref., 147.

PAPER yarn comes on the market with a very high percentage of moisture, often with 40–80 parts of moisture per 100 of dry weight. In cold weather the moisture freezes and destroys the cohesion of

the individual fibres so that the yarn or fabric loses its strength and elasticity. Total destruction may take place if the yarn is suddenly subjected to intense cold. It is better, therefore, to retain the heat which is liberated when a portion of the water in the yarn freezes, so that it may moderate the rate of cooling. Yarns packed in closed boxes and transported in covered wagons are less liable to injury by frost than when they are packed in crates or sacks and are exposed to draughts. When paper yarn shows the presence of ice, it should not be worked up in the frozen state but should first be slowly thawed. In frosty weather the weaving room should not be allowed to get cold; warps which have become stiff cannot be stretched tightly and uniformly without many breaking.—J. F. B.

*Soda wood pulp by Ungerer's process; Manufacture of — on the large scale*. C. Ziegelmeyer. *Papier-Zeit.*, 1918, 42, 1855—1856. *Z. angew. Chem.*, 1918, 31, Ref., 174—175.

IN Ungerer's process the digestion of wood by the soda or sulphate process is carried out fractionally on the counter-current principle. The digestion liquor passes from boiler to boiler in a battery in such a way that the nearly spent lye comes into contact with the freshly charged wood and the fresh lye is fed on to a partly digested charge, exerting its maximum solvent action on the difficultly soluble incrusting and colouring matters. The process gives an exceptionally light coloured pulp. The washing waters are not mixed with the black liquors but are used for preparing fresh liquor, so that the spent lye is not diluted before evaporation and there are no effluents. The plate evaporator designed by the author gives a 24–30 fold evaporation. The pulp mill at Stuppach is producing 5–5½ tons of pulp daily by this process, and considerable extensions are contemplated. The Ungerer process is capable of producing excellent pulp from coarse-grained wood; the pulp can be fully bleached with 1–2% of bleaching powder, and owing to its low ash it is very suitable as a substitute for cotton for munition purposes.—J. F. B.

*Soda and sulphite pulps; Differentiation of —*. P. Klemm. *Woch. Papierfabr.*, 1918, 48, 2159—2161. *Z. angew. Chem.*, 1918, 31, Ref., 175.

IN the sulphite process certain residues of the wood substance survive and are found in the paper, whereas by the alkaline process the purification is far more complete. These residues of cell-contents, which are particularly to be found in the cells of the medullary rays, may be made visible by staining and serve for the differentiation of soda and sulphite pulps. The residues are found even in bleached sulphite pulps and exist as aggregates or chains of small spherical elements. Their staining capacity depends on the presence of resin; with an aqueous-alcoholic solution of Sudan III. with a little glycerin, they are stained red; with zinc chloride-iodine, sulphur-yellow. In preparing the pulp for microscopic examination care must be taken to avoid dissolving the resin by the caustic soda. Medullary ray cells are so numerous that the presence of even 5–10% of sulphite pulp in a mixture can thus be detected. For quantitative estimation of mixtures of sulphite and soda pulps, a solution of rosaniline sulphate with a little alcohol and sulphuric acid is of service; the contents of the pitted pores are strongly stained in the case of sulphite pulps but not with soda pulps. The inner side of the fibre wall of sulphite pulp is more strongly dyed than the outer. In zinc chloride-iodine, the sulphite fibres show a characteristic venation.—J. F. B.

*Sulphite waste liquors; Concentration of* —. E. Oman. *Papierfab.*, 1918, 15, 605–607. *Z. angew. Chem.*, 1918, 31, Ref., 176.

ONLY about 16% of the solid constituents of the sulphite waste liquors can be utilised by fermentation. The waste liquors, after distillation of the spirit, may be evaporated and used as a binder for sand moulds in iron foundries. In place of evaporation, which is expensive, the author suggests concentration by freezing, which may take place either before or after the fermentation. Sulphite liquor freezes at 0.5° C., and the sugar is not injuriously affected, as it is during evaporation. By use of a centrifugal the ice crystals are easily removed, and with repeated freezing it is possible to obtain a liquor with 35% of dry substance. The cost of evaporating 1 ton of water is ten times as great as that of freezing out 1 ton of ice when water power is used for the refrigerators.—J. F. B.

*Cork slabs from cork waste.* H. Ost. *Z. angew. Chem.*, 1918, 31, 105–108.

SLABS of considerable strength may be made by compressing ground cork waste and heating it at 160°–180° C. while under compression. No binding agent is required, and it has been suggested that the natural resin of the cork serves as a binder. Various samples of ground cork were extracted several times for 24 hours with alcohol and with chloroform in the cold. The maximum amount of the united extracts, in the case of cork dust, was 9.35% of the air-dry weight. Higher results were obtained when water was also used, e.g., up to 10.3%, since the tannins are also extracted, but water has an injurious action on the cork substance. The extract was resinous and began to melt at 150° C. Equivalent weights of comparative samples of natural and extracted granulated cork were compressed to form plates of various thickness, then heated in the moulds for about 3 hours at 180° C. or for 15–20 hours at 160° C. At the higher temperature a slight browning of the cork substance was observed, but at 160° C., in spite of the longer time, there was no perceptible decomposition. Tensile tests were made in a cement-testing machine; the strength depended on the degree of compression and the heating. The more favourable results with plates compressed under 7 atmos. varied from 6 to 8 kilos. per sq. cm.: some very heavily compressed plates showed 14 kilos. per sq. cm. The plates from the extracted cork were only slightly inferior to those from the natural cork. The resistance to water and steaming was almost entirely a function of the temperature of heating while under compression; plates heated at 50°–100° C. readily disintegrated, while those heated at 180° C. were the most resistant. Heating before compression was useless. The compressed plates adhered firmly to the walls of the mould, but pieces of tissue paper placed between prevented this adhesion and showed no oily or resinous stain. The author concludes that resinous extractive matters play no part in binding the compressed plates, but that their solidity depends on an interlocking or felting of the cork cells. Without heating, the elasticity of the cork is restored by steaming and the plates fall to pieces, but at 160°–180° C. incipient decomposition destroys the elasticity and the felting of the cells is permanent and rigid.

—J. F. B.

*Recovery of grease from waste materials by the solvent extraction process.* Garner and Carmichael. See XII.

*Carbohydrates other than sugar [e.g., waste liquors from cellulose manufacture] which are suitable as yeast foods.* Bokorny. See XVIII.

## PATENTS.

*Woollen goods; Process for protecting* — from moths. E. Naefe, Berlin-Britz. Ger. Pat. 304,503, Feb. 27, 1917.

The fabric is impregnated with soluble salts of alginic acid and then treated in a bath of antimony salts for about 30 minutes.—J. F. B.

*Wool; Method of cleaning, carbonising, and sterilising raw* —. H. Hey, Dewsbury. Eng. Pat. 116,763, June 23, 1917. (Appl. No. 9034 of 1917.)

UNSCOURED raw wool is steeped for 1½ hours in a bath containing 4.5% of sulphuric acid and 1% of 40% formaldehyde; the wool is removed and hydro-extracted and then dried at about 80° C. until the vegetable matter becomes friable. The dried wool is treated twice with a volatile solvent in a centrifugal degreasing machine, being centrifuged after each treatment. The adhering solvent is evaporated, the wool is passed between fluted rollers, and the pulverised impurities are removed by opening and beating the wool in a current of air.

—J. F. B.

*Boiler for treating substances, particularly cellulose, suspended in liquids, with steam.* F. Bück. Vienna. Ger. Pat. 302,893, Nov. 5, 1915. Under Int. Conv., Oct. 25, 1915.

The boiler is provided with a tubular vertical extension open at the top to increase the pressure of liquid in the body of the vessel. A mechanical circulating device is situated in a pipe open at both ends through which liquid is pumped from near the bottom of the boiler and delivered at a higher level. Pipes are arranged to deliver live steam and fresh water or liquor near the bottom of the vessel, and a conical gas or vapour collector formed of a series of overlapping plates is suspended in the upper part of the boiling vessel and communicates with a relief pipe which extends up the tubular extension and terminates above the level of the liquid therein. The pipe through which the fresh water or liquor is introduced is coiled around the upper surface of the conical vapour collector. The apparatus is suitable for the continuous or intermittent washing of cellulose or nitrocellulose while boiling.—J. F. B.

*Paper; Process for utilising waste* — for re-making into white paper. J. Galloway and A. Stratton, Portobello. Eng. Pat. 115,940, June 23, 1917. (Appl. No. 9045 of 1917.)

WASTE paper is boiled with 60–80% caustic soda under a steam pressure of 35–40 lb. per sq. in., washed in the boiler with warm water, then broken and washed in breaking engines; the pulp is passed over sand tables and through strainers, the excess of water is removed by drum washers, and the concentrated pulp bleached and treated for the manufacture of paper.—J. F. B.

*Plants for papermaking; Treatment of certain* —. C. J. Stewart, London, H. D. Hall, Croydon, and H. Beadle, Sidcup, Exors. of C. Beadle. Eng. Pat. 116,005, Sept. 22, 1917. (Appl. No. 13,679 of 1917.)

CERTAIN fibrous materials such as many freshly cut green crop plants, not highly lignified, particularly *Hedychium coronarium* and cotton seed hull fibre, are reduced to pulp for papermaking by treatment in the beating engine with a hot alkaline liquor below the boiling point. In the case of fresh *Hedychium*, 6% of its weight of caustic soda may be used at 80° C. and the process may last for 2–4 hours; in many cases the alkali is neutralised



by the organic matters extracted from the fibre but if an excess is employed it may be removed by neutralisation or washing. If desired, the juices of the plants may be removed before the alkaline treatment. Material may be crushed and steeped in an alkaline liquid, stored and transported, and subsequently treated with hot alkali in the beating engine.—J. F. B.

*Paper-filler; Liquid — and process of making the same.* W. N. Kohlins, Newark, N.J. U.S. Pat. 1,261,135, Apr. 2, 1918. Date of appl., July 18, 1917.

"SATIN WHITE" is liquefied by mixing with it less than 3% of gum arabic, dextrin, and "saccharin," and the fluid mixture is used as a paper-filler.  
—J. F. B.

*Paper for textile purposes; Manufacture of —.* G. und H. Beneke Ges., Lübau. Ger. Pat. 304,772, Sept. 29, 1916.

The paper pulp or the finished paper is treated with a very dilute solution of a colour-lake ("Farb-lack") in benzine, which increases its resistance to water and acids.—J. F. B.

*Paper textiles; Process for cleaning old —.* T. Goldschmidt A.-G., Essen. Ger. Pat. 305,427, Sept. 11, 1917.

The textiles are freed from large impurities by mechanical treatment and are then subjected consecutively to treatments with water and bleaching powder or chlorine solution, and finally to a mechanical finishing operation, e.g. in drying machines, presses, or calenders. The treatment in the various baths should not be so prolonged as to loosen the texture of the fabric to such an extent that it cannot be restored in the finishing.—J. F. B.

*Waste paper; Process for decolorising —.* W. Abele, Berlin-Wilmersdorf. Ger. Pat. 305,343, May 8, 1917.

The liquid used for removing ink, etc., from the waste paper is circulated continuously through a vessel containing the paper and through a heater.

*Cellulosic materials and products thereof; Process of treating the residual liquor obtained in the hydrolysis of —.* T. B. Wagner, Brooklyn, N.Y. U.S. Pat. 1,261,323, Apr. 2, 1918. Date of appl., Sept. 17, 1917.

The fermented sugar-containing liquor resulting from the hydrolysis of cellulosic material is concentrated, after the distillation of the alcohol, the concentrated product containing more than 30% of reducing sugars (calculated as dextrose) and approximately 25–35% of water.—J. F. B.

*Paper pulp and other fibrous materials; Apparatus for beating —.* Apparatus for treatment of paper pulp and other fibrous materials. C. M. Cowan and J. Brown, Musselburgh. Eng. Pats. 115,972 and 116,040, July 31, 1917. (Appl. Nos. 10,999 and 18,952 of 1917.)

*Straining pulp and the like.* J. Paramor, Rickmansworth, Assignor to the Watford Engineering Works, Ltd., Watford, and J. M. Henry, Newbury. U.S. Pat. 1,261,054, Apr. 2, 1918. Date of appl., Dec. 12, 1916.

SEE Eng. Pat. 4186 of 1915; this J., 1916, 418.

*Paper-making machine.* W. A. Aitken, Gravesend. U.S. Pat. 1,264,254, Apr. 30, 1918. Date of appl., Dec. 22, 1916.

SEE Eng. Pat. 106,228 of 1916; this J., 1917, 708.

*Fireproofing solution.* U.S. Pat. 1,261,736. See I.

*Preparation of azo colouring matters from lignin-sulphonic acid or its salts.* Eng. Pat. 103,479. See IV.

*Process for obtaining an extract of proteolytic enzymes.* Eng. Pat. 106,504. See XVIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Silk; Bleaching of raw — by permanganate.* E. Ristenpart. Färber-Zeit., 1918, 29, 37–38.

THE *aqua-regia* bleach is entirely obsolete and uncertain in its action; the nitrite bleach is safer and less costly but it always leaves a yellow with a greenish or brownish tone, according to the duration of treatment. The permanganate bleach is cheaper than the peroxide process. Raw silk is wetted out hot with "Monopol" soap, rinsed, and treated for half an hour at the ordinary temperature with 90 times its weight of a 0.1% solution of potassium permanganate. In presence of 0.1% of sodium hydroxide the action of the bleaching bath is very deleterious; the silk loses about 6% in weight and the tensile strength and stretch are seriously lowered. The neutral permanganate bath also removes about 6% in weight, but the strength is unaffected and the stretch is lowered to a less extent than with the alkaline solution. In neutral permanganate there is a liberation of alkali as the result of the reaction, and the best results are obtained in presence of 0.1% of sulphuric acid, with a loss of weight of 0.5%. The silks are cleared with a solution of 3 grms. of bisulphite and 1.5 c.c. of sulphuric acid per litre. The colour obtained is not, however, a real white but is considerably better than with the older processes. When the permanganate bleach in presence of acid was repeated three times in succession, instead of an improvement in colour, the reverse was obtained, the colour falling off to a brownish shade, with increasing loss of weight and tensile properties. Treatment with 0.1% "blankit" solution at 40°–60°C. after the removal of the manganese with bisulphite removed the red tinge from the bleached "white" but left the yellow more prominent.  
—J. F. B.

*Dyeing; Theories of —.* The theory of the acid dyebath for wool. M. Fort. J. Soc. Dyers and Col., 1918, 34, 124–126.

REFERRING to the experiments by Harrison (this J., 1918, 238 A), who showed that sulphuric acid could be entirely removed by washing wool with distilled water, the author states that this differs from his own experience. There is a large amount of evidence relating to the equilibrium in dyeing and stripping and the chemical properties of fibre and dyestuff to be dealt with before the chemical theory of dyeing, in the main aspect of the process, can be dismissed as inadequate. The value of a theory must be measured by its practical results and the theory of the acid-wool compound which the author has supported is more fruitful in technical application than theories based on remote and little understood electrical phenomena. Protein substances of the most diverse origin and physical structure all

behave very similarly in the acid dye bath and their chemical relationships are far more obvious than any other properties on which a theory of dyeing could be based. Moreover, analogies which have been drawn in support of the author's views, such as the mechanism of the dyeing of cotton impregnated with benzidine (this J., 1916, 304) and the reaction between magnesium oxide and Crystal Scarlet, are too strong to admit of any other than a chemical explanation. Lastly Harrison's experiments do not explain the decomposition of neutral salts by wool in order to attain combination with sulphuric acid.—J. F. B.

*Adsorption compounds [of dyes].* R. Haller. *Kolloid-Zeits.*, 1918, 22, 113—133.

THE so-called adsorption compounds formed by dyes with other substances behave as mechanical mixtures rather than as chemical compounds. The mode of segregation of the adsorbed substance varies with the structure and physical properties of the adsorbent. The adsorption of a given dye by a crystalline substance, such as barium sulphate, is apparently very different from the adsorption which occurs with a colloidal substance, such as cotton wool, but the differences are probably entirely due to the difference in the physical characteristics of the two adsorbents. The adsorption effect is also appreciably different, so far as microscopic observations go, if the adsorbent is employed on the one hand in sol form and on the other in the form of a coarse powder. When the adsorption compounds are treated with chemically indifferent solvents, the effects observed are those which would be expected on the assumption that the "compounds" are mechanical mixtures. The absorption spectra of the solutions which are obtained by dissolving the adsorption compounds in suitable solvents are also readily accounted for on the mixture hypothesis. The adsorption compounds which are formed by easily fusible adsorbents, such as stearic and palmitic acids, melt at temperatures which are near to the melting points of the pure adsorbents. The general physical properties of the adsorption compounds are therefore such as may be readily interpreted on the assumption that these are mechanical mixtures of the adsorbent and adsorbed substance. The action of mordants, on the other hand, is probably due to the formation of chemical compounds between the mordant and the dye, and these compounds subsequently form adsorption compounds or intimate mechanical mixtures with the fibre in the process of dyeing.—H. M. D.

*Black dyeing on iron mordant.* O. Diehl. *Färber-Zeit.*, 1917, 28, 231. *Z. angew. Chem.*, 1918, 31, Ref., 136.

A black, which possesses the advantage of satisfying all requirements as regards fastness and not injuring the wearing qualities of the most delicate fabrics, is produced by dyeing a mixture of alizarin and dinitroresorcinol on an iron mordant.—J. F. B.

*Naphthylamine Claret; Dyeing to the shade of —.* O. Diehl. *Färber-Zeit.*, 1917, 28, 212—213. *Z. angew. Chem.*, 1918, 31, Ref., 136.

OBJECTIONS to the use of Naphthylamine Claret, on account of the easily decomposable printing colour and injury to the white of goods prepared with  $\beta$ -naphthol, led to experiments to obtain the same shade by dyeing with alizarin and Alizarin Cyanin on an alumina mordant. The brightness and fastness to light and washing were perfectly satisfactory. The procedure adopted is as follows:—The material is either printed with thickened aluminium acetate or padded with unthickened aluminium

acetate on the hot flue and the white discharged with citric acid. After the usual passage through the preliminary steamer the goods are degummed and dyed in a mixture of Alizarin, bluish, and about " $\frac{1}{2}$  N" Alizarin Cyanin R, oiled, steamed, and soaped.—J. F. B.

*Textiles; Action of laundry agents on —.* A. Grün and J. Jungmann. *Seifenfab.*, 1917, 37, 507—510, 529—531, 553—555, 579—581, 603—606. *Z. angew. Chem.*, 1918, 31, Ref., 146.

AFTER 30 washings with hard soap, soda, sodium silicate and perborate, respectively, linen and cotton textiles showed higher strength values with hard water than with soft water. With soft water sodium silicate showed a distinct injurious action on cotton, and perborate was still more harmful. Goods washed with soap improve in lustre and show a brilliant white colour by reflected light; goods washed in soda show a strong yellowing in transmitted light with a slight shade of pink; by reflected light they appear slightly grey. After washing with silicate the goods are white but quite without lustre, being dull and chalky. The handle of goods washed with soda or silicate is very poor. The loss of tensile strength suffered by textiles in washing is not a simple measure of the changes which have taken place in the fibres. In estimating the loss of quality the external appearance of the fabric must be taken into account and particularly the splitting of the fibres as observed microscopically. Perborate in presence of soap is decomposed with equal rapidity in distilled and in tap water. Soda slightly retards the decomposition; silicate makes a solution of perborate in hard water practically stable and has some influence in the same direction in cold distilled water. The alkalinity of perborate solutions is of little importance compared with the rapidity with which they part with their oxygen.—J. F. B.

*Utilisation of the red dye obtained from the leaf-sheaths and stalks of certain varieties of sorghum.* Dudgeon. See IV.

#### PATENTS.

*Bleaching and washing baths; Process for recovery of oxalates and pyrophosphates from exhausted —.* O. Reich, Lindenbergl. Ger. Pat. 304,601, Mar. 17, 1917. Addition to Ger. Pat. 300,523.

THE bleaching baths are diluted until pale in colour, treated with ammonium salts, and the pyrophosphoric acid is precipitated by manganese or magnesium salts; the oxalic acid is precipitated from the filtrate by the addition of alkaline-earth compounds.—J. F. B.

*Bleaching apparatus.* F. Donahue, Utica, N.Y. U.S. Pat. 1,263,045, Apr. 16, 1918. Date of appl., Apr. 30, 1917.

A CLOSED kier is provided with a perforated false bottom and with a vertical circulating pipe passing through the false bottom to the upper part of the kier. Steam is supplied below the false bottom through a steam jet tube extending up into the circulating pipe. In addition to this, an external circulating pump is provided which can be arranged to draw liquid either directly from the kier below the false bottom or from either of two cisterns which are adapted to receive liquid discharged from the bottom of the kier, such liquid being delivered by the pump into the upper part of the kier.—J. F. B.



*Dyeing machines, washing off machines, and the like.* J. Farrar, Halifax, and A. R. Whitehead, Leeds. Eng. Pat. 115,082. (Appl. Nos. 5785, Apr. 25, and 9502, July 3, 1917.)

THE dyeing vat is constructed with a false bottom composed of boards spaced apart and has a circulating chamber at one end separated from the vat by a division board. The dye or other liquor is circulated from the circulating chamber, passing up between the boards of the false bottom into the vat and back over the dividing board into the circulating chamber. When used for washing off, fresh water enters the dyeing vat at the end remote from the circulating chamber, through apertures in a transverse pipe arranged to assist the circulation of water through the vat, and the water overflows and escapes at the circulating chamber, or at or near the division board, through a trough from which the upper layer, with the floating impurities, is discharged. The lid of the vat, from which the hank frame is suspended, is provided with sprays from which water can be sprayed on to the heads of the hanks after removal of the liquor.—J. F. B.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,261,498 and (B) 1,261,499, Apr. 2, 1918. Dates of appl., (A) Feb. 15, (B) Jan. 31, 1917. (See also this J., 1918, 265 A.)

(A) THE receptacle contains a series of removable fibre chambers adjacent to each other, each chamber having a non-perforated side and a foraminous bottom fitted with a number of vertical spindles; a second chamber below the fibre chamber contains a series of vertical rods, and a third chamber, also containing rods, is situated above the fibre chamber. A small testing device similar to the main device but without the third upper chamber is connected with the large one below the foraminous bottoms and means are provided for forcing liquid through the two systems simultaneously. (B) The foraminous plates below and above the fibre chamber are composed of series of converging and diverging elements with openings between the elements; means are provided for adjusting and maintaining the upper plate at varying distances from the lower one. A liquid supply chamber is situated below the fibre chamber formed between the two plates, and a chamber below the liquid supply chamber contains a series of nested members converging downwards, so arranged as to allow the passage of a liquid between them. A smaller testing device is provided and also means for conducting air and circulating liquid through the two devices equally.

—J. F. B.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,261,500 and (B) 1,261,501, Apr. 2, 1918. Dates of appl., (A) Apr. 28, and (B) May 16, 1917. (See also this J., 1918, 297 A.)

(A) THE foraminous cylinder on which the fabric is rolled is provided with annular members capable of sliding inwards thereon and other annular members sliding over the first, the inner faces of all of them forming a surface capable of abutment upon the edges of the rolled-up fabric. A second foraminous cylinder is spaced inside the first and is revolvable therewith; a series of tubes connects the entire inner face of the second cylinder with a source of liquid supply and the liquid passes into the space between the two cylinders and thence through the first cylinder during the rotation; steam is also introduced between the two cylinders and the adjustable members to prevent the passage of the liquid through any portion of the first cylinder which is not covered by the fabric. (B) The receptacle contains a reel with non-perforated sides, an inner foraminous cylinder integral with the sides of the reel with longitudinal openings, an outer

foraminous cylinder having a series of members extended outwards and inwards with openings in the sides thereof, an adjustable foraminous belt to enclose the fabric and carrying members extended inwards, a series of tubes covering the inner face of the inner foraminous cylinder, means for continuously passing liquid through the system and for passing air through the tubes during the revolution of the reel.—J. F. B.

*Silk-dyeing.* S. Saxe, New York. U.S. Pat. 1,263,126, Apr. 16, 1918. Date of appl., Aug. 16, 1917.

SILK is dyed and weighted by treatment with a strong extract of wood of Osage orange, or in dyeing black, the silk is mordanted and dyed, then worked in a strong extract of Osage orange, and the black-dyeing completed.—J. F. B.

*Dyestuffs; Process for developing dyeings on vegetable fibres with diazotisable substantive* —. Act.-Ges. f. Anilinfabrikation. Ger. Pat. 303,409, Apr. 4, 1917.

THE dyestuffs are diazotised on the fibre in the usual way and developed with an N-alkyl derivative of chloro-*m*-phenylenediamine. The colours so obtained are generally bluer or of a more distinctly reddish blue shade than those developed with chloro-*m*-phenylenediamine.—J. F. B.

*Proteins; Purification of products of hydrolysis of* — [for use as detergents, etc.]. C. Bennert, Cöpenick. Ger. Pat. 304,003, Dec. 24, 1915.

CRUDE solutions containing protalbic and lysalbic acids or their salts, such as are obtained by the hydrolysis of proteins by caustic alkalis, are treated with calcium or magnesium hydroxide or a mixture of both. These protein derivatives are useful as detergents and are also employed in the textile industry, particularly in dyeing and printing. For this purpose they have to be purified from gelatinous and other by-products which give insoluble compounds with calcium and magnesium salts.—J. F. B.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

[Sulphuric] acid mixing; Graph for —. E. C. Craven. Chem. News, 1918, 117, 217–218.

A GRAPHIC method is described for determining the proportions of oleum and C.O.V. to be mixed in making highly concentrated sulphuric acid. Two parallel side scales are provided with a slide between them. One of the scales is marked with C.O.V. concentrations in per cent.  $\text{H}_2\text{SO}_4$ , and the other with oleum strengths in per cent.  $\text{SO}_3$ . Unit lengths on each side correspond to the same percentage difference of  $\text{SO}_3$ . The slide is marked with an inclined line divided into 100 parts, joining two corresponding points on the scales, e.g., 100%  $\text{H}_2\text{SO}_4$  and 81.63%  $\text{SO}_3$ . The slide is set with one end of the line against the desired strength of acid and the percentage strengths of the storage tanks of oleum and C.O.V. joined by a line. The intersection point on the divided line gives the reading for the relative proportions of oleum and C.O.V. to be mixed.—W. F. F.

*Oxides of nitrogen; Reduction of the* — to ammonia. Stability of nitric oxide. P. A. Guye and F. Schneider. Helvetica Chimica Acta, 1918, 1, 33–52.

IN Switzerland the conditions are such that oxides of nitrogen can be manufactured more conveniently than ammonia, so that a knowledge of the condi-

tions under which the former are reduced to the latter becomes necessary. The authors have carried out such an investigation, using a special apparatus in which known mixtures of hydrogen with the respective oxides of nitrogen were passed over heated reduced nickel as catalyst. In order to decompose nitric oxide under atmospheric pressure, it is necessary to keep the temperature at or above  $575^{\circ}\text{C}$ . for several hours, even in the presence of spongy platinum as catalyst. Under these conditions, and contrary to what takes place under pressure, there is no formation of nitrous oxide. The primary products of decomposition are nitrogen and oxygen; on cooling, the oxygen reacts with undecomposed nitric oxide to give nitrogen peroxide. Nitrous oxide decomposes into nitrogen and oxygen. In the reduction of nitric and nitrous oxides and nitrogen peroxide by hydrogen, with nickel as catalyst, two reactions take place simultaneously, the one giving ammonia and the other nitrogen. Both in the reduction of nitric oxide and of nitrogen peroxide, the formation of intermediate products (nitrous oxide and nitric oxide respectively) does not occur. Temperatures of  $250^{\circ}$ – $300^{\circ}\text{C}$ . appear to be the best for the formation of ammonia. Below these temperatures the reduction takes place slowly, whilst above these temperatures the nickel gradually loses its activity and the ammonia is appreciably decomposed into its elements. Nitric oxide gives the best yields, 70% of the gas on an average being transformed into ammonia; 25–39% of nitrogen peroxide and only 3–7% of nitrous oxide is reduced to ammonia. The reduction of the oxides of nitrogen to ammonia does not, therefore, appear to be an advantageous process from the manufacturing point of view, except, perhaps, in the case of nitric oxide, where a more systematic study of the conditions may give better results.—T. S. P.

*Nitrates: Gasometric determination of —.* C. A. Hill. *Analyst*, 1918, 43, 217–218.

The usual method of determining nitrates by shaking with sulphuric acid in a nitrometer filled with mercury has many disadvantages; if an external reaction flask is used, this must be filled previously with a gas which is inert towards nitric oxide. Carbon monoxide may be used for the purpose and is conveniently prepared by heating a mixture of sodium formate and concentrated sulphuric acid.—W. P. S.

*Iodides; Detection of — in the presence of cyanides.* L. J. Curtman and C. Kaufman. *J. Amer. Chem. Soc.*, 1918, 40, 914–917.

The detection of iodide in presence of relatively large quantities of cyanide has been examined by comparative tests with potassium nitrite, potassium permanganate, hydrogen peroxide, and chlorine as oxidising agents. The results show that the interference of cyanide is least when potassium permanganate is employed to liberate iodine. A method is described by means of which it is possible to detect 1 mgrm. of iodide in the presence of 500 mgrms. of cyanide. The cyanide is precipitated by the addition of cobalt nitrate, and after the addition of asbestos fibre the mixture is boiled for half a minute and filtered. The filtrate and washings are then tested for iodide by the addition of 1 c.c. of chloroform, 2 c.c. of 3*N*  $\text{H}_2\text{SO}_4$ , and 2 drops of 0.5% potassium permanganate.—H. M. D.

*Alkali phosphates: Reaction between the — and magnesium chloride.* D. Balareff. *Z. anorg. Chem.*, 1918, 102, 241–246.

The author shows that the precipitate formed by the addition of magnesium chloride to disodium hydrogen phosphate is liable to contain alkali in the form of  $\text{MgNaPO}_4$ , the amount of alkali depending

upon the conditions of precipitation. In saturated solutions of  $\text{MgCl}_2$  and  $\text{Na}_2\text{HPO}_4$  the amount is large, in dilute solutions small. When a dilute solution of magnesium chloride is added slowly to a 10% solution of  $\text{K}_2\text{HPO}_4$ , the crystalline precipitate formed consists of practically pure  $\text{MgKPO}_4 \cdot 6\text{H}_2\text{O}$ . By heating to  $100^{\circ}\text{C}$ . this salt is converted into the monohydrate. When the two solutions are mixed in the reverse order, the composition of the precipitate is uncertain and variable, there being present probably  $\text{MgKPO}_4 \cdot \text{aq.}$ ,  $\text{MgHPO}_4 \cdot \text{aq.}$ ,  $\text{Mg}(\text{OH})_2$ , and  $\text{Mg}_3(\text{PO}_4)_2$ . When dilute (0.2 mol.) solutions are used, the precipitate is almost pure  $\text{MgHPO}_4 \cdot \text{aq.}$  The rubidium salt  $\text{MgRbPO}_4 \cdot 6\text{H}_2\text{O}$  can be obtained practically pure under the same conditions as the potassium salt. (See also *J. Chem. Soc.*, Aug., 1918.)—E. H. R.

*Stannic fluoride; Behaviour of solutions of —.* N. H. Furman. *J. Amer. Chem. Soc.*, 1918, 40, 906–914.

Stannic fluoride hydrolyses completely in aqueous solution in accordance with the equation  $\text{SnF}_4 + 4\text{H}_2\text{O} = \text{Sn}(\text{OH})_4 + 4\text{HF}$ . From this solution the tin is precipitated by hydrogen sulphide; but this does not take place in presence of a sufficient quantity of hydrofluoric acid. From experiments made with solutions of stannic chloride, to which alkali fluorides and varying quantities of hydrofluoric acid were added, it appears that the absence of precipitation by hydrogen sulphide requires not only the presence of fluoride but also of free acid. The free acid prevents the hydrolysis of the stannic fluoride. The preparation of pure stannic fluoride by a method which involves the use of a minimum quantity of platinum ware is described. (See also *J. Chem. Soc.*, Aug., 1918.)—H. M. D.

*Gold [compounds] at high temperatures and pressures; Chemistry of —.* H. H. Morris. *J. Amer. Chem. Soc.*, 1918, 40, 917–927.

Certain gold compounds are reduced when heated in a steel bomb in contact with water at high temperatures. Gold hydroxide is reduced at  $320^{\circ}\text{C}$ ., and reduction occurs at a lower temperature in presence of sodium or magnesium chloride. Calcium chloride retards the decomposition. Gold chloride gives metallic gold at about  $370^{\circ}\text{C}$ ., but in presence of small quantities of sodium, magnesium, or calcium chloride a temperature of  $450^{\circ}$ – $460^{\circ}\text{C}$ . is required. Calcite and magnesite reduce gold chloride in aqueous solution at about  $310^{\circ}\text{C}$ ., but a temperature of about  $500^{\circ}\text{C}$ . is required in the presence of sodium, magnesium, or calcium chloride. The reduced gold dissolves in hot concentrated hydrochloric acid if this contains gold chloride. Aurous chloride is thereby formed and may be titrated with potassium permanganate.—H. M. D.

*Spent oxide; Analysis of —.* V. Hottenroth, Wentzel, E. Wolff, H. Drehschmidt, and Wirts. *Ver. deutsch. Gaswerke A.-G. Z. angew. Chem.*, 1918, 31, 127–128.

A series of criticisms on the method of determining sulphur in spent oxide (this *J.*, 1918, 240 A). Hottenroth points out that substances other than sulphur are extracted by carbon bisulphide. Washing the extracted sulphur with ether does not purify the sulphur completely. Wentzel recommends the extraction of the sulphur by carbon tetrachloride in a modified Soxhlet apparatus, Wolff converts the extracted sulphur into sulphate by fusion with sodium carbonate and potassium nitrate, and precipitates the sulphate with barium chloride, and Drehschmidt is also of opinion that the sulphur should be converted into sulphate and determined as such, since purification by ether is unsatisfactory.—W. P. S.



*Use of wood in chemical apparatus.* Schorger. See I.

*Effluents from ammonia-recovery plant of coke ovens.* Marsson and Weldert. See IIa.

*Some chemically reactive alloys.* Ashcroft. See X.

*Influence of iron, tungsten, and nickel upon the resistance of aluminium to acids.* Von Zeerleder. See X.

*Determination of nitric acid in bismuth subnitrate.* Luce. See XX.

*Estimation of chromium in presence of iron.* Schorlemmer. See XXIII.

*Determination of carbonates and bicarbonates.* Mestrezat. See XXIII.

*Detection of anions.* Feigl. See XXIII.

*Volumetric determination of chlorine, bromine, cyanogen, and mercuric ions.* Votocek. See XXIII.

*Acidimetry of coloured solutions: an application of the pocket spectroscope.* Tingle. See XXIII.

*Use of hydrofluoric acid in analysis.* Furman. See XXIII.

*New method of estimating mercury by means of zinc filings.* François. See XXIII.

#### PATENTS.

*Sulphuric acid; Process of concentrating* —. *Concentrating apparatus.* (A) I. Hechenbleikner, Charlotte, N.C., (B) I. Hechenbleikner, Assignor to Southern Electro-Chemical Co., New York. U.S. Pats. (A) 1,264,182 and (B) 1,264,509, Apr. 30, 1918. Dates of appl. (A) Feb. 10, 1917, (B) Nov. 6, 1914.

(A) A BATH of the acid is maintained at a temperature below its boiling point, and hot air is passed through the liquid to absorb and carry off volatile impurities. Weak acid is admitted to the bath, after being heated by the gases escaping from the bath. (B) The apparatus comprises means for leading the hot gas into the bath in a number of separate streams at a point below the surface, the current of weak acid being fed from a point above the container to replenish the bath, and being heated by the escaping gas.—B. N.

*Sulphuric acid; Denitration of* —. I. Hechenbleikner, Charlotte, N.C., Assignor to The Southern Electro-Chemical Co., New York. U.S. Pat. 1,264,512, Apr. 30, 1918. Date of appl., Feb. 10, 1917.

SULPHURIC acid, containing nitrogen oxides, is sprayed into a current of steam, and oxygen is introduced into the resulting gases to produce nitric acid fumes, the latter being subsequently condensed.—B. N.

*Gases [sulphur dioxide]; Method and apparatus for segregating and recovering* —. *Art of and apparatus for treatment of corrosive gases.* F. A. Eustis, Milton, Mass. U.S. Pats. (A) 1,265,892 and (B) 1,265,893, May 14, 1918. Date of appl., Dec. 19, 1917.

(A) In a process for recovering sulphur dioxide from furnace gases, the solution obtained by scrubbing the latter with water is subjected to the combined action of heat and vacuum, and the sulphur dioxide liberated is deprived of water vapour by means of refrigerating and desiccating agents. (B) To minimise corrosion of apparatus by acid gases containing condensed water (e.g. the moist sulphur dioxide obtained in the foregoing process), the gas is deprived of water by stages, in each of which the gas is first heated to above the dew point of the contained water vapour.—W. E. F. P.

*Hydrochloric acid from gaseous chlorine and hydrogen; Process of obtaining* —. C. W. Baumann, Düsseldorf-Unterrath. Ger. Pat. 305,306, July 31, 1917. Addition to Ger. Pat. 301,903.

Gaseous hydrogen chloride is added to the mixture of chlorine and hydrogen introduced into the explosion cylinder as described in the chief patent. This prevents the overheating of the explosion motor and thus enables the process to be carried out continuously.—C. A. M.

*Compounds of potassium and aluminium; Recovery of soluble* — *from silicates.* B. F. Halvorsen, Christiania, Norway. Eng. Pat. 107,012, May 30, 1917. (Appl. No. 7745 of 1917.) Under Int. Conv., May 30, 1916.

A MIXTURE of finely divided alkali aluminium silicate (felspar) and calcium cyanamide is heated, either to about 650° C. in the presence of superheated steam, or under pressure with water in an autoclave to above 100° C. The addition of salts, e.g., chlorides, nitrates, sulphates, etc., facilitates the reaction. When the ammonia has been driven off, the solution is filtered, neutralised with an acid, e.g., carbonic acid or nitric acid, the precipitated alumina is filtered off and the solution evaporated until crystallisation begins. A comparatively pure potassium salt separates out on cooling.—L. A. C.

*Silicon-bearing earths, rocks, minerals, and compounds; Process for decomposing* —. A. D. Hone, Peterborough, Canada. Eng. Pat. 116,118, May 9, 1917. (Appl. No. 6602 of 1917.)

THE finely divided material is mixed with a solid compound containing fluorine in sufficient quantity to remove the silicon as silicon fluoride, and an acid capable of decomposing the compound—e.g. sulphuric acid—either with or without the application of heat. The silicon fluoride formed is passed into water, thereby forming silica and hydrofluosilicic acid. The hydrofluosilicic acid is treated with another compound—e.g. the potassium compound obtained in the decomposition—thus forming a fluosilicate which may be used in the decomposition of other material.—L. A. C.

*Alkalis and alumina; Method of obtaining* — *from silicates containing them.* F. A. Rody, Johnson City, Tenn., Assignor to The Metallurgical Co. of America, New York. U.S. Pat. 1,263,705, Apr. 23, 1918. Date of appl., May 18, 1915. Renewed July 13, 1917.

A MIXTURE of felspar, leucite, or similar silicate, and an alkaline-earth oxide is heated to a sintering temperature, the clinker is digested with boiling water, and the dilute solution of alkali aluminate so obtained is used to treat further quantities of clinker until it becomes sufficiently concentrated.

—A. B. S.

*Salt manufacture; Method and system of* —. C. L. Well, Port Huron, Mich. U.S. Pat. 1,261,331, Apr. 2, 1918. Date of appl., May 10, 1917.

Is a method of producing salt from brine the latter is evaporated, first at the boiling-point to form small crystals, and then below the boiling-point to produce relatively large crystals. Of the two evaporators employed, the second is egg-shaped in cross-section and provided with means for regulating the depth, and hence the surface area, of liquid.—W. E. F. P.

*Ammonia; Production of* —. P. R. Hershman, Assignor to Armour Fertilizer Works, Chicago. Ill. U.S. Pat. 1,261,526, Apr. 2, 1918. Date of appl., Dec. 22, 1914.

ALUMINIUM nitride, alone or mixed with calcium oxide, is treated with superheated steam while maintained at a temperature approximating to but lower than 600° C.—W. E. F. P.

*bleach; Method of making high-strength* —. K. P. McElroy, Washington, D.C. U.S. Pat. 1,261,572, Apr. 2, 1918. Date of appl., Aug. 30, 1917.

From the products of reaction between steam and chlorine at about 100° C., aqueous hydrochloric acid is removed by condensation, and the residual vapour (hypochlorous acid) is absorbed by caustic alkali.—W. E. F. P.

*Zinc chloride; Production of* —. *Producing zinc chloride from ores*. F. K. Cameron and J. A. Cullen, Salt Lake City, Utah, and R. W. Hyde, New York, Assignors to American Smelting and Refining Co., Salt Lake City. U.S. Pats. (A) 1,261,685 and (B) 1,261,686, Apr. 2, 1918. Dates of appl., June 18 and July 17, 1917.

(A) OXIDISED zinc ore is treated with sufficient strong hydrochloric acid to combine with the bases present. The mass is then evaporated to dryness, maintained at about 300° C. until evolution of hydrochloric acid ceases, and treated with water to dissolve the zinc chloride. (B) Oxidised zinc ore containing lead is mixed with a metal chloride, and the mixture is heated to volatilise the lead as chloride. Zinc chloride is then recovered from the residue by leaching.—W. E. F. P.

*Kelp-dryer*. S. R. Oppenheim, Keeler, Cal. U.S. Pat. 1,266,041, May 14, 1918. Date of appl., June 7, 1917.

A HORIZONTAL, rotary cylinder is provided internally with lateral vanes between which a number of parallel cross plates are disposed obliquely. The vanes extend along the entire length of the cylinder and are inclined near the feed end, their inner edges being advanced in the direction of rotation so as to form lifting pockets. The kelp is caused to travel horizontally by the rotation of the cylinder.

—W. E. F. P.

*Steatite; [Electro-osmotic] process of removing liquid [water] from* —. B. Schwerin, Assignor to Elektro-Osmose A.-G. (Graf Schwerin Ges.), Frankfurt, Germany. U.S. Pat. 1,266,230, May 14, 1918. Date of appl., Nov. 11, 1914. Renewed Oct. 24, 1917.

STEATITE is finely subdivided in the presence of an electrolyte suitable for bringing the solid body into the sol condition, and of a colloid body (e.g. sodium silicate), and then subjected to electro-osmotic treatment.—W. E. F. P.

*Chalk; Process of making light precipitated* —. N. Statham, Hastings-upon-Hudson, Assignor to Industrial Chemical Co., New York. U.S. Pat. 1,266,339, May 14, 1918. Date of appl., July 25, 1917.

PURE hydrated lime liquor, having a density between 25° and 35° Tw. (sp. gr. 1.125–1.175), is sprayed repeatedly through an atmosphere rich in carbon dioxide and under a pressure of 60–80 lb. per sq. in. The liquor is then filtered and the residue dried at 110°–120° C. without agitation.

—W. E. F. P.

*Exchange silicates; Process of making* —. *Process of making exchange bodies*. G. Rudolf, London, Assignor to The Permutit Co., New York. U.S. Pats. (A) 1,263,706 and (B) 1,263,707, Apr. 23, 1918. Dates of appl., Dec. 9 and 22, 1916.

(A) A rocky material containing acid-soluble silicates is extracted first with hydrochloric acid and then with soda solution. The acid solution is neutralised with the soda solution, producing a precipitate which is dried at a low temperature. (B) A solution of an alkaline silicate is mixed with such a proportion of a solution of an aluminium salt containing an acid radicle that the mixture is distinctly alkaline to methyl orange, but not to phenolphthalein.—A. B. S.

*Gases [containing phosphorus pentoxide and silicon fluoride]; Condensing* — *from electric furnaces. Cooling and condensing gases*. I. Hechenbleikner, Charlotte, N.C., Assignor to Southern Electro-Chemical Co., New York. U.S. Pats. (A) 1,264,510 and (B) 1,264,511, Apr. 30, 1918. Date of appl., Feb. 10, 1917.

(A) GASES containing phosphorus pentoxide and silicon fluoride are treated to produce phosphoric acid and hydrofluosilicic acid, by first removing dust from the gases whilst they are in a heated state, and then electrically precipitating the phosphorus pentoxide as a solid, which is brought into contact with water to form phosphoric acid. The residual gas is passed through an absorption and condensation system in contact with water to form hydrofluosilicic acid. The acid is finally concentrated. (B) The hot gases are passed through a chamber in contact with water-cooled units to cool them and separate dust, and are then treated as described above.—B. N.

*Mono- and diammonium phosphate; Manufacture of* —. I. Hechenbleikner, Charlotte, N.C., Assignor to Southern Electro-Chemical Co., New York. U.S. Pats. (A) 1,264,513 and (B) 1,264,514, Apr. 30, 1918. Date of appl., Feb. 10, 1917.

(A) DILUTE phosphoric acid, containing 35 to 43%  $P_2O_5$ , is showered in a downward direction, and then collected to form a bath, and ammonia gas is introduced in divided streams at a point below the surface of the bath. The resulting gases flow from the bath as an upward counter-current into contact with the downward flow of acid to the bath. The heat of neutralisation is used to evaporate water from the bath and produce dry, non-hygroscopic, ammonium phosphate crystals. (B) The stream of phosphoric acid is showered downwards to preheat it by the escaping gas, and the liquid is conducted in a stream into contact with a stream of ammonia gas flowing in the opposite direction. The resulting crystals are dried by the conjoint effect of the heat of neutralisation and heat applied externally by means of an air blast.—B. N.



*Boron derivative of pyrophosphoric acid; Manufacture of a [non-hygrosopic] —.* B. Levin, Hale, Chester. Eng. Pat. 116,735, May 16, 1917. (Appl. No. 7015 of 1917.)

Pyrophosphoric acid (or orthophosphoric acid) is heated to 250° C., and a boron compound, e.g., boric anhydride, boric acid, or metaboric and/or pyroboric acid, added in such quantity that one atom of boron is provided for each two atoms of phosphorus. Heating is continued, the temperature being raised if necessary, until the mass is worked up to a homogeneous paste, which is run into trays and dried in a heated oven. The product, a pyrophosphoboric acid, having the formula  $BHP_2O_5$ , is non-hygrosopic, and may be ground to a powder or dissolved in water and converted into salts.

—L. A. C.

*Chlorides; Electrolysis of — [and production of chlorohydrocarbons]. Manufacture of alkalis [and chlorohydrocarbons].* K. P. McElroy. Assignor to Chemical Development Co., Washington, D.C. U.S. Pats. (A) 1,264,535 and (B) 1,264,536, Apr. 30, 1918. Dates of appl., (A) Dec. 9, (B) June 22, 1915.

(A) An aqueous solution of a chloride is electrolysed in a suitable chamber, at a temperature about 100° C., the alkali formed being removed and recovered. During the electrolysis, an olefine, oil gas, or unsaturated hydrocarbon, is conveyed into the vicinity of the anode at a rate sufficiently rapid to combine with the chlorine as quickly as it is produced. The vapours of the chlorinated product and aqueous vapour are cooled to condense and return the aqueous distillate, and the vapours are then further cooled to collect the chlorinated product. (B) A chloride solution is electrolysed in a thin layer between a pair of parallel diaphragms separating the anode and cathode, and during the electrolysis the anode is maintained in an atmosphere of an olefine or oil gas.—B. N.

*Potassium salts from mother-liquor salts; Process of separating —.* G. Sterling, Salt Lake City, Utah. U.S. Pat. 1,264,572, Apr. 30, 1918. Date of appl., Sept. 17, 1917.

An intimate mixture of mother-liquor salts (from brine) with silica and limestone is heated, with stirring, to effect combination between the silica and the magnesium and sodium (present as sulphate) of the mother-liquor salts and the calcium of the limestone. The mixture is then further heated to volatilise potassium salts and sodium chloride, whereby a condensed product relatively rich in potassium and free from magnesium salts is obtained. The silica of the original mixture may be replaced by a potassium-bearing silicate, in which case the non-volatile residue is treated to convert the insoluble potassium of the silicate into a soluble salt, and re-heated to recover the latter.

—W. E. F. P.

*Nitrogen compounds from metal carbides and nitrogen; Process of obtaining —.* V. Thrane, Christiania. Ger. Pat. 302,583, Oct. 29, 1916. Under Int. Conv., Nov. 29, 1915.

That part of the apparatus in which the metal carbide reaches the reaction temperature is constructed in the form of an inclined plane, so that when the reaction is complete the vessel containing the charge falls by gravity, and is thus automatically withdrawn to a cooler part of the apparatus with the object of preventing reversal of the reaction. The apparatus may also contain a cylindrical space having in its floor a turntable upon which the reaction vessels rest, and by means of which they may be conveyed to the inclined plane.

—C. A. M.

*Sulphites; Process of manufacturing solid neutral — from bisulphite solutions.* Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 304,867, Jan. 5, 1917.

A solution of a bisulphite is decomposed by means of oxides or carbonates suspended in the mother liquor from which the sulphite has separated in a previous operation. When spent the mother liquor is renewed by saturation with sulphur dioxide.—C. A. M.

*Elements of the air; Process for the separation by liquefaction and rectification of the —.* L'Air Liquide, Soc. Anon. pour l'Etude et l'Exploit. des Proc. G. Claude, Paris. Eng. Pat. 114,817, Oct. 17, 1917. (Appl. No. 15,082 of 1917.) Under Int. Conv., Apr. 13, 1917.

In Eng. Pat. 3326 of 1911 (this J., 1912, 335) is described a method of separating pure oxygen and pure nitrogen from liquid air, but with the disadvantage of a poor yield of oxygen, a large part of this gas leaving the apparatus mixed with nitrogen and argon. In order to avoid this loss, the liquid oxygen from the main rectifying column is introduced only towards the lower plates of the secondary rectifying column, and the escaping gases, after passing through a heat exchanger, are compressed and returned to a nest of tubes immersed in liquid oxygen at the bottom of the latter column, in which they are liquefied, with the exception of a small residue of nitrogen. The liquid is then passed up a tube which enters the secondary rectifying column on a level with the exit tube, and, as rectification proceeds, the escaping gases become poorer and poorer in oxygen. If the gaseous residue in the nest of tubes corresponds to the proportion of nitrogen accompanying the liquid oxygen—e.g., 2 or 3%—the escaping gas, part of which is extracted after leaving the heat exchanger, consists almost entirely of oxygen and argon. Of the air introduced into the apparatus, about four-fifths should be liquefied in a system of coils in the main column, and about one-fifth by the liquid oxygen in the bottom of the secondary column, whence the liquid produced is introduced into the main column to compensate for loss of liquid oxygen through the tube conveying the liquid to the lower part of the secondary column.

—L. A. C.

*Sulphides; Process for the production of metallic —.* C. O. Griffith, Wormit, Scotland. U.S. Pat. 1,261,023, Apr. 2, 1918. Date of appl., June 7, 1917.

See Eng. Pat. 105,830 of 1916; this J., 1917, 646.

*Sulphate of cadmium solutions; Electrolysis of —.* E. J. Hunt, Oldbury, and W. T. Gidden, Warley, Assignors to Chance and Hunt, Ltd., Oldbury. U.S. Pat. 1,264,802, Apr. 30, 1918. Date of appl., June 24, 1916.

See Eng. Pat. 14,273 of 1915; this J., 1916, 839.

*Reflux condensing apparatus for cooling salt solutions or other liquids.* Ger. Pat. 305,240. See I.

*Process of separating gases or vapours from gaseous mixtures.* Ger. Pat. 304,603. See I.

*Manufacture of ammonia or its salts.* Eng. Pat. 115,449. See IIa.

*Method of distilling pyroligneous acid.* U.S. Pat. 1,261,344. See IIb.

*Process for obtaining artificial magnesite for the production of artificial stone, cements, pigments, etc.* Ger. Pats. 305,418, 305,449, 305,446, and 305,447. See IX.

*Utilisation of nitre-cake [for treatment of copper ores].* Eng. Pat. 116,181. See X.

*Process of preventing agglomeration and hardening of ammonium sulphate during storage.* Ger. Pat. 305,715. See XVI.

*Process of preparing non-deliquescent water-soluble halogen calcium compounds.* Ger. Pat. 305,367. See XX.

### VIII.—GLASS; CERAMICS.

*Optical glass; Nomenclature of* —. W. Zschokke, Z. f. Instrumentenkunde, 1918, 38, 49–54.

The various systems of nomenclature of optical glasses are confusing and irrational, being based on historical considerations or chemical character without any relation to the optical properties. The new Sendlinger optical glass works, Berlin, has introduced a system which is intended not only to classify but to give an indication of optical properties also. The glasses are divided into four groups, "Kron," "Flint," "Barion," and "Barint," the last two names being contractions of "Barium-Kron" and "Barium-Flint," and covering the newer barium silicate glasses. The individuals in these groups are characterised by affixing two numbers which indicate the optical properties—e.g., Kron 567/628 indicates a crown glass of refractive index 1.567 and dispersive power (the  $\nu$  of Abbe) 62.8.—H. J. H.

*Silica bricks; Composition of refractory* —. J. S. McDowell. Eng. and Min. J., 1918, 105, 954–956.

The thermal conductivity of silica bricks is, according to Dudley, 25% greater than that of fireclay bricks of good quality. The greater part of the expansion which silica bricks undergo on burning is permanent and is due to the inversion of quartz. The temporary or true thermal expansion occurs largely over a narrow range of temperature and to it is attributed the tendency of silica bricks to spall. Bricks made of Medina quartzite from Huntingdon County, Pa., were subjected to eight and ten burnings respectively. Expressed in per cent. by volume their mineral compositions were: After 8 burns: silicates 14, cristobalite 23, tridymite 53%; after 10 burns: silicates 11, cristobalite 28, tridymite 61%. These results together with others obtained previously are shown graphically. The percentage of "quartz plus silicates" reaches an approximately constant value of 13% after the third burn and probably the whole of this 13% represents silicates formed by the lime used as bond and the impurities in the raw material. A completely inverted brick should contain about 87% tridymite and 13% silicates. Apparently 16 or 17 burnings, or one continuous burning for a month, would be needed to reach this composition, so that the cost is prohibitive. So far as permanent expansion is concerned, there is little advantage in more than one burning and, for practical purposes, none after the third burning. A silica brick made of Baraboo quartzite contained: quartz plus silicates 67%, cristobalite 27%, tridymite 6%; a brick of Medina quartzite contained: quartz plus silicates 25%, cristobalite 71%, tridymite 4%. The difference is attributed to differences in the

texture of the quartzites. The Baraboo quartzite being much coarser in grain than the Medina quartzite it cannot invert so rapidly. Whilst considering Wernicke and Wildschrey's statement that ordinary quartzites do not produce good silica bricks as too sweeping, the author confirms their general conclusion that the finer grained quartzites with a cement of amorphous silica are the most suitable for making bricks. He also confirms their statement that the selection of rock of the proper texture offers greater possibilities for the production of the best (tridymite) bricks than any alteration in the process of manufacture. A coarse texture brick spalls less than one composed of fine grains, but insufficient grinding may induce other defects. As there are often wide variations in the textures of quartzites of the same geological location, the averages of a sufficiently large number of grain-size measurements should be taken.—A. B. S.

*Porcelain and magnesia; Electrical resistivity of — at high temperatures.* P. H. Brace. Amer. Electrochem. Soc., Apr.—May, 1918. [Advanced proof.] 8 pages.

The porcelain used for sparking-plug insulators is unsatisfactory when employed as an insulator at 1000° C. because of current-leakage. Test-pieces, about 1 in. by 0.8 in., of electrically fused magnesite (containing 94% MgO) and of the same magnesite, fused electrically, then crushed to powder, moulded under heavy pressure, and burned at 1700° C., were placed on a grid of square nickel wire supported on an asbestos pad and loaded with a weight of about 11 lb., the weight being placed on an asbestos pad supported on a nickel grid. The loaded test-pieces were heated in a muffle furnace at 1000° C. for 24 hrs. and allowed to cool quickly. The temperature was then adjusted to the required value and time allowed for the test-piece to attain a uniform temperature. After this, an electric current of constant voltage (120 volts) was applied through the grids at each end of the test-piece and the current was measured at intervals. After a sufficient number of readings had been taken, the current was reversed and further readings were made. The results, plotted graphically, showed that the apparent resistivity is a function of the time during which the current is applied, the final value in some cases being several times the initial one. When the current was reversed the resistivity was lowered, but gradually rose to its former value. This occurred even after repeated reversals. Fused magnesite has a higher resistivity and a higher ratio of initial to final resistivity at each temperature than a piece of the porcelain used for sparking plugs, and the resistivity attains a constant value more quickly. This appears to be due to the porcelain being a complex mixture of mutually soluble oxides, whereas the magnesia is a relatively pure material. The moulded sample of magnesia had slightly lower resistivity values than the other sample, but the difference was not marked; it may be due to the introduction of impurities during grinding and firing. At 910° and 990° C., the porcelain behaved like a storage battery and on stopping the current it gave an electromotive force of 0.7 volt, in the opposite direction, which decayed slowly, but was appreciable after half an hour. This behaviour suggests electrolytic phenomena.

—A. B. S.

*Magnesia-silica mixtures; Crushing strength of — at high temperatures.* O. L. Kowalko and O. A. Hougen. Amer. Electrochem. Soc., Apr.—May, 1918. [Advance copy.] 11 pages.

MIXTURES of magnesia and silica in various proportions were made into cylinders 2.16 in. high by



1 in. diam., fired at 2100° C., and afterwards reheated in an electric furnace at the rate of 10° C. per min. under a load of 66.5 lb. per sq. in., the temperature at which failure occurred being noted. The heat-treatment during the first firing and the rate of heating under load had considerable influence on the result. The best results were obtained with mixtures containing 7 to 8% of silica: these failed at 1870° C., whilst pure magnesia failed at 1680° C. The failure of cylinders of pure magnesia was gradual; that of the mixtures was abrupt. When the cylinders were fired at or above 1900° C. they lost weight owing to the reaction between them and the carbon supports used to transmit the pressure. Cylinders fired at 1800° C. contained periclase enveloped in and bonded with forsterite. Below 1500° C. there was little forsterite and the strength was low; at 2000° C. the forsterite collected into separate crystals of larger size and consequently the strength of the cylinders was reduced. When the cylinders were heated and then quenched in cold water, the formation of forsterite was repressed though some was still formed in the grain-boundaries of the periclase.—A. B. S.

#### PATENTS.

*Glass furnaces.* A. F. Peeters, Lingedijk, Holland. Eng. Pat. 116,477, Feb. 11, 1918. (Appl. No. 2439 of 1918.)

In a rectangular tank furnace the "burner" is placed at the end of the working space, the gases travelling first through the working space and then through the melting space, instead of in the opposite direction, as is customary. Advantages of the new arrangement are that the glass is melted on the counter-current principle, the solid materials being fed into the coolest part of the furnace and the molten glass flowing gradually to the zone of the highest temperature; the distribution of heat in the furnace allows the glass to be melted more efficiently; the burning fuel (e.g. tar) enters so hot a portion of the furnace that no deposition of carbon occurs and no undesired coloration of the glass need be feared.—A. B. S.

*Glass and method of making it.* E. W. Enequist, Brooklyn, N.Y. U.S. Pat. 1,261,015, Apr. 2, 1918. Date of appl., July 20, 1917.

Fused lepidolite (lithia mica) is added to a glass batch and the whole is then fused so as to produce a glass of superior strength, temper, and appearance.—A. B. S.

*Ceramic and like ware: Manufacture of [lustrous]* —. Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. Eng. Pat. 113,777, Sept. 27, 1917. Under Int. Conv., Feb. 28, 1917. (Appl. No. 13,963 of 1917.)

Lustrous coatings on ceramic ware are produced by applying the coating material in the form of a colloidal sol and then firing the ware to a temperature below the melting point of the coating. Suitable coating materials are clays, many silicates, zirconia and other oxides, carborundum, etc. These may usually be converted into the sol state by suspending them in water containing a small proportion of an electrolyte and allowing the coarser particles to settle; by the method described in Eng. Pat. 14,235 of 1912 (see Addition to Fr. Pat. 426,072; this J., 1912, 1189); or by alternate treatment with acids and alkalis. The degree of lustre obtained varies with the degree of dispersion of the colloid.—A. B. S.

*Kiln; Sectional* —. R. H. McElroy, Assignor to International Clay Machinery Co., Dayton, Ohio. U.S. Pat. 1,264,316, Apr. 30, 1918. Date of appl., July 2, 1917.

A RECTANGULAR kiln, having a perforated sole, is provided with a number of sections, arranged longitudinally, each section having bag-walls at the sides, forming conduits for the heated air and the products of combustion, together with longitudinal connecting flues between the sections, and lateral hot air flues or *vice versa*.—A. B. S.

*Kiln; Compartment* —. R. H. McElroy, Assignor to International Clay Machinery Co., Dayton, Ohio. U.S. Pat. 1,264,317, Apr. 30, 1918. Date of appl., Sept. 1, 1917.

A KILN is divided into compartments, which are connected by flues not in alignment, the length of each flue being greater than the width of one compartment, so that several compartments may be heated simultaneously. The compartments are also connected by air flues, which in turn are connected by lateral flues, and by means of a hood the lateral flues may be connected with either the main draught flue or the waste-heat flue through uptake flues.

—A. B. S.

*Kiln-system.* J. T. H. Warwood, Wadsworth, Ohio. U.S. Pat. 1,264,722, Apr. 30, 1918. Date of appl., July 7, 1917.

A SERIES of round kilns with fire-boxes, perforated hearths, and vertical flues is connected through the perforated hearths with an upper, central, longitudinal flue, and the vertical flues are connected with a lower, central, longitudinal flue. These upper and lower flues are connected to each other by means of damper-controlled ports, an upper lateral flue, and transverse flues meeting the upper central flue between the ports and the dampers. A lower lateral flue is connected to the lower central flue through a transverse passage and to an exhaust. The portions of each kiln connected to the upper and lower central flues respectively may be connected *vice versa*.—A. B. S.

*Vitreous enamel; Production of articles enamelled with a* —. K. Waga, Brooklyn, N.Y., U.S.A. Eng. Pat. 116,361, June 11, 1917. (Appl. No. 8319 of 1917.)

SHEETS of aluminium or its alloys are heated to 500°–700° C. and covered with a thin layer of finely powdered glass, enamel, or other vitrifiable material, so as to form an adherent coating which will not crack when the sheets are afterwards made into articles of the desired shapes.—A. B. S.

(A) *Aluminous abrasive.* (B) (c) *Aluminous abrasive and process of making the same.* L. E. Saunders and R. H. White, Niagara Falls, N.Y., Assignors to Norton Co., Worcester, Mass. U.S. Pats. (A) 1,263,607, (B) 1,263,709, and (c) 1,263,710, Apr. 23, 1918. Dates of appl., (A) Dec. 26, (B) and (c) Aug. 2, 1917.

(A) A CRYSTALLINE, aluminous abrasive containing material quantities of an acid oxide (as silica) and an alkali, so proportioned as to yield a relatively weak abrasive grain as compared with aluminous grains of similar acid oxide (silica) content, but free from alkali. (B) A crystalline abrasive with properties similar to (A) is made by fusing a mixture of alumina and a sodium compound in an electric furnace. The sodium compound is

vaporised and causes individual crystals of the solidified product to be perforated or cellular. (c) An abrasive with properties similar to (A) and (B) is made by mixing bauxite with a small proportion of an alkali compound, with or without carbon, and fusing in an electric furnace.—A. B. S.

*β-Alumina; Product [abrasive] containing — and process of preparing the same.* L. E. Saunders and R. H. White, Niagara Falls, N.Y., Assignors to Norton Co., Worcester, Mass. U.S. Pat. 1,263,798, Apr. 23, 1918. Date of appl., Mar. 2, 1917.

An aluminous material is fused with sufficient sodium carbonate to convert a material proportion, for example, more than half, of the alumina into the β-modification. The product may be used as an abrasive.—A. B. S.

*Earthenware and like articles; Cranks for supporting — during the process of firing.* W. P. Edwards, and J. Edwards and Sons, Ltd., Stoke-on-Trent. Eng. Pat. 116,211, Feb. 13, 1918. (Appl. No. 2580 of 1918.)

## IX.—BUILDING MATERIALS.

*Cork slabs from cork waste.* Ost. See V.

*Composition of refractory silica bricks.* McDowell. See VIII.

### PATENTS.

*Concrete or cement substances, and the composition thereof.* L. Lunden, London. Eng. Pat. 114,641, Mar. 6, 1917. (Appl. No. 3317 of 1917.)

CONCRETE or cement substances are made by intimately mixing Portland cement or the like; vegetable matter such as moss, turf, seaweed, cork, or fibres; finely divided iron ore or other metallic ore; a binder consisting of 5 kilos. of fresh quick-lime, 3 kilos. of aluminium sulphate, and 1 cubic metre of water; and fatty matter introduced in the form of strings of jute or other textile material or fragments of pumice, cork, etc., impregnated with oil or grease. The binder is prepared in a mechanical mixer, the vegetable matter is added, and then the cement is introduced slowly and is followed by the ore, the mixing being continued without intermission during the whole of the time. The impregnated strings or other material are arranged in layers in the composition whilst it is still soft or they may be arranged in conjunction with the ordinary reinforcement. As the cement or concrete dries, the fatty matter gradually permeates it, producing a structure of great resiliency and resistance to shock.—A. B. S.

*Cement from blast-furnace slag; Manufacture of —.* A. A. J. Deckers, Paris. Eng. Pat. 114,794, Dec. 11, 1917. (Appl. No. 18,280 of 1917.)

GRANULATED slag is ground under water, dried in a centrifuge until it contains less than 7% of water, and then mixed with a suitable proportion of ground lime and the mixture ground to the requisite fineness. By the preliminary grinding of the slag the enmeshed water is liberated and its removal facilitated; if the water were not removed, the cement mixture would set hard in the mill and could not be ground.—A. B. S.

*Plaster composition.* P. S. Auneke, Duluth, Minn., Assignor to Celite Products Co., Los Angeles, Cal. U.S. Pat. 1,264,747, Apr. 30, 1918. Date of appl., Aug. 21, 1916.

HYDRATED lime and amorphous silica (kieselguhr) are ground together to form a plaster composition.—A. B. S.

*Plaster composition.* H. Blumenberg, jun., Los Angeles, Cal., Assignor to F. Blumenberg, New York. U.S. Pat. 1,266,200, May 14, 1918. Date of appl., Oct. 1, 1917.

THE material contains equal parts of dead-burnt gypsum and hemi-hydrated calcium sulphate.

—W. E. F. P.

*Magnesite; Process for obtaining artificial — for the production of artificial stone, cements, pigments, etc.* Harburger Chem. Werke Schön & Co., and W. Daitz, Harburg, Ger. Pats. (A) 305,448 and (B) 305,449, Mar. 3; (C) 305,446, Oct. 10, and (D) 305,447, Dec. 1, 1914.

(A) IS the usual method of recovering magnesia by the addition of a mixture of lime and magnesia to a liquor containing magnesium chloride, residues containing calcium hydroxide are produced which react very slowly and delay the process of recovery. Magnesium hydroxide also separates very slowly towards the end of the process. These difficulties are overcome by applying pressure and heat to the whole of the mixture or to the liquid obtained after separating the bulk of the precipitated magnesia. A pressure of 1–3 atmospheres is usually sufficient. (B) In the production of magnesia as free as possible from lime, and therefore suitable for pigments, excess of precipitant is used and the residual liquor then contains some magnesium chloride as well as calcium chloride. This waste liquor is used to dilute a further quantity of magnesium chloride liquor previous to the treatment of the latter with a mixture of lime and magnesia, and its content of magnesium chloride thus utilised. (C) To a liquor containing magnesium chloride is added a mixture of lime and magnesia containing lime equivalent to the magnesium chloride. The mixture is stirred thoroughly and heated, with constant stirring, until a stiff paste is produced. The solid matter is then separated, dried and carefully calcined. The calcined magnesia possesses a strong binding power which makes it specially useful in the manufacture of artificial stone and cement. (D) Burned dolomite or other mixture of lime and magnesia is treated with sufficient water to slake it partially and is then added to a liquor containing magnesium chloride so as to produce a slurry containing magnesium oxide and hydroxide. The suspended matter is separated, dried, and carefully calcined. The partially hydrated mixture of lime and magnesia may be sifted before use so as to eliminate the coarser particles. The use of a partially hydrated mixture yields a material of superior shaping and binding power and permits a magnesium chloride liquor of higher concentration to be employed.—A. B. S.

*Cementing materials; Method of manufacturing — and products thereof.* H. W. Charlton, Jones Point, N.Y., U.S.A. Eng. Pat. 115,946, June 27, 1917. (Appl. No. 9249 of 1917.)

SEE U.S. Pats. 1,242,421, 1,242,422, and 1,249,028 of 1917; this J., 1917, 1237; 1918, 59 A.

*Fireproofing solution.* U.S. Pat. 1,261,736. See I.

*Bituminous binder.* U.S. Pat. 1,264,932. See IIA.



## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Steels; Critical points of pure carbon* —. P. Bardenheuer. *Ferrum*, 1917, 14, 129–133, 145–151. *Z. angew. Chem.*, 1918, 31, Ref., 170.

THE A<sub>1</sub> point occurs in all plain carbon steels at 740° C. A<sub>1</sub>1, however, rises from 706° C. for almost carbonless iron to 722° C. for the eutectoid composition, after which it remains constant. The hysteresis in the A<sub>1</sub> point is thus apparently related to the ferrite present. A<sub>2</sub> and A<sub>2</sub>2 are found as separate points up to 0.41% C, and both occur at 770° C. irrespective of the carbon content. The  $\beta$ — $\gamma$  change for a steel with 0.06% C occurs at 911° C., A<sub>3</sub> being at 886° C. The A<sub>3</sub> hysteresis, it is stated, decreases from 25° C. for pure iron to nil for 0.9% C. The A<sub>3</sub> point is lowered rapidly by carbon when present up to 0.41%, the  $\gamma$ — $\alpha$  change being then lowered more slowly to 0.9%, the rates being 3:1. The eutectic point is given as 0.85% C. —F. C. Th.

*Steel; Magnetic analysis of carbides found in different kinds of* —. T. Ishiwara. *Sci. Rep. Tôhoku Imp. Univ.*, 1918, 6, 285–294.

THE magnetic properties of cementite obtained from various kinds of steel have been examined, and evidence of the decomposition of the carbide is afforded by the sudden increase in the susceptibility between 650° and 730° C. After the heat treatment the presence of magnetite formed by the oxidation of the cementite is recognised by the transformation which occurs at 589° C. Natural magnetite and chemically prepared magnetic oxide are differentiated by their transformation temperatures, these being 580° and 510° C. respectively. Separation of cementite from the solid solution of an eutectoid steel begins at about 130° C., and is nearly complete at 360° C. The three ranges of temperature separated by 130°, 360°, and 600° C. correspond respectively with martensite, troostite, and sorbite. According to magnetic observations, martensite is a solid solution of the carbide in  $\gamma$ -iron, whilst troostite represents the stages in which cementite separates out in colloidal form, the limit to this series being reached at 360° C., with the formation of osmondite. Sorbite has the same magnetic susceptibility as pearlite. Residues from quenched specimens contain only a small quantity of cementite, but the amount of this increases with the tempering temperature up to 360° C. The residues are very reactive and ignite in the air, but the oxidisability decreases as the tempering temperature increases. The examination of the carbides from special steels has shown that the double carbides of iron and manganese and of iron and tungsten are magnetic, whilst the residues from chromium steel and high-speed tool steel are paramagnetic.—H. M. D.

*Iron-carbon alloys; The processes of melting and freezing in* —. R. Ruer and F. Goerens. *Ferrum*, 1917, 14, 161–177. *Z. angew. Chem.*, 1918, 31, Ref., 168.

THE “equilibrium” temperature for the melting and freezing of the cementite eutectic of white pig iron is given as 1145° C., and that of the graphite eutectic, i.e., grey iron, as 1152° C. It is shown that in pure iron-carbon alloys the formation of graphite occurs both on heating and cooling as a result of the presence of the liquid phase, cementite or solid solution being of minor importance. The precipitation occurs directly from the liquid phase and not by decomposition of a previously separated

crystalline phase. The “equilibrium” temperature for the formation of pearlite, i.e. the mean of A<sub>1</sub> and A<sub>1</sub>1, is given as 721° C. corresponding to 0.9% C.—F. C. Th.

*Carbon steels; Determination of the velocities of cooling necessary to realise the hardening of* —. P. Chevenard. *Comptes rend.*, 1918, 166, 682–685.

Using the photographic method previously described (see this J., 1917, 881) the author has studied the cooling curves of steels containing from 0.2 to 0.8% carbon. Different rates of cooling were obtained by using cooling atmospheres consisting of varying proportions of nitrogen and hydrogen. For different temperatures of heating, plotting carbon content against velocity of cooling, the curves which mark the limit of appearance of martensite were determined. As the carbon content decreases from 0.8%, the maximum velocity of annealing (marked by first appearance of martensite) relative to a given temperature of heating,  $\theta_c$ , increases at first slowly, but below 0.5% carbon it rises with extreme rapidity. Thus it is impossible to lower to any marked extent by simple cooling the temperature of transformation of pure iron. For high carbon steels, A<sub>1</sub> falls rapidly to a very low value when  $\theta_c$  rises and consequently it is possible to define, for a given mode of cooling, a minimum temperature of complete hardening  $\theta_1$ . When, however, the carbon content is low, A<sub>1</sub> and A<sub>2</sub> tend slowly towards distinct limits according to the method of cooling. There is thus, for each velocity of cooling, a maximum intensity of hardening, which is greater as the velocity is higher. In other words, in regard to efficiency of hardening, it is possible to compensate, to a certain extent, for an insufficient speed of cooling by increasing the temperature of heating, but this compensation is less possible the lower the carbon content of the steel. —W. G.

*Steel; Macrostructure of* —. A. Portevin and V. Bernard. *Rev. Mét.*, 1918, 15, 273–280.

THE macrostructure of steel, which is determined by the first freezing out from the liquid state, is best developed by etching a polished section with Stead's reagent (this J., 1915, 616) as modified by Le Chatelier (this J., 1915, 1056; 1917, 1051), and then lightly repolishing. The reagent brings out well the original dendritic structure as a result of the changes in the phosphorus content. An identical structure in cast and unworked steel is shown by sulphur printing. The etching also enables the exterior elongated crystallisation of a casting or ingot to be distinguished from the internal equi-axed type. The macrostructure of forged steels shows the deformed dendrites drawn out in the direction of the elongation. When the working has been sufficient, the dendrites are drawn out into a bundle of parallel lines in a longitudinal section. Transversely the initial structure is retained but on a much finer scale. The longitudinal structure enables the degree of forging to be estimated. Heat treatments used in practice do not modify the macrostructure. Heating to 850° to 900° C. for 3 to 6 days results in diffusion occurring, with the result that the primary structure is lost. —F. C. Th.

*Mild steel; Influence of hot working on the mechanical properties and structure of low carbon* —. F. Wüst and F. Huntington. *Stahl u. Eisen*, 1917, 37, 829–836, 849–857. *Z. angew. Chem.*, 1918, 31, Ref., 167–168.

THE metal was rolled at temperatures between 550° C. and 1100° C. in 50° C. steps and then tested in tension and under impact. Three distinct zones of temperature could be distinguished by the resist-

ance to impact, the yield point, and structure, namely above A3, between A3 and A1, and below A1. In iron rolled above A3 marked brittleness is developed depending on the extent of the over-heating. This condition, however, can be entirely removed by subsequent rolling. Between A3 and A1 the resistance to impact increases with the rolling up to a certain critical amount of work, after which it rapidly decreases. Below A1 the impact value falls off steadily as the work put upon the metal increases. At the temperatures of A1 and A3 the material shows a maximum loss of resistance to impact for all degrees of work. As the work is increased on the metal rolled at temperatures above A3 the yield point rises slightly. Between A3 and A1 considerable variations occur; the yield point at first increases rapidly with the degree of work to a "critical" value, after which a sudden fall to a very low figure occurs. Below A1 the yield rises with the degree of work, reaching very high figures. The highest values of the yield point are found at the lower temperatures and fall off quickly to 900° C., the lowest values being found for the pieces rolled at 1100° C. The maximum stress follows unmistakably but less definitely the changes in the yield point and the elongation is, in general, inversely proportional to the yield. The principal changes in structure are a refining of the grain above A3, a crushing of the crystals between A3 and A1, which leads at a critical combination of deformation and temperature to the development of a coarse grain, and below A1 an elongation of the crystals with traces of recrystallisation.

—F. C. Th.

*Steel; Gases in alloy* —. A. Stadeler. Stahl u. Eisen, 1917, 37, 1075—1077. Z. angew. Chem., 1918, 31, Ref., 168.

THE gases dissolved in steel consist chiefly of carbon monoxide and hydrogen. Hydrogen predominates at low temperatures, but as the temperature is raised, decreases to a minimum value, after which it again rises with further heating. Carbon monoxide is present only in small quantities at low temperature, but rises on heating to a maximum and then commences to decrease. At high temperatures carbon monoxide and hydrogen are present in more or less equal amounts. Carbon dioxide, methane, and nitrogen do not as a rule constitute more than 5% of the total gases present. The evolution of gases on boring is related to the critical points, the greatest development occurring at these temperatures. In special steels the gases present decrease as the nickel, silicon, chromium, manganese, or other special constituent is present in increasing amounts. The gases dissolved are similar to those present in carbon steels with the exception that silicon and manganese lower the content of carbon monoxide and increase that of hydrogen. Chromium appears to increase the amount of nitrogen present. As in the case of ordinary steels the largest volumes of gas are set free in the region of the critical points.—F. C. Th.

*Steel; Influence of titanium on the formation of pearlite in carbon* —. R. Vogel. Ferrum, 1917, 14, 177—197. Z. angew. Chem., 1918, 31, Ref., 170.

THE alloys were prepared from ferrotitanium, it being found impossible to use pure titanium reduced from the oxide. The addition of titanium has no influence on the temperature of the A1 point, but between 1% and 6% Ti this element gives rise to a constituent resembling osmondite. Self-hardening steels never occur in the normal pearlite-free titanium alloys, the suppression of the carbon change arising from the increased inertia and lowered velocity of the pearlite transformation due to the presence of the titanium.

Very slowly cooled titanium steels are hard and withstand heating above the critical point without losing their hardness. When as a result of prolonged heating such steels are rendered pearlitic they are always harder than the plain steel of the same carbon content. Long annealing above the change point and not too slow cooling results in the loss of the hardness. The tensile properties of these steels are related to their content of titanium nitride and carbide, the first being extremely hard and the second very brittle. By melting these alloys in an atmosphere of hydrogen the formation of the nitride at the expense of some of the titanium carbide dissolved in the steel is prevented. The hardness of steels of constant carbon content but of increasing titanium is increased, while at the highest percentage the toughness is somewhat reduced.—F. C. Th.

*Electric steel castings*. R. F. Flinterman. Amer. Electrochem. Soc., Mar., 1918. [Advance copy.] 9 pages.

GREATER output of steel for castings resulted from replacing the copper spacers between the bus bars of an Heroult furnace by "vulc-asbestos" spacers and insulating the various bus-bar leads from one another by means of mica. The fall in voltage was much reduced due to the reduction of skin effect, and it is suggested that further improvement might be effected by replacing the solid bus construction entirely by flexible cable carried directly through to the electrode clamps, even allowing for induction set up in the steel structure of the furnace.—C. A. K.

*Tungsten steel; Structure of — and its change under heat treatment*. K. Honda and T. Murakami. Sci. Rep. Tohoku Imp. Univ., 1918, 6, 235—283.

SYSTEMATIC observations have been made of the magnetic properties and microstructure of tungsten steels containing 0.12 to 1.57% C and 0.23 to 30% W. Iron and tungsten form only one compound,  $\text{Fe}_2\text{W}$ , and the tungsten in tungsten steels may be present in this form or as a constituent of tungsten carbide, WC. At the ordinary temperature, iron tungstide is soluble in iron to an extent represented by 9% of tungsten, and in steels which contain larger quantities of tungsten, the tungstide occurs as a separate phase in globular form. Since a portion of the carbon in the steels forms tungsten carbide, the quantity of tungsten required for the appearance of the globules increases with the carbon content. Tungsten carbide combines with cementite to form the double carbide,  $4\text{Fe}_3\text{C} \cdot \text{WC}$ , with a critical temperature at 400° C. The double carbide dissociates above the Acl point, but recombination of the components occurs during cooling, the final result being an eutectic mixture with ferrite. At high temperatures, tungsten carbide reacts with iron according to the equation,  $\text{WC} + 5\text{Fe} = \text{Fe}_5\text{C} + \text{Fe}_2\text{W}$ . The resulting lowering of the transformation points increases with the tungsten content and reaches a maximum when this corresponds with the saturated solution of the tungstide in iron. In steels which contain more than 9% W, the lowered Ar1 point is constant at about 440° C. provided the rate of cooling is normal. If a tungsten steel with the lowered Ar1 point is reheated above the Acl point (to about 900° C.) and cooled, the Ar1 transformation takes place at the correct temperature. This is due to the fact that tungsten carbide is formed in accordance with the equation,  $\text{Fe}_5\text{C} + \text{Fe}_2\text{W} = \text{WC} + 5\text{Fe}$ . The rate of cooling has a marked influence on the transformation points. A large number of photomicrographs and curves accompany the authors' description of their observations and facilitate the interpretation of the somewhat complicated relations.—H. M. D.



*Nickel steels; Cause of irreversibility of* —. K. Honda and H. Takagi. Sci. Rep. Tohoku Imp. Univ., 1918, 6, 321–340.

THE magnetic behaviour indicates that the irreversibility of nickel steels is to be attributed to the influence of dissolved nickel on the A3 transformation. The irreversibility is not confined to steels containing less than 35% Ni, but probably extends to a nickel content of about 70%. A theoretical explanation of the influence of nickel on the magnetic behaviour of iron is given. The non-expansibility of the alloy containing 35.4% Ni (invar) may be accounted for without assuming the existence of the compound,  $\text{Fe}_3\text{Ni}$ .—H. M. D.

*Gas- and water-pipes; Corrosion of — in clay soils containing calcium sulphate.* P. Medinger. J. Gasbeleucht., 1918, 61, 73–77, 89–91.

Iron piping, especially cast iron, laid in clay soils containing calcium sulphate and bicarbonate, suffered unusually rapid corrosion, becoming soft and spongy without losing its form or changing its volume. In the case studied most of the pipe had disintegrated, leaving a residue of iron in the metallic condition, graphite, and silicon, phosphorus, and sulphur in oxidised forms. The corrosion is apparently electrolytic, due to local action of iron and graphite couples, although the E.M.F. of this couple (0.6 volt) is much less than the decomposition potential of water. Even 0.1 volt suffices to cause current flow due to diffusion of ions, which prevents the establishment of permanent equilibrium. The concentration of  $\text{H}^+$  ions from the bicarbonate is the deciding factor on the rate of corrosion. The addition of calcium sulphate increases the concentration of  $\text{H}^+$  ions. The clayey character of the soil leads to increased retention of electrolytes which operate as above, and also to the exclusion of oxygen, which retards corrosion by the formation of a protective oxide layer and consequent interruption of current flow. In such soils it is recommended to lay the pipes in a layer of sand or porous earth. Care should be taken that the protective coating of paint, etc., on new pipes is intact, especially at the flanges, which are more liable to abrasion.—H. J. H.

*Electric [steel] furnace; The Booth-Hall* —. W. K. Booth. Amer. Electrochem. Soc., May, 1918. [Advance copy.] 11 pages.

THE Booth-Hall furnace is of the vertical are conducting hearth type, an auxiliary electrode acting as a return for the current in the starting operation, to ensure a positive and balanced load. As soon as a pool of molten metal is formed in the hearth, the auxiliary electrode is drawn away and the normal working through the conducting hearth commences. In the two-phase furnace two grids embedded in the hearth and insulated from each other are so related to the main electrodes that the current from the two phases crosses in the bath, ensuring good circulation and mixing of the molten metal. The hearth (either acid or basic) is sintered together in thin layers by using the main electrodes in conjunction with the auxiliary electrode.—C. A. K.

*[Electric] furnace; The Vom Baur are* —. J. H. vom Baur. Amer. Electrochem. Soc., May, 1918. [Advance copy.] 7 pages.

IN the design of the Vom Baur solid bottom, poly-phase, arc furnace for melting ferro-alloys or steel (U.S. Pat. 1,252,633; this J., 1918, 156 A), particular stress is placed on the contour of the furnace walls, whereby local destruction of the brick-work is minimised. The central electrode carries

the heaviest current, compensating for the heap of metal naturally piled near the centre during charging, and the smaller electrodes can be tilted to facilitate the melting of this metal heap, or to search for local cold metal. In the normal vertical position of the electrodes the heat at the slag line and also over the entire side walls is uniform. Disturbances of the high power factor, and of the phases and arcs are avoided by keeping all heavy copper conductors well away from the furnace casing and electrode standards.—C. A. K.

*Carbon in steel; Rapid determination of* —. W. L. Jackson. Met. and Chem. Eng., 1918, 18, 564–565.

A COMBUSTION tube of fused silica is wrapped with resistance wire for a length of 9 in., and packed with quicklime in a steel tube casing. Steel samples are burned in small boats made from nichrome ribbon. Oxygen is admitted so as to allow a maximum of 15 lb. pressure to the train of absorption vessels, the pressure being registered by a mercury gauge. Previous to entering the tube it is washed by means of stick caustic potash, strong caustic potash solution, soda-lime, and calcium chloride contained in four towers of about 7 in. height, the stoppers of which are clamped in. After combustion the gas passes to three sets of zinc-phosphoric anhydride tubes and thence to Johnson absorbing bulbs. Three combustion tubes are used for each furnace—one running, one being weighed, and one in reserve. Results on plain carbon steels check to the third decimal place, and the author has repeatedly made 90 determinations in eight hours.—C. A. K.

*Sulphur in iron and steel; Determination of* —. H. Pinsl. Chem.-Zeit., 1918, 42, 363–371.

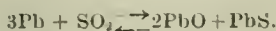
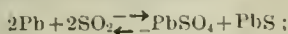
THE hydrogen sulphide evolved when iron is treated with hydrochloric acid may be absorbed in 5% sodium hydroxide solution; the absorption is complete if the alkali solution is contained in a vessel 19 cm. high by 20 mm. wide. The sulphide is then determined by precipitation as cupric sulphide after the alkaline solution has been neutralised, or it may be titrated iodometrically.—W. P. S.

*Milling [gold and silver ores] in cyanide solution.* A. W. Allen. Met. and Chem. Eng., 1918, 18, 515–519.

ONE of the chief advantages of milling in cyanide solution is the intimate contact resulting between the coarse metal and solvent, though it renders the process of amalgamation only partly effective. Only 90–95% of the gold is usually recoverable by cyaniding, whereas if the gold is amenable to amalgamation, practically 100% can be recovered. The fact that all the gold is soluble in cyanide is reaffirmed, but loss is occasioned, amongst other reasons, by the absorption of metal-bearing solution by colloid particles. Pyritic ores may be roasted, with recovery of sulphur, and the gold is then released in a form suitable for extraction. The acidity of ores having an acid reaction should be neutralised before cyaniding to avoid the loss of hydrocyanic acid. By milling in cyanide solution the possibility of mechanical loss is augmented as the quantity of metal-bearing solution is increased and for the same reason the cost of precipitation is higher. The process is specially applicable when there is no advantage in a preliminary alkaline treatment of the ore, when amalgamation would result in no appreciable saving, or when adequate dewatering prior to cyaniding is impracticable.—C. A. K.

*Copper; Influence of lead on molten — containing gases.* W. Stahl. Metall u. Erz, 1917, 14, 365—366. Z. angew. Chem., 1918, 31, Ref., 155.

LEAD when present in copper melts, influences the duration of the reactions and removes absorbed sulphur dioxide, hydrogen, and carbon monoxide, thus rendering the metal dense and preventing it from rising. The reduction of the cuprous oxide by lead,  $2\text{Cu}_2\text{O} + 2\text{Pb} = 4\text{Cu} + 2\text{PbO}$ , prevents the reaction,  $2\text{Cu}_2\text{O} + \text{Cu}_2\text{S} = 6\text{Cu} + \text{SO}_2$ , from occurring. In the temperature range  $550^\circ\text{--}900^\circ\text{C.}$ , and with high pressure of sulphur dioxide—up to 850 mm. of mercury—lead also reacts with sulphur dioxide according to the equations:



At lower pressures of sulphur dioxide and higher temperatures these reactions are reversed. At  $1300^\circ\text{C.}$  and low pressure—the conditions of the molten copper bath—lead does not react with sulphur dioxide by itself, but the reaction  $2\text{Pb} + 2\text{Cu} + \text{SO}_2 = 2\text{PbO} + \text{Cu}_2\text{S}$  takes place.—F. C. Th.

*Copper cyanide plating bath; Experiments with the —.* F. C. Mathers. Amer. Electrochem. Soc., May, 1918. [Advance copy.] 6 pages.

THE solution recommended is copper cyanide 4.5% (6 oz. per gallon), sodium cyanide 4.3 to 4.7% (5.7 to 6.3 oz.), sodium carbonate 4% (5.3 oz.). This bath, which is more concentrated than that usually employed, gave approximately 100% yields at both electrodes unless the operating conditions were severe or too much cyanide was present, when the cathode yields tended to become lower. The bath can be worked at 6 to 9 amps. per sq. ft. Much better cathode yields were obtained when the baths were operated at  $80^\circ\text{C.}$  than at the ordinary temperature. Using the stronger solution recommended, a thicker deposit can be obtained without bad edges as well as higher cathode yields than with weaker solutions. Very little free cyanide is required. Sodium carbonate is of advantage, as is also probably sodium sulphate. Sodium hydroxide should not be added.—F. C. Th.

*Copper; Effect of iron sulphate in the electrolytic precipitation of — from sulphate solution with insoluble lead anodes.* E. F. Kern. Amer. Electrochem. Soc., May, 1918. [Advance copy.] 11 pages.

THE presence of iron lowers the efficiency of the electrolytic precipitation of copper from sulphate solutions when insoluble lead anodes are used, the loss of efficiency increasing with the quantity of iron present. This effect also increases as the amount of copper decreases, hence the efficiency decreases progressively during the electrolysis. The lowering of efficiency is due to the oxidation of ferrous sulphate to the ferric state at the anode and the reduction of ferric sulphate at the cathode. When iron is present a higher current efficiency is obtained by working with a lower current density at the cathode than at the anode. A smoother and purer deposit was obtained at  $50^\circ\text{C.}$  than at lower temperatures. Iron in solution renders the deposit less smooth and less dense, and reduces the voltage required for the electrolysis from 2 to approximately 1.8 volts.—F. C. Th.

*Zinc ores; Anthracite as a reducing agent for —.* O. Mühlhaeuser. Metall u. Erz, 1918, 15, 162—165.

ANTHRACITE is considered to be specially suitable for the reduction of zinc ores. It gives a high yield of a very porous coke, and coking occurs at

a lower temperature than in the case of bituminous coals, and there is no risk of dilution of the zinc vapour by large volumes of volatile products. The use of anthracite in the muffle increases the yield of metal, since a greater charge of ore may be introduced into a furnace of given capacity. By sifting out the finest portions, which are high in ash, from the anthracite, the value of the fuel is increased and the resistance to the circulation of the gases is diminished.—F. C. Th.

*Brass furnace; A rocking electric —.* H. W. Gillett and A. E. Rhoads. Met. and Chem. Eng., 1918, 18, 583—590.

A NEW type of arc furnace, developed as the result of investigation by the U.S. Bureau of Mines, consists of a drum, 5 ft. long and 5 ft. in diameter, lined to a depth of 12 in. and having a capacity of about 1300 lb. per 2 hrs. The drum is mounted to rotate about an axis slightly inclined to the horizontal, has a charging opening and pouring spout at the top, and is provided at each end with a water-cooled, graphite electrode, 4 in. in diameter, the latter being threaded for continuous feed and adjusted by hand. Single-phase, 60-cycle current, stepped down to 120—130 volts, is used, with an external reactance to stabilise the arc. The current varies between 1000 and 2000 amp. (averaging about 1650), the open circuit voltage being about 106—116 under load; and the power input is varied by altering the length of the arc. By means of an adjustable control device, the furnace is rocked automatically, a complete oscillation of a "safe rock" ( $80^\circ$ ) and a "full rock" ( $200^\circ$ ) occupying  $13\frac{1}{2}$  and  $33\frac{1}{2}$  seconds, respectively. The rocking angle is increased from  $80^\circ$ , when the charge begins to melt, to  $200^\circ$ , when melting is complete; at full rock the metal washes the whole circumference of the hearth except for the portions adjoining the charging door. In a continuous run extending over four days, during which over 34 short tons of metal was melted, the power consumption averaged 257 kilowatt-hours per ton charged. The cost of melting was found to be much less in the rocking electric furnace than in coke-fired furnaces. On the basis of a 10-hour day, the total cost by the former method was about one-half of that for crucibles alone by the latter (at present prices); and the amount of metal saved amounted to twice the cost of the coke consumed in the crucible process.—W. E. F. P.

*Aluminium; Metallography of —. Recrystallisation and grain growth.* R. J. Anderson. Faraday Soc., July 23, 1918. (Advance proof.) 5 pages.

THE first effect produced by annealing cold-worked aluminium is a small increase in hardness, further heating resulting in a softening. There may be a drop in the scleroscope hardness before microscopically visible internal changes occur. The temperature of most rapid grain growth resulting from the annealing of the cold-worked metal is a function of the deformation, being quite definite for a definite amount of cold work. Samples of aluminium hot rolled at  $455^\circ\text{C.}$  showed no grain growth when annealed up to  $425^\circ\text{C.}$ , and recrystallisation was incomplete. The scleroscope hardness was, however, only 5, i.e., mechanically full softening had occurred. At  $485^\circ\text{C.}$  full recrystallisation had taken place, accompanied by some grain growth. The time of the annealing is of importance since 10 mins. at  $595^\circ\text{C.}$  did not produce complete recrystallisation, although 60 mins. at  $540^\circ\text{C.}$  had done so. The more highly strained material is more readily recrystallised than less severely deformed metal, both as regards time and temperature. The results indicate that aluminium follows the same annealing laws as do other metals.

—F. C. Th.



*Aluminium sheet; Annealing and recrystallisation of cold-rolled* — R. J. Anderson. *Met. and Chem. Eng.*, 1918, 18, 523–527.

ANNEALING of cold-rolled aluminium sheet is usually carried out at a temperature of 375° C. for a period of 18–30 hours, and gives a maximum softness from the standpoint of mill operation corresponding to a hardness number of 4 to 5 as tested by the Shore scleroscope. If annealed for say 30 hours at 400° C., the scleroscope hardness will be about 4, but the sheet will be in a weakened condition and show a relatively large grain size, whereas if the period of annealing is only 10 mins., the grain size will be smaller, and the hardness still about the same figure. The occurrence of blistering during annealing is uncertain, but probably depends on the speed of pouring and the content of gases of the original cast metal. Long period annealings are harmful, giving a poor drawing quality and large grain size, and the common practice of exposing sheets for say 24 hours at 375° C. is unnecessary, as adequate softening can be effected by a short exposure at this temperature. (See also this J., 1918, 245 A.)—C. A. K.

*Aluminium; Influence of iron, tungsten, and nickel upon the resistance of — to acids.* Von Zeerleder. *Metall u. Erz*, 1917, 14, 325–330. *Z. angew. Chem.*, 1918, 31, Ref., 166.

THE addition of iron in all concentrations exerted a harmful influence. The resistance to sea water of a 10% iron alloy was only  $\frac{1}{4}$  that of pure aluminium. The addition of 1.5% W to the iron-aluminium alloy improves it slightly. The mechanical properties were also better, the brittle alloy being rendered tough and malleable. A partial substitution of nickel or cobalt for the iron renders the resistance to acids still worse.

—F. C. Th.

*Tin; Electrolytic refining of* — E. F. Kern. *Amer. Electrochem. Soc.*, May, 1918. [Advance copy.] 12 pages.

THE effect of the addition of aloin, gelatin, pectone, quinoline, oil of cloves, and boric acid to the electrolyte was to restrain the formation of non-adherent deposits. The best deposits were obtained from a solution containing 5 grms. of tin as stannous fluosilicate and 7.5 grms. of hydrofluosilicic acid per 100 c.c., with 1 gm. of aloin in 500 c.c. Aloin was a suitable addition agent only in the case of stannous fluosilicate electrolyte. Hydrofluoric acid to the extent of 60% of the total acid content is not detrimental. The best current density for the solution recommended was 24 amps. per sq. ft. of cathode, with a voltage of 0.4 volt.

—F. C. Th.

*Zinc [in alloys]; Gravimetric and volumetric determination of — as zinc mercury thiocyanate.* G. S. Jamieson. *J. Amer. Chem. Soc.*, 1918, 40, 1034–1039.

LUNDELL and Bee's method of estimating zinc in alloys as zinc mercury thiocyanate (*Trans. Amer. Inst. Met.*, 1914, 146) is criticised. The factor for obtaining the weight of zinc should be 0.13115, not 0.1266, since the precipitate has the composition  $\text{ZnHg}(\text{SCN})_2$ , after being dried at 102°–108° C. Excellent results are obtained by using ammonium thiocyanate instead of the potassium salt. Cadmium, cobalt, copper, bismuth, manganese, and mercurous compounds should be absent. Nickel in small amounts does not interfere appreciably. Ferric compounds in appreciable quantities should be reduced by sulphur dioxide. Arsenious compounds need not be removed. Instead of weighing the zinc mercury thiocyanate the following volu-

metric method, based on the equation:  $\text{ZnHg}(\text{SCN})_2 + 6\text{KIO}_3 + 12\text{HCl} = \text{ZnSO}_4 + \text{HgSO}_4 + 2\text{H}_2\text{SO}_4 + 4\text{HCN} + 6\text{KCl} + 6\text{KCl} + 2\text{H}_2\text{O}$ , gives trustworthy results. The zinc mercury thiocyanate corresponding with 0.03–0.10 gm. of zinc is precipitated and washed, the filter containing the precipitate is transferred to an 8-oz. titration bottle, a thoroughly cooled mixture of 25 c.c. of concentrated hydrochloric acid and 10 c.c. of water, followed by 7–8 c.c. of chloroform, is added, and a solution of potassium iodate (19.644 grms. in 1000 c.c. 1 c.c.=0.00100 gm. of zinc) is at once added rapidly while the bottle is well shaken. When the iodine liberated in the first stage of the reaction has disappeared, the titration is continued slowly, the closed bottle being thoroughly shaken after each addition until the iodine colour has disappeared from the chloroform indicator, which marks the end-point. If more than 50 c.c. of the iodate solution is required, a further 10–15 c.c. of concentrated hydrochloric acid must be added in order to prevent hydrolysis of the iodine monochloride. (See also *J. Chem. Soc.*, Sept., 1918.)—C. S.

*Alloys; Thermo-electromotive force of some* — M. A. Hunter and J. W. Bacon. *Amer. Electrochem. Soc.*, May, 1918. [Advance copy.] 14 pages. (See also this J., 1918, 32 A.)

OF the base-metal thermo-couples now on the market the most widely used are iron-constantan and nickel-aluminium-nickel-chromium. The former is satisfactory below 1000° C., the chief cause of its failure being the rapid oxidation of the iron. The E.M.F. of the couple is high and varies almost as a straight-line function of the temperature. It also maintains its calibration well. The latter couple may possibly be used up to 1400° C., but the E.M.F.-temperature curve is not so good nor is the original calibration so well maintained. Copper-nickel-chromium alloys are suggested for thermo-couple elements. The E.M.F.-temperature curves are approximately straight lines; the materials have high melting points and resist oxidation well, and large variations in composition have little effect on the E.M.F. The best combination among those tried was Cu 50: Ni 50: Cr 20, or Cu 50: Ni 50: Cr 10, combined with Ni 100: Cr 10.—F. C. Th.

*[Metals;] New thermo-electric effect [in —].* C. Benedicks. *Rev. Mét.*, 1918, 15, 320–332.

IN a homogeneous metallic circuit an asymmetrical temperature distribution produces an electromotive force (this J., 1917, 36). If, for instance, two fine platinum wires are laid upon each other in the form of a cross, and an end of each connected to a galvanometer, deflections corresponding to a millivolt are obtainable by slightly heating one arm. If the area of contact of the wires is increased by pressure, so that the temperature difference between the two arms is lowered by conduction, the E.M.F. is also lowered. The effect with a ferro-silicon containing 50% Si is much greater than with platinum. With a tungsten wire of 0.22 mm. diameter an E.M.F. of 19 millivolts was obtained when one arm of the cross was heated to 900° C. Surface oxidation, especially in the cases of tantalum and iron, results in perturbations. Errors in thermo-couples etc. arising from this cause may be eliminated by producing by pressure etc. efficient contact between the wires.—F. C. Th.

*[Ore] concentrates; Approximate determination of the minerals in —.* K. Thomas and F. W. Apgar. *Met. and Chem. Eng.*, 1918, 18, 514.

MICROSCOPIC examination of the concentrates and tailings from mill practice gives a simpler and more rapid indication of the quantitative mineral

composition than a complete chemical analysis. A sample is spread out evenly on a microscopic slide and the number of grains of each or of similar constituents in the field of vision is counted, at the same time noting the average size of grain for each. A percentage volume analysis is then readily obtained if the grains are of even size, otherwise a simple correction to allow for the difference in grain size is applied. The percentage by weight is obtained by multiplying by the specific gravity. The important minerals are easily distinguished under the microscope by colour and surface appearance, and if a large number of the grains are complex the desirability of finer grinding is indicated.—C. A. K.

*Molybdenite; Identification of* —. F. C. Fuchs. Eng. and Min. J., 1918, 105, 991.

A LITTLE of the mineral suspected to contain molybdenite is added to molten caustic potash. Within 5 mins., if molybdenite be present, swelling takes place, then rapid solution, and the production of an intense yellowish-red colour. When cool, if a few drops of water be added to the fusion, followed by drops of hydrochloric acid, the colour changes, and spots of blue, green, yellow, and red appear. It is suggested that molybdenite ( $\text{MoS}_2$ ) yields one atom of S to the molten potash with the formation of a red double sulphide. The method can be utilised for the rapid solution of molybdenite for purposes of analysis.—C. A. K.

*Use of wood in chemical apparatus.* Schorger. See I.

*Chemistry of gold compounds at high temperatures and pressures.* Morris. See VII.

*Use of hydrofluoric acid in analysis.* Furman. See XXIII.

*Acidimetry of coloured solutions; an application of the pocket spectroscope.* Tingle. See XXIII.

*Influence of impure zinc oxide on the determination of manganese.* Muller. See XXIII.

#### PATENTS.

[Steel] alloy. J. Churchward, Lakeville, Conn. U.S. Pats. (A) 1,261,742 and (B) 1,261,743, Apr. 2, 1917. (Appl. No. 15,116 of 1917.)

A STEEL alloy containing Ni about 1–5%, Mo about 0.25–3%, Mn under 0.70%, and (A) V about 0.15–0.50%, or (B) Ti from a trace to about 0.50%.

—T. H. B.

*Annealing high speed steel; Process of* —. F. Bagliardi, Milan. Eng. Pat. 116,829, Oct. 18, 1917. (Appl. No. 15,116 of 1917.)

HIGH speed steel is heated to incipient redness ( $500^{\circ}$ – $550^{\circ}$  C.), placed under a heap of cinders until it assumes a blue-black colour, and then immersed in water or greasy substances, preferably a paste consisting of 20% of soft animal grease which does not contain stearine, 70% of tallow, and 10% of wood charcoal.—C. A. K.

*Rust-preventing process.* D. Reichenstein, Zürich. Eng. Pat. 103,474, Nov. 14, 1916. (Appl. No. 16,309 of 1916.) Under Int. Conv., Feb. 3, 1916.

IRON surfaces (e.g., of steam turbines and boilers) exposed to the action of steam and air are protected from rusting by the addition of ammonia or other volatile base to the feed water or to the mixture of steam and air in the apparatus. Alternatively the volatile base may be formed in the boiler by reaction between a salt of the base and a stronger base.—C. A. K.

*Cast iron; Method of producing* —. F. A. Hirz, Struthütten. Ger. Pat. 303,657, Sept. 5, 1916.

IRON and steel scrap are melted together with a slag in a cupola. Certain impurities, such as oxygen compounds and sulphur, pass from the iron into the slag, whilst manganese, silicon, etc., pass from the slag into the iron. By regulating the character of the slag the purification may be carried to any desired extent.—F. C. Th.

*Iron ores, furnace dust, etc.; Process for briquetting* —. W. Mathesius, Nicolassée. Ger. Pat. 304,820, Oct. 27, 1915. Addition to Ger. Pat. 300,461.

THE components required for the preparation of the highly basic binding agent, rich in iron oxide, described in the chief patent, are obtained from the ore to be briquetted. For example, in the case of ores containing insufficient of the basic calcium compounds, part of the iron oxide is removed by magnetic separation and the residue is calcined. The friable ore may, as an alternative, be separated into a number of portions of different chemical composition, one or more of which may be used as binding material. The ore may be broken up by first calcining it and then quenching it, after which magnetic separation may be effected.

—F. C. Th.

*Ferro-phosphorus; Process of making* —. *Process of producing iron phosphide from iron phosphatic material.* H. A. Webster, Columbia, Tenn., Assignor to J. J. Gray, jun., Rockdale, Tenn. U.S. Pats. (A) 1,264,236 and (B) 1,264,237, Apr. 30, 1918. Date of appl., Oct. 13, 1916.

(A) A CHARGE consisting essentially of a fluxing material, carbon, and iron phosphate is heated in a closed furnace to a temperature sufficient to cause the oxygen in the phosphate to combine with the carbon, while maintaining the partial pressures of the reaction products above the normal. Ferro-phosphorus containing more than 22% of phosphorus is obtained. (B) A mixture of phosphatic material, a fluxing agent, and carbon in excess is heated in a rotary furnace. Iron-bearing material may be added to combine with elemental phosphorus to prevent its loss in the exit gases.—C. A. K.

*Ferro-silicon; Process for the manufacture of* —. T. A. F. Holmgren, Stockholm, J. O. Aqvist, and G. Helsing, Trollhättan, Sweden. Ger. Pat. 303,480, Oct. 26, 1916. Under Int. Conv., Oct. 29, 1915.

AT a very high temperature in the electric furnace carbon will reduce quartz and clay. The quartz is the more easily reduced, a very high temperature and prolonged period being required for the preparation of aluminium. By working under conditions such that the quartz is practically completely reduced while the alumina remains untouched, and in the presence of iron, ferro-silicon is produced. The iron may be produced simultaneously by the reduction of iron pyrites.

—F. C. Th.



*Crucible and like furnaces; Means for preheating the charge in* — J. Gaunt, D. Brookfield, and J. Tylor and Sons, Ltd., London. Eng. Pat. 115,222, Apr. 30, 1917. (Appl. No. 5095 of 1918.)

THE cover of a crucible furnace of the type described in Eng. Pat. 114,684 (this J., 1918, 308 A) carries a hopper lined with refractory material, and this lining extends downwards to form part of the lining of the cover and is preferably curved so that the gases arising from the furnace chamber will be deflected and caused to come into intimate contact with the charge in the hopper. The cover is mounted in such a way that it can be raised and swung clear of the body of the furnace.

*Copper alloys; Process of making* — H. Eldridge, New York. Eng. Pat. 115,917, June 4, 1917. (Appl. No. 8000 of 1917.)

SEE U.S. Pat. 1,246,571 of 1917; this J., 1918, 62 A. The copper is heated to a temperature below its melting point and is subjected, in the solid condition, to the vapour of the alloying metal, the more fusible alloy melting and running off, thus exposing new surfaces of the copper.

*Alloy.* C. Vickers, Niagara Falls, N.Y., Assignor to The Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,264,459, Apr. 30, 1918. Date of appl., Sep. 27, 1916.

IN an alloy of copper, aluminium, and iron, the aluminium constitutes not less than 7% nor more than 12%, and the iron not less than 3% nor more than 5% (preferably 3–4%) of the whole, the proportion of the aluminium to the iron being not more than  $2\frac{1}{2}$  to 1.—T. H. B.

*Metals such as tin and zinc; Method and apparatus for oxidising or volatilising* — H. Maconochie, London, and D. de Ros, Greenhithe. Eng. Pat. 115,870, Apr. 23, 1917. (Appl. No. 5694 of 1917.)

THE flame from a burner supplied with liquid or gaseous fuel is caused to heat the bottom of a hearth containing a charge of metal, and is then directed down on to the charge and heated air is caused to mingle with the fume given off from the metal, in a chamber connected to the hearth chamber. There are four horizontal, superposed and interconnected chambers, the lowermost being the primary combustion chamber, the chamber next above serving to heat the bottom of the third chamber, which is provided with a hearth for the metal and a deflector for directing the flame on to the charge, whilst the fourth chamber serves for conducting away and over the hearth the gases, products of combustion, fume, etc. The third chamber is so constructed as to allow of the removal and replacement of the hearth. The fourth chamber is provided with air-heating conduits having openings for the admission of some of the heated air to the chamber to oxidise the fume from the hearth. —T. H. B.

*Tin and lead fumes; Method for removing — from smelter gases.* H. Hocking and E. Apor, Budapest. Ger. Pat. 303,764, July 28, 1914.

THE gas is drawn or forced through a washing chamber divided by partitions into a series of smaller spaces. The high velocity of the gas produces in the liquid a copious foam which retains the particles of dust.—F. C. Th.

*Nickel-copper matte; Process for refining* — G. T. Holloway, London. Eng. Pat. 116,139, May 30, 1917. (Appl. No. 7742 of 1917.)

UNROASTED nickel-copper matte is alloyed with blister or crude copper, preferably containing nickel,

so as to obtain a product of sufficiently low sulphur content, and is then cast into anodes for electrolytic refining. Metals of the platinum group are said to be more readily recovered when an excess of copper over nickel is present.—C. A. K.

*Nitre cake; Utilisation of* — [for treatment of copper ores]. W. H. Hyatt, Long Ditton, Surrey, and E. N. Fellowes, London. Eng. Pat. 116,181, July 6, 1917. (Appl. No. 9803 of 1917.)

A SUITABLE copper ore, e.g. a carbonate ore containing about 5% of copper carbonate, is added to about its own bulk of a solution of nitre cake containing three parts by weight of water to one of nitre cake. The mixture is heated at about 100°C. until all the copper is dissolved. The copper is subsequently recovered from the solution by the usual methods, and the sodium salts by evaporation and recrystallisation, or the solution may be neutralised with a suitable carbonate, e.g., chalk, sodium carbonate, or, preferably, ammonium carbonate, and used as an insecticide.—L. A. C.

*Ore separating machines.* Fraser and Chalmers, Ltd., London, and W. Bullock, Bexley, Kent. Eng. Pat. 116,228, Dec. 29, 1917. (Appl. No. 19,235 of 1917.)

THE material to be separated falls from a hopper down a vertical or inclined air space of annular form separating a wheel, possessing magnetic permeability, from the pole-faces of an electromagnet. The non-magnetic particles fall freely through the air space, while the magnetic portion is carried on the wheel beyond the field produced by the polar surfaces. The wheel is preferably formed as the frustum of a cone revolving between similarly inclined pole-faces, whereby the air space may be suitably adjusted by raising or lowering the wheel, the periphery of which may be serrated.—C. A. K.

[*Ore*] *roasting furnaces.* F. Rowley and A. M. M. Merton, London. Eng. Pat. 116,235, Jan. 10, 1918. (Appl. No. 588 of 1918.)

IN a multiple-shaft roasting furnace having a number of superimposed hearths, the ore is spirally rabbled by means of rotating shafts arranged in pairs, the rabblers attached to the first shaft moving the material spirally outwards and those on the second shaft spirally inwards before the ore is dropped on to a lower hearth on which a similar motion is produced. A separate charging hole is provided for each pair of shafts and the various pairs are spaced so far apart that the material in its progress through the furnace is rabbled by only one pair of shafts and to all intent is roasted in a separate two-shaft furnace. The roaster gases however travel over the hearths of a series of the two-shaft furnaces, thus making the apparatus a multiple furnace in regard to the heating. —C. A. K.

*Roasting furnace.* A. Zavelberg, Hohenlohehütte. Ger. Pat. 303,543, July 8, 1916.

THE volume of the space over the roasting hearth is reduced by building a second arch of brickwork below and independent of the original arch of the furnace. The air supplied is brought into closer contact with the hearth.—H. J. H.

*Zinc; Process for the production of* — O. Baltin, Lipine, Germany. U.S. Pat. 1,261,342, Apr. 2, 1918. Date of appl., Feb. 25, 1914.

NAPHTHALENE is added to a mixture of the zinc ore and reducing agent, and the whole briquetted. —T. H. B.

*Zinc; Method of recovering — from waste solutions containing zinc and sodium sulphates.* O. Blumpage, Duisburg. Ger. Pat. 304,937, Jan. 14, 1917.

A PORTION of the solution which has been treated with quicklime, and then separated from the insoluble calcium sulphate, is used as a precipitant. The caustic soda is converted into sodium sulphate and hydrated zinc oxide precipitated. The addition of quicklime to this sodium sulphate solution regenerates caustic soda which may be used again.—F. C. Th.

*Tungsten or molybdenum articles; Process of coating — with precious metals.* F. A. Fahrenwald, Cleveland, Ohio, dedicated to the Government and people of the United States. U.S. Pat. 1,261,110, Apr. 2, 1918. Date of appl., May 31, 1916. (Dedicated to the public.)

ARTICLES of ductile tungsten or molybdenum are immersed in a bath of a fused oxygen salt of an alkali metal and then in a bath of molten gold at not more than 1350° C. for half a minute. The articles thus coated with gold are then ready for soldering or brazing. A coating of silver, platinum, or palladium instead of gold, or of an alloy of any of these metals, may be used.—J. H. J.

*Tungsten; Method of preparing alloys of — with precious metals.* G. Fuchs, Berlin. Ger. Pat. 301,551, Feb. 29, 1916.

ONE at least of the metals to be alloyed is first prepared as a basis alloy with nickel, iron, or aluminum. A gold alloy containing 75 parts of gold to 10–15 of tungsten and 10–15 parts of nickel, may be used in jewellery as a substitute for platinum. It can be cast, hammered, and rolled and takes a much finer polish than platinum does. An alloy of 80 parts of fine silver, 10–15 of nickel, and 10–15 parts of tungsten possesses a higher specific gravity and greater strength than the gold alloy. It is much less affected by atmospheric oxidation than silver and takes a better polish. These alloys may also find technical application on account of their resistance to acid attack.—F. C. Th.

*Metals [tungsten]; Extraction of — from their ores.* E. M. Hamilton, Assignor to Hamilton, Beauchamp, Woodworth, Inc., San Francisco, Cal. U.S. Pat. 1,261,383, Apr. 2, 1918. Date of appl., Mar. 14, 1917.

ONE or concentrate containing tungsten is reduced to a suitable condition of fineness, and mixed with water and an alkali carbonate or bicarbonate to form a pulp, the ratio of solution to ore being approximately between 1:1 and 2:1. The pulp is heated under pressure, then filtered, and tungstic acid is separated from the solution by addition of an acid. An excess of alkali carbonate or bicarbonate is used, but not sufficient to cause appreciable solution of silica.—T. H. B.

*[Ore] concentration process.* W. A. Scott, Chicago, Ill. U.S. Pat. 1,261,303, Apr. 2, 1918. Date of appl., Aug. 27, 1917.

A MIXTURE of an inert gas, such as air, with a permanent bubble-modifying gas, *e.g.*, methane, coal gas, acetylene, etc., is introduced into a body of ore pulp, preferably through a porous medium. A column of bubbles is formed above the normal level of the ore pulp, and the effect of the bubble-modifying gas is to cause certain of the mineral constituents to adhere to the bubbles. Good results have been obtained in the concentration of the zinc ore of Butte, Montana, by using acetylene as bubble-modifying gas.

*Sulphide ores; Method of treating —.* F. K. Cameron and J. A. Cullen, Salt Lake City, Utah, and R. W. Hyde, New York, Assignors to American Smelting and Refining Co., Salt Lake City. U.S. Pat. 1,261,694, Apr. 2, 1918. Date of appl., June 18, 1917.

SULPHIDE ore is added to boiling sulphuric acid of at least 50° B. (sp. gr. 1.53), elemental sulphur being distilled off and metal values recovered from the residue.—W. E. F. P.

*Silicious ores; Method of treating —.* G. H. Wigton, Eureka, Utah. U.S. Pat. 1,264,586, Apr. 30, 1918. Date of appl., Sept. 22, 1916.

FINELY-GROUND silicious ores with a base metal content insufficient to form a slag at the volatilising temperature of the metallic compound, are mixed with an excess of alkali or alkaline-earth chloride, and the metallic chloride separated by distillation. The metallic chloride is subsequently reduced to the metallic state, the chlorine being recovered in the slag which may be used in the treatment of a fresh charge of ore.—C. A. K.

*Metals from ores and other materials; Distilling process for recovering —.* Process for recovering mercury. C. H. Fulton, St. Louis, Mo., Assignor to Metallurgical Laboratories, Inc., Chicago, Ill. U.S. Pats. (A) 1,264,628 and (B) 1,264,629, Apr. 30, 1918. Date of appl., Apr. 21, 1917.

(A) ORES or other metal-bearing materials are formed into briquettes with or without reducing materials and suitable binders and are distilled in an electrical resistance furnace, the blocks themselves constituting the resistors. It is necessary that the briquettes should not appreciably alter their shape or volume during the distillation. (B) The process described under (A) is applied to mercury ores.—C. A. K.

*[Ore] reduction apparatus.* P. J. MacDonald, Los Angeles, Assignor to C. L. Claffin, jun., Bakersfield, Cal. U.S. Pat. 1,264,832, Apr. 30, 1918. Date of appl., Sep. 18, 1916.

MEANS are provided for delivering oxide ore into a reduction chamber and agitating the uppermost stratum of the ore with a reducing agent, the under portion being relatively at rest. Heat is applied and the waste gases are carried away through a separate outlet.—C. A. K.

*Metal scrap and ores; Apparatus for treatment of — with acids.* H. Stegmeyer, Charlottenburg. Ger. Pat. 303,475, Mar. 5, 1914.

THE acid liquor and the metal or ore are conveyed along one or more troughs to which a reciprocating motion is applied. The trough communicates with the liquor reservoir through a valve which is operated by the pulsations and so causes a circulation of acid. The latter can be arranged to flow in the same direction as the material or in counter current as desired.—H. J. H.

*Sintering fine and powdery material; Process for —.* W. Schumacher, Berlin. Ger. Pat. 303,799, Nov. 4, 1916.

FINE ore or flue-dust is mixed with fuel and subjected in a vertical furnace to a blast of strongly heated air, such as the hot blast of a blast-furnace.—F. C. Th.



*Briquetting fine ores; Process for* —. Brück, Kretschel, und Co. Osnabrück. Ger. Pat. 304,484, Feb. 27, 1915.

SLAKED lime and the dust from a blast furnace smelting hematite ores are added to the ore as a binding agent and the mixture is compressed, and treated with steam. Very good briquettes can be made from 84% of concentrates, 12% of hematite flue dust, and 4% of slaked lime.—F. C. Th.

*Aluminium; Process for the preparation of* — suitable for the decaridation of melts of iron and steel. Rombacher Hüttenwerke A.-G., I. Bronn, and W. Schemmann, Rombach. Ger. Pat. 304,645, July 6, 1917.

THE aluminium is melted in a crucible and granulated by pouring into flowing water. This carries it along into a collecting chamber from which it is removed and placed on the pan-shaped cover of the melting furnace and quickly dried by the waste heat of the furnace.—F. C. Th.

*Phosphoric slags; Process for the production of high-percentage* — of high solubility in citric acid in the basic open-hearth process. B. Queling, Saarbrücken. Ger. Pat. 304,894, Feb. 20, 1917. Addition to Ger. Pat. 301,839.

THE dephosphorising period is divided into one or more stages after each of which the slag is transferred to a special slag furnace. This eliminates the possibility of the reduction of phosphorus from the slag and its re-absorption by the metal during recarburisation.—F. C. Th.

*Phosphoric slags; Method for obtaining highly* — in the manufacture of ingot-iron or steel from sulphurous pig iron. B. Queling, Saarbrücken. Ger. Pat. 304,895, Feb. 7, 1917. Addition to Ger. Pat. 301,839.

TO the bath of metal in the mixer briquettes of burnt lime, manganese oxide, and carbon are added. The solubility of sulphur in pig iron is lessened by a high carbon content, while that of the slag is increased. The presence of the manganese oxide accelerates the desulphurisation. (See also Ger. Pats. 302,283 and 304,894; this J., 1918, 377 A, and preceding.)—F. C. Th.

*Tin-plates or sheets and other like metal coated plates or sheets; Appliances used in the manufacture of* —. D. Davies, Llanelly, and D. Jones, Glanamman. Eng. Pat. 116,250, May 2, 1917. (Appl. No. 6501 of 1918.)

*Wrought-iron; Manufacture of* —. J. Aston, Assignor to A. M. Byers Co., Pittsburgh, Pa. Reissue 14,457, Apr. 2, 1918, of U.S. Pat. 1,255,499, Feb. 5, 1918. Date of appl., Feb. 19, 1918.

SEE this J., 1918, 212 A.

*Electrode as used for welding purposes.* M. M. Irvine, Glasgow. U.S. Pat. 1,261,395, Apr. 2, 1918. Date of appl., Sept. 17, 1917.

SEE Eng. Pat. 108,789 of 1916; this J., 1917, 1100.

*Metal articles; Scaling and annealing of* —. F. Perry, Assignor to Metalloids, Ltd., Tipton. U.S. Pat. 1,261,606, Apr. 2, 1918. Date of appl., Feb. 15, 1917.

SEE Eng. Pat. 102,660 of 1916; this J., 1917, 143.

*Scotch hearth; Mechanically-operated* —. A. S. Moses, New York, Assignor to St. Louis Smelting and Refining Co., St. Louis, Mo. U.S. Pat. 1,263,695, Apr. 23, 1918. Date of appl., Apr. 3, 1916.

SEE Eng. Pat. 103,205 of 1916; this J., 1917, 294.

*Slags; Generation of steam from the heat contained in* —. C. Vautin, London. U.S. Pat. 1,264,229, Apr. 30, 1918. Date of appl., Oct. 24, 1914.

SEE Eng. Pats. 28,015 of 1913 and 2393 of 1914; this J., 1915, 89.

*Method and apparatus for segregating and recovering gases [sulphur dioxide]. Art of and apparatus for treatment of corrosive gases.* U.S. Pats. 1,265,892 and 1,265,893. See VII.

*Production of zinc chloride. Producing zinc chloride from ores.* U.S. Pats. 1,261,695 and 1,261,696. See VII.

*Manufacture of cement from blast-furnace slag.* Eng. Pat. 114,794. See IX.

## XI.—ELECTRO-CHEMISTRY.

*Electrical resistivity of porcelain and magnesia at high temperatures.* Brace. See VIII.

*Electric steel castings.* Flinterman. See X.

*The Vom Baur [electric] arc furnace.* Vom Baur. See X.

*The Booth-Hall electric [steel] furnace.* Booth. See X.

*Some chemically reactive alloys.* Ashcroft. See X.

### PATENTS.

*Furnaces; Electrically heated resistance* —. H. A. Greaves and H. Etchells, Sheffield. Eng. Pat. 115,866, Apr. 11, 1917. (Appl. No. 5072 of 1917.)

THE furnace is so constructed that leakage currents may be usefully employed to produce uniform heating, the crucible chamber to be heated being built of refractory material, which is a poor electrical conductor when hot, *e.g.*, silica ware. The ducts surrounding the chamber are filled with an electrical resistor of granular form, and the partitions which separate the ducts are made of refractory materials, which are fairly good second class electrical conductors when hot, *e.g.*, chromite, magnesite, silundum. The electrical current is supplied at the free end of each duct by separate electrode pole-pieces directly connected to the feeding conductors, the ducts being in the form of continuous loops which unite in a common chamber under the crucible chamber. Leakage currents pass from each heating unit through the partitions to the units on either side.—B. N.

*Electrical contacts; Method of making* —. G. A. Rignoni, Genoa, Italy. U.S. Pat. 1,264,685, Apr. 30, 1918. Date of appl., Sept. 30, 1916.

CONTACTS of tungsten, molybdenum, or a metal of the iron group, of an exceedingly high welding point, for use in contact breakers in magnetos employed for the ignition of explosive mixtures in internal combustion engines, are made by uniting the metal to its support by electro-thermal welding at a very high temperature, then coating the support and its contact with good conducting metal, such as copper or silver, with the exception of the two faces between which the break takes place, in order to reduce the resistance.—B. N.

*Carbon electrodes; Manufacture of* —. B. E. D. Kilburn, London. From Det Norske Akieselskab for Elektrokemisk Industri Norsk Industri-Hypotekbank, Christiania. Eng. Pat. 116,853, Jan. 9, 1918. (Appl. No. 551 of 1918.)

ELECTRODES are manufactured in continuous lengths, in the furnace in which they are used, by feeding a suitable carbonaceous mixture into a mould leading to the furnace, the mixture being preferably rammed round a metallic core. In the case of a vertical electrode, the lower end is rapidly heated by the arc or melting crater, while the upper portion is baked partly by the heat of the furnace and partly by the heat generated by the current passing through the metallic core. As the electrode burns away fresh portions of the mixture are added in the mould and the electrode pushed down. The upper portion of the electrode may be heated more gradually by an independent circuit, the main current only traversing the harder baked section.

—C. A. K.

*Spraying electrode for the electrical purification of air or gases containing dust or fog.* Metallbank und Metallurgische Ges. A.-G., Frankfurt. Ger. Pat. 305,363, May 23, 1914.

AN electrode for discharging a fine spray of sparks in all directions consists of a vessel charged with water and made of porous material or of perforated metal provided with a mantle of porous material. The vessel or its mantle may, for instance, be made of earthenware or porcelain or other mineral substance, which is either used in the crude condition and maintained in the desired shape, or has been subjected to treatment which does not destroy its porosity. The liquid may be drawn into the electrode from a suitable vessel by the capillary action of the electrode itself or its mantle.

—C. A. M.

*Electric storage batteries; Manufacture of plates for* —. H. Wade, London. From L. H. Flanders, Jenkintown, Pa., U.S.A. Eng. Pat. 115,865, Apr. 4, 1917. (Appl. No. 4885 of 1917.)

SEE U.S. Pat. 1,228,551 of 1917; this J., 1917, 892.

*Carbon electrodes; Ovens or furnaces for use in baking* — and for other purposes. R. K. Wehner, Newark, N.J., U.S.A. Eng. Pat. 116,455, Nov. 7, 1917. (Appl. No. 16,263 of 1917.)

SEE U.S. Pat. 1,251,309 of 1917; this J., 1918, 114 A.

*Apparatus for the electrical separation of suspended particles from gases.* Ger. Pat. 303,078. See I.

[Electro-osmotic] process of removing liquid [water] from stearite. U.S. Pat. 1,266,330. See VII.

*Electrolysis of chlorides [and production of chloro-hydrocarbons]. Manufacture of alkalis [and chloro-hydrocarbons].* U.S. Pat. 1,264,535 and 1,264,536. See VII.

*Apparatus for sterilising water by means of ozone.* U.S. Pat. 1,264,360. See XIXb.

*Sterilisation of water and removal of iron and manganese therefrom by electrolytic means.* Ger. Pat. 301,585, 302,226, and 302,227. See XIXb.

## XII.—FATS; OILS; WAXES.

*Fatty oils; Process for deodorising* —. W. P. Schuck. Met. and Chem. Eng., 1918, 18, 608—609.

DEODORISATION of any oil or fat is effected by blowing hydrogen or a gas containing hydrogen as its main constituent through the heated oil, in a vessel from which atmospheric oxygen is excluded. In the commercial application of the process the volatile fatty acids, etc., are condensed, and washed from the hydrogen, which is then dried and used again. No hardening of the oil takes place, since the treatment is effected in the absence of a catalyst. In comparative tests in which an oil was treated in this way with pure hydrogen and pure nitrogen, and the fumes condensed, it was found that the total iodine value of the condensed liquid and of the residual oil was lower when hydrogen than when nitrogen had been used. In the nitrogen experiments the oils had a burnt taste, and in some cases had only been partially deodorised, whilst they contained about three times as much free fatty acids as the oils which had been treated with hydrogen. When a current of hydrogen is made to bubble through a heated oil the free fatty acids begin to be removed at about 210° C., and in most cases are reduced to within 0.15% in 20 mins. at 275° C. Coconut oil containing over 14% of free fatty acids had the amount reduced to 0.12%, whilst fish oil with over 5.14% yielded a product containing only 0.14% of free fatty acids, the loss on purification being only 1.1% more than was attributable to the removal of the acids. The condensed fatty acids are available for the manufacture of soap. Oils thus deodorised keep sound very much longer than oils deodorised by steam-treatment. For example, coconut oil deodorised as described was pleasant to the taste after four months' exposure in a loosely covered tin, whilst the same oil deodorised by steam had become rancid in a few weeks under the same conditions. It is suggested that this process may be used for the purification of oils which have been burnt in cooking and of garbage fats, and that it will be of value for deodorising castor oil and removing the free fatty acids, without interfering with its medicinal properties. (See also U.S. Pat. 1,260,072 of 1918; this J., 1918, 312 A.)—C. A. M.

*Linseed oil; Study of the oxidation of* —. I. G. E. Holden and L. G. Radcliffe. J. Soc. Dyers and Col., 1918, 34, 138—146.

WEIGHED quantities (about 1 grm.) of linseed oil were placed on pieces of cotton cloth, about 3 in. square, previously freed from mineral matter and fat; these were introduced into a series of three or four connected gas wash-bottles and a slow current of air was drawn through the series of bottles. It was found that the linseed oil in the first bottle of a series dried more rapidly than that in the second, whilst the second was more rapid than the third, and so on; but that on continuing the current of air for periods ranging from 450 to 500 hours the units of each series showed equal increments in weight (17 to 18%). On heating the bottles containing the dried samples for definite periods at 100° C., this original gain was reduced to a gain of only 1.0 to 3.5%. In further experiments in which quantities of 0.1 to 0.125 grm. of linseed oil were spread on roughened glass plates and exposed to definite temperatures, it was found that the velocity of drying increased with the temperature, but that the higher the temperature the lower the maximum increase in weight, and the less the accelerating influence of any driers present on the drying of the oil. When dried at or just below 34° C. linseed oil gains from 17 to 18% in weight,



whereas when dried at 100° C., the maximum increase is much less (e.g., 7.8%), and at 150° C., no increase in weight is observed, probably owing to accelerated formation and evolution of volatile products. An equal drying capacity is shown by the linoleate or resinates of the same metal containing the same proportion of that metal, but manganese has six times the efficiency of lead as a drier. The rate of drying of linseed oil is slightly increased by the presence of turpentine. Linseed oils which dry most slowly will be found to contain a higher proportion of non-drying substances than do rapidly drying oils. Oils with low iodine values require a longer time to dry than those with higher iodine values. On treating dried linseed oil films with ether, the amount of organic matter removed from films dried at 100° C. is much less than from films dried at 15° C., and a higher proportion is extracted from films from oils having the lower iodine values. These results indicate that the saturated glycerides have an important influence on the rate of drying of the oils, and the ultimate decomposition of a film of linseed oil appears to be due to the decomposition and emanation of the non-drying substances in the film. The greater durability of a linseed oil film which has been dried at a high temperature must be attributed to the more rapid volatilisation of the non-drying substances by the heat prior to the formation of the linoxyn. Drying of an oil is accelerated by the action of ultra-violet rays, so much so that linseed oil by itself under these conditions dries as rapidly as an oil containing a drier in solution. Volatile products from linseed oil oxidised in a current of air at 100° C. gave the reactions of unsaturated aldehydes and contained drops of an oily substance consisting of traces of free fatty acids in admixture with saponifiable matter. The non-drying constituents of the linseed oil undoubtedly affect the degree of hardness of the oxidation product. They are decomposed under the influence of ultra-violet rays, as is shown by the fact that linseed oil attains only 12 to 13% increase in weight, as compared with 17 to 18% under normal conditions of light (see also this J., 1914, 266).—C. A. M.

*Oil in seeds; Determination of* — W. B. Gray. J. S. African Assoc. Anal. Chemists, 1918, 1, 5-7.

For extraction of the oil from oil seeds Kreussler's apparatus is more satisfactory for use with ether than Soxhlet's apparatus, only a few c.c. of the solvent in excess of that needed to saturate the sample being required. Small seeds can be completely extracted in about 2½ hours, whilst palm kernels, copra, etc., require 8 hours. In the case of coarsely ground material the sample should first be extracted for 4 hours, then ground in a mortar, and the extraction continued for a further 4 hours. Thorough drying of the seeds before extraction is essential to prevent extraction of gum resins from the husks. In the case of drying oils the residue left on evaporating the extract should be dried in a current of dry hydrogen, coal gas, or carbon dioxide.—C. A. M.

*Oilicica oil—a new drying oil.* E. R. Bolton and C. Revis. Analyst, 1918, 43, 251-254.

The kernels of seeds known in Brazil as *oilicica* or *oilizika*, and probably derived from *Conepia grandifolia* (Rosaceæ) yielded 62% of an oil which was semi-solid at the ordinary temperature, but at higher temperatures consisted of a solid fat in a liquid medium. The oil had the following characters:—M. pt., incipient fusion, 21.5° C.; complete fusion, 65.1° C.; sp. gr. at 15.5°/15.5° C., 0.9694; free fatty acids as oleic, 5.7%; unsaponif. matter,

0.91%; saponif. value, 188.6; and iodine value, 179.5; m. pt. of fatty acids, 53.7°; solidif. pt. of fatty acids, 67.0° C. When brominated the oil yielded only traces of insoluble bromides, but, on standing for 2 to 3 days, the solution became deep brown and a pale liquid separated. When heated to 250° to 270° C. in an atmosphere of carbon dioxide no solidification took place, whereas tung oil became solid under the same conditions, but at 300° C. polymerisation occurred, with the formation of a stiff, clear, transparent jelly, which when mixed with an oil, such as linseed oil, or a solvent, such as chloroform, swelled up and became gelatinous. The jelly formed by tung oil on polymerisation is turbid. When heated in a thin film at 100° C. the new oil gained in weight 4% (tung oil 2 to 3%), and the oxidised skin was practically insoluble in petroleum spirit, chloroform, or acetone. A solution of the oil in commercial benzene when allowed to dry on glass formed a more uniform and transparent skin than did tung oil. The lead soap was more granular than that of tung oil and was soluble in small quantities in linseed oil. The oil will probably be suitable for the manufacture of linoleum as well as for varnish.

—C. A. M.

*Sanga-sanga (Ricinodendron africanum); Chemical study of the nuts of* — Pieraerts. Ann. du Musée colon. de Marseille, 1917, 5, 27-37. Bull. Agric. Intell., 1918, 9, 456.

THE nuts of *Ricinodendron africanum*, a large tree growing in the French Congo and other countries of West Equatorial Africa, are of about the size of a hazel nut. Unsorted nuts yielded 28% of kernels, which contained 17.64% of water and 55.29% of a pale yellow oil with the following characters:—Sp. gr. 15°/15° C., 0.9345; ref. index at 19.5° C., 1.5028; acid value, 0.86; saponif. value, 194.4; Hehner value, 98.85; and m.pt. 32.3° to 34.5° C. The oil contained 9.77% of glycerol, and yielded no bromides insoluble in ether. When shaken with a saturated solution of iodine in chloroform it set to a gelatinous mass. "Sanga-sanga" oil dries more rapidly than linseed oil, and could be used for waterproofing, for caulking ships' hulls, and as mortar or lamp oil. The oil-cake is rich in nitrogen and phosphates, and if proved to be free from toxic substances would be an excellent feeding-stuff.

—C. A. M.

*Fats from Rhus laurina and R. diversiloba.* J. B. McNair. Bot. Gaz., 1917, 64, 330-336.

Fats isolated from *Rhus laurina* and *R. diversiloba* had the following constants respectively:—Sp. gr. (18.5° C.) 0.8987, 0.9872; solubility 136, 170 mgrms. per litre in 95% alcohol at 20° C.; iodine value (Hübl) 11.44, 8.79; saponif. value, 157.1, 220.6; m.pt., 74°, 53° C. The substances appear to be more similar to Japan wax than to any other fat. A decrease in the poisonous properties of the fruit of *R. diversiloba* occurs simultaneously with an increase in the fat content, but this does not appear to be due to the conversion of the poison into fat.

*Grease from waste materials; Recovery of* — by the solvent extraction process. J. H. Garner and J. F. Carmichael. J. Soc. Dyers and Col., 1918, 34, 113-122.

THE recovery of grease and fatty acids from the effluents of wool scouring is capable of very profitable extension. After precipitation by nitre cake the magma is pressed in one of two ways: either in the so-called oven press after being wrapped in sacking by hand, or in a steam-heated filter-press into which the previously heated magma is forced by steam pressure or compressed air. The cake



from the former retains 12–15% of moisture and that from the latter up to 35%, and the dry substance from both still contains 20–30% of grease which should be recovered by extraction with solvents. Moreover the dry sewage sludge of industrial towns contains 15–40% of grease, the lower value representing purely domestic sewage and the lowest limit of profitable recovery treatment. Sludges containing over 30% of fat on the dry substance are best treated in two stages, *viz.*: by hot pressing for the direct recovery of grease, and subsequent extraction of the cakes by solvents, whereas those containing less than 30% should be pressed for the removal of water only and treated directly with solvents. In either case, the sludge must not be precipitated by lime but only by acids. For profitable treatment with solvents, the moisture in the cake must be reduced to 15–20%, preferably in mechanical drying apparatus heated by means of waste heat. The Ruggles-Coles dryer consists of two inclined concentric cylinders arranged so that fine gases pass downwards through the central cylinder and back up through the annular space between the two, while the disintegrated cake passes down the annular space. The Dublin dryer consists of a series of shelves around a vertical shaft, every alternate shelf being attached to the shaft; a series of rakes and scrapers on the lower sides of the shelves pushes the material from one shelf to the other against an ascending current of hot air. The B.T.W. dryer consists of two inclined rotary steel cylinders placed one above the other in a brick flue; the material passes down the upper one in which it is broken up and rubbed by means of scrapers and teeth and passes into the lower one in which it is tossed about by lifting vanes and dried. The solvent extraction plant consists of extraction vessel, still, and condenser. The material is charged into the extraction vessel on a canvas filter bed supported by perforated plates. The depth of material charged depends on its physical condition and its freedom from a tendency to sink together and clog. An open material with 45–55% of mineral matter may be packed to a depth of 8–9 ft., whereas one containing only 25% of mineral matter and a large amount of organic matter may only be worked with a depth of 4–5 ft. Solvent (petroleum spirit) is admitted to the space below the grid and gently boiled so that the vapour passes upwards and saturates the material with hot solvent. A continuous percolation of dissolved grease thus takes place into the space below the grid and the process is continued for 6–10 hours until the material is exhausted. The grease solution is then blown into the still and the solvent left in the material is recovered by steaming. A copper tubular counter-current condenser gives the best results; failing that, a multitubular cast-iron condenser; the ordinary steel coil condenser does not last, owing to the corrosive action of the volatile fatty acids. The grease is refined by boiling with dilute sulphuric acid, stirring for 20 mins., and allowing to settle for 24–48 hours. The loss of solvent amounts to 3–4.5 galls. per ton of material treated. The exhausted material contains 4–5% of fat and about 10% of moisture and is in a suitable condition for use as a fertiliser. The cost of the process depends very largely on the richness of the material worked; figures are given based on 15% of recovered grease, showing a cost of £9 15s. per ton of grease.—J. F. B.

*Sulphonated oils; Analysis of* —. G. W. Schultz. J. Amer. Leather Chem. Assoc., 1918, 13, 190–192.

THE method of R. Hart (this J., 1917, 1139) has been modified in some particulars, and results obtained by it compared with those given by the

method of the American Leather Chemists' Association Committee. The acid value of the fatty acids of the oil before sulphonation cannot be used in calculating total fat, as the sulphonation lowers the acid value of the acids. Unsaponifiable matter is determined after the saponification value. The solution is made alkaline and diluted so as to contain about 50% of alcohol, boiled for a few minutes, and subsequently extracted with petroleum spirit. The extracted soap solution from the determination of unsaponifiable matter is carefully boiled to dryness, the residue dissolved in water, and the solution boiled to expel alcohol. The fatty acids are then precipitated and extracted by means of ether, the ether solution washed with concentrated sodium sulphate solution, and the ether distilled off. The saponification value of the fatty acids is then determined (not the acid value, on account of lactones and anhydrides). If the oil contains ammonia, the free fatty acids cannot be determined as usual. The following method is given: 10 grms. of oil is dissolved in 95% alcohol and titrated with sodium hydroxide in presence of phenolphthalein; the solution is boiled for  $\frac{1}{2}$  hr. to expel ammonia, cooled, again neutralised with standard alkali, and the process repeated until no more ammonia is expelled by boiling. The solution is then diluted with water and titrated with sulphuric acid, using methyl orange as indicator, to determine alkali present as soap. The following calculation gives the neutral fat: subtract the ammonia content (expressed as mgrms. of potash per gram. of oil) from the saponification value of the sample, and further deduct the free fatty acids and acids present as soap (again expressed in mgrm. potash), and divide the result by the saponification value of the fatty acids. The result multiplied by 100 is the percentage of neutral fat. Analyses show that the neutral fat determined as above is much higher than the amount found by the Committee method; nevertheless the author considers the figures reliable.—F. C. T.

*Water-in-oil emulsions.* Schlaepfer. See IIA.

*Chemical composition of artificial asphalts.* Marcussen. See IIA.

#### PATENTS.

*Catalysers for reduction or hydrogenation purposes; Method of producing — and apparatus therefor.* Soc. de Stéarinerie et Savonnerie de Lyon, and P. Berthon, Lyons, France. Eng. Pat. 107,004, May 19, 1917. (Appl. No. 7259 of 1917.) Under Int. Conv., May 19, 1916.

CATALYSTS such as finely divided reduced nickel, cobalt, manganese, copper, etc., for hydrogenation processes or for the reduction of organic substances and fatty bodies, are prepared by reducing the metallic oxides at about 250° C. in a neutral liquid medium. The medium may be a hydrocarbon obtained from solid paraffin, vaseline, heavy petroleum oil, etc., which is not susceptible of hydrolysis or saponification, with the addition of 0.25 to 0.5% of stearic acid. The material is placed in vertical tubes mounted in a cylindrical casing, and superheated steam is circulated round the tubes to maintain them at about 250° C. The metallic oxide is then added and a strong current of hydrogen injected into the space below the lower tube-plate through a perforated diaphragm to distribute the gas into the tubes. Finely perforated plates are also arranged in the tubes. Very finely divided metal is obtained after about 2.5–3 hours and the liquid medium is separated by centrifuging. The catalyst is obtained in the form of cakes containing a high proportion of metal.—W. F. F.



*Oil or the like; Presses for expressing* —. A. W. French, Piqua, Ohio. U.S.A. Eng. Pat. 107,200, June 11, 1917. (Appl. No. 8347 of 1917.) Under Int. Conv., June 10, 1916.

THE press-plates are composed of a supporting plate and a mat plate provided with the necessary perforations and channels. The under sides of the plates have dependent projecting bars, whilst there are upward projecting guides on the edges of the mat plates, so arranged as to "telescope" into the dependent bars of the plate immediately above, when the plates are brought together. The upward bars also serve as guides in inserting the bag containing the meal.—C. A. M.

*Animal and vegetable oils, fats, and greases; Treatment of* —. C. F. Paul, jun., Newark, N.J., Assignor to The Spalding By-Products Co., New Haven, Conn. U.S. Pat. 1,264,206, Apr. 30, 1918. Date of appl., May 17, 1916. Renewed Dec. 13, 1917.

GREASE is forced, together with an aqueous solution of an alkali peroxide, through a narrow orifice, or is otherwise emulsified with the alkali, while a jet of air under pressure is directed upon the material as it leaves the orifice, or air is blown into the mixture to effect fine subdivision. The mixture is then treated with sulphuric or other acid to form the sulphate or other water-soluble salt of the alkali, and the grease is separated from the aqueous solution of such salt.—C. A. M.

*Surgical dressing [from beeswax]. Product of beeswax and process of manufacturing the same.* K. J. Holliday, Glen Osborne Borough, Pa., Assignor to Holliday Chemical Co., Pittsburgh, Pa. U.S. Pats. (A) 1,264,185 and (B) 1,264,186, Apr. 30, 1918. Dates of appl., (A) Sept. 25, and (B) Sept. 26, 1917.

(A) THE surgical dressing consists of a composition of paraffin and the esters and hydrocarbons of beeswax, and a gum of the "elimi" group, in the proportions 95 to 65% of paraffin, 5 to 35% of beeswax, and 0.1 to 5% of gum, having a definite melting point from 120° to 140° F. (59° to 60° C.). The material is substantially free from all free acids, and is capable when melted of forming an elastic, non-cracking, and non-irritating covering. (B) A neutral product free from acids and containing the neutral esters and hydrocarbons of beeswax, is prepared by mixing melted beeswax with an alkaline solution to form an emulsion, and separating the soaps and waxes from the emulsion whilst in a heated state. The soaps are then eliminated from the product, which is non-irritating to the flesh, and non-corrosive when in contact with metals.—B. N.

*Dispersoids [emulsions, etc.]; Process for stabilising* —. L. Sarason, Charlottenburg. Ger. Pat. 302,093, Apr. 29, 1915.

DISPERSOIDS which do not form hydrosols may be rendered stable by the use of nucleic acid solution (e.g., yeast nucleic acid) as a protective colloid. Examples are given of the formation of emulsions of linseed oil with glycerin and pressed yeast, and of a powder colour with pressed yeast, warmed to liquefaction. It is preferable to add a preservative such as formaldehyde.—B. V. S.

*Emulsions of organic chlorides containing more than one carbon atom; Process of preparing* —. Chem. Fabrik Stockhausen und Co. Ger. Pat. 304,909, Oct. 9, 1907.

CHLORINE compounds containing more than one carbon atom, such as tetrachloroethane, trichloro-

ethylene, etc., are emulsified by means of compounds containing a sulpho group, such as sulphonated oils and fats, sulphonated fatty acids, especially Turkey-red oil, or the gelatinous soaps prepared from sulphonated castor or other oils (Ger. Pat. 113,433; see Eng. Pats. 2896 of 1896 and 24,868 of 1897; this J., 1896, 363; 1898, 162).

—C. A. M.

*Coconut oil; Process of extracting* —. G. G. Turner, New York, and C. H. Leighton, Brooklyn, N.Y., U.S.A. Eng. Pat. 115,877, May 18, 1917. (Appl. No. 7210 of 1917.)

SEE U.S. Pat. 1,247,597 of 1917; this J., 1918, 34 A.

*Oils, fats, and greases; Apparatus for extraction of — and of gelatin.* H. Engel, London. U.S. Pat. 1,264,288, Apr. 30, 1918. Date of appl., July 3, 1917.

SEE Eng. Pat. 108,014 of 1916; this J., 1917, 1018.

*Preparation of catalytic material employed for the promotion of synthetic chemical reactions.* Eng. Pat. 115,924. See I.

*Process for increasing the decolorising power of bleaching earth [fullers' earth].* Ger. Pat. 305,896. See I.

*Process for removing fat from sewage sludge.* Ger. Pat. 305,768. See XIXb.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Conifer resin.* C. G. Schwalbe and W. Schulz. Z. angew. Chem., 1918, 31, 125—127.

SEASONING renders the resin in pine wood insoluble to a great extent, and the longer a wood is kept, particularly in the form of sawdust, the smaller is the quantity of resin which can be extracted even by a series of different solvents used successively. This fact explains the varying quantities of fat recorded as being present in the resin.

—W. P. S.

*Study of the oxidation of linseed oil.* Holden and Radcliffe. See XII.

*Oiticica oil—a new drying oil.* Bolton and Revis. See XII.

*Chemical composition of artificial asphalts.* Marcusson. See IIa.

### PATENTS.

*Whiting; Process of making a substitute for* —. H. B. Kipper, Solvay, N.Y. U.S. Pat. 1,264,092, Apr. 23, 1918. Date of appl., Apr. 30, 1915.

A FILLER of the character of whiting is prepared by incorporating with a finely divided inorganic earthy material, e.g. calcium carbonate, a small percentage of salt of a higher fatty acid, such as oleic acid.

—E. W. L.

*Carbon or lampblack [from natural gas]; Manufacture of* —. L. E. Hirt, Charleston, W. Va. U.S. Pat. 1,264,796, Apr. 30, 1918. Date of appl., July 21, 1915.

THE character of carbon black derived from natural gas depends upon the proportion of oil vapour in the gas. To obtain uniform products the oil content of the gas is reduced below a predetermined minimum, e.g. by treatment with cold oil. After the treatment with cold oil, the gas may be intimately mixed with oil at a higher temperature so as to restore to it a definite quantity of oil vapour, which may be in excess of that removed.—C. A. M.

*Lute*. C. E. Ferris, Eaton Rapids, Mich. U.S. Pat. 1,263,398, Apr. 23, 1918. Date of appl., Jan. 26, 1918.

THE lute consists of sulphur 60. and aluminium 40%. —E. W. L.

*Phenolic condensation product varnish; Art of preparing and handling* —. L. V. Redman, Evansston, and A. J. Weith and F. P. Brock, Assignors to Redmanol Chemical Products Co., Chicago, Ill. U.S. Pat. 1,261,615, Apr. 2, 1918. Date of appl., May 8, 1917.

A VARNISH is prepared in the form of two solutions which are kept in separate containers until just before use. One portion consists of a solution of a soluble condensation product of a phenolic substance and a methylene compound containing such excess of the former as to be incapable of yielding a final insoluble product, e.g., 1½ phenolic group to each methylene group. The second portion is a solution of a quantity of an active methylene compound sufficient to combine with the soluble phenolic condensation product and yield a varnish. —C. A. M.

*Resins; Manufacture of* —. F. W. Sperr, jun., Oakmont, and M. Darrin, Wilkinsburg, Pa., Assignors to H. Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,263,813, Apr. 23, 1918. Date of appl., Dec. 7, 1917.

RESINS are manufactured from compounds of the coumarone-indene group by subjecting such compounds to heat and a pressure of 50–300 lb. per sq. in.; or by subjecting a solution of such compounds to heat and pressure, then distilling, and blowing a gaseous fluid through the heated residue. —E. W. L.

*Resins, oleo-resins, oils, and turpentine; Process of extracting* — from resinous woods. D. J. Ogilvy, Cincinnati, Ohio. U.S. Pat. 1,264,551, Apr. 30, 1918. Date of appl., Sept. 10, 1912.

FINELY divided resinous wood is boiled with a mixture of water and a volatile solvent for resins and oils, and the mass of resinous and oily constituents which separates is distilled to separate the lighter turpentine and oils from the heavier oils and resins. The amount of volatile solvent used for the extraction should be greater than the proportion of natural solvent in the wood.—C. A. M.

*Process for obtaining artificial magnesite for the production of artificial stone, cements, pigments, etc.* Ger. Pats. 305,448, 305,449, 305,446, and 305,447. See IX.

*Process for stabilising dispersoids [emulsions, etc.].* Ger. Pat. 302,093. See XII.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

### PATENTS.

*Rubber; Method of vulcanising* —. W. P. Payne, New Haven, Conn., Assignor to L. Candee and Co. U.S. Pat. 1,263,462, Apr. 23, 1918. Date of appl., Sep. 10, 1915.

RUBBER and leather are united by assembling the constituent parts in such a way that the pores of the leather will afford passages for the escape of vapours and gases generated in the rubber during vulcanisation, then subjecting the rubber side to a fluid pressure, exerting a suction on the leather side, and heating the whole at a vulcanising temperature for the time necessary to effect vulcanisation.—E. W. L.

*Rubber waste; Process of reclaiming* —. F. L. Kryder, Akron, Ohio, and E. W. Snyder, Indianapolis, Ind. U.S. Pat. 1,263,567, Apr. 23, 1918. Date of appl., Jan. 4, 1918.

RUBBER waste is heated with a mixture of aniline oil, 10; rubber resin, 10; a mineral hydrocarbon, 2; and carbolic acid, 1 part, under pressure, and a further mixture of acetic acid, 5; turpentine, 1; and naphtha, 3 parts is introduced, the pressure being thereby increased without substantial increase of temperature; the reclaimed rubber is separated from the liquid mass.—E. W. L.

*Rubber coagulum or similar material and processes for preparation of the same for vulcanisation*. W. J. Mellersh-Jackson, London. From General Rubber Co., New York. Eng. Pat. 116,323, June 1, 1917. (Appl. No. 7887 of 1917.)

SEE U.S. Pat. 1,259,794 of 1918; this J., 1918, 313 A.

*Rubber coagula or similar materials and process for the preparation of the same for latex for vulcanisation*. W. J. Mellersh-Jackson, London. From General Rubber Co., New York. Eng. Pat. 116,326, June 1, 1917. (Appl. No. 7905 of 1917.)

SEE U.S. Pat. 1,259,793 of 1918; this J., 1918, 313 A.

## XV.—LEATHER; BONE; HORN; GLUE.

*Tanning; Theory of* —. A reply. J. A. Wilson. J. Amer. Leather Chem. Assoc., 1918, 13, 177–189. (See this J., 1918, 313–314A.)

LYOTROPE influence is apparently related to the hydration of ions, but its mechanism is not understood. It cannot therefore be used to explain the phenomena of the swelling of gelatin in acids. Objection is also taken to Bennett's statement that the adsorption law rests upon as secure a foundation as the theory of ionisation. The work of Gibbs applies only to true solutions. The chief authorities regard the electrical charges of colloidal particles as due to chemical combination, and Zsigmondy gives equations for the action of acids and alkalis on proteins identical with those proposed by Procter and Wilson. On the assumption that gelatin and hydrochloric acid combine to form a highly ionisable salt, equations can be derived with which the experimental results agree exactly. The fact that an empirical adsorption formula can be found to fit the results shows that such adsorption is really chemical combination. Bennett's views on chrome tanning are vitiated by the fact that colloidal chromic oxide is positively charged, whereas his theory requires a negative charge.—F. C. T.



*Chrome [tanning] liquors; Acidity of* —. A. W. Thomas and M. E. Baldwin. J. Amer. Leather Chem. Assoc., 1918, 13, 192–203.

DETERMINATIONS of the acidity of a chrome liquor and a chromium sulphate solution were made by means of the hydrogen electrode. On dilution, while the basicity (*i.e.*, the ratio of chromium to  $\text{SO}_4$  combined with chromium) remains constant, the acidity diminishes, though not so rapidly as the concentration of the solution. For instance, in the original liquor the concentration of hydrogen ion was 0.0049 mol. per litre; when this liquor was diluted to 20 times its volume the concentration immediately fell to 0.0008 and diminished further during 7 days to 0.00055. Equilibrium is attained most rapidly with the most dilute solutions. When the original liquor was diluted to 800 times its volume, equilibrium was reached immediately. The addition of acid or alkali to a chrome liquor causes an immediate rise or fall in the concentration of hydrogen ions, after which equilibrium is attained slowly by a considerable diminution or increase according to whether acid or alkali has been added. The basicity alters according to the amount of added acid or alkali.—F. C. T.

*Hide and pelt; Biological and chemical constituents of* —. Elastins in the tanning process. W. Moeller. Collegium, 1918, 105–117, and 125–137.

THE elastin content of the hide depends largely on the feeding and age of the animals. Elastin is more or less completely desulphurised by means of alkali, but is only dissolved by boiling water under pressure. It differs from collagen in yielding tyrosine among its decomposition products, in being more resistant to the action of acids and alkalis, and in being attacked by trypsin. The preliminary processes aim at overcoming as far as possible the indifference of the elastin and making it more like collagen in its behaviour towards tannins. Trypsin digests elastins, hence bating removes them from the hide. Experiments show that the decomposition products of the elastins are particularly suitable for destroying the compounds of lime with collagen. Together with this action, the removal of the elastic fibres results in a complete change in the structure of the hide. The falling of hides and skins is due to the peptising action of the decomposition products of elastin on the lime compounds, and not to the removal of the lime or elastin.—F. C. T.

*Utilisation of the red dye obtained from the leaf-sheaths and stalks of certain varieties of sorghum.* Dudgeon. See IV.

*Analysis of sulphonated oils.* Schultz. See XII.

*Estimation of chromium in presence of iron.* Schorlemmer. See XXIII.

#### PATENTS.

*Leather; Process for drying japanned or "patent"* —. C. Heyl, Worms, Germany. Eng. Pat. 113,622, Feb. 21, 1918. (Appl. No. 3143 of 1918.) Under Int. Conv., Apr. 27, 1916.

JAPANNED or patent leather is dried under the action of ultra-violet rays in the presence of vapours of ammonia derivatives (hydrazine), or nitrogen, or other inert gases, the drying process being much more rapid than when ammoniacal vapours are used.—C. A. M.

*Leather; Process for drying japanned or patent* —. C. Heyl, Worms, Germany. Eng. Pat. 112,460, Jan. 2, 1918. (Appl. No. 148 of 1918.) Under Int. Conv., Nov. 14, 1916.

IN the drying of patent leather by means of ultra-violet rays the formation of ozone, which is deleterious, is avoided by filtering off all rays of wavelength greater than  $200\mu$ , or else a source of light is used which does not emit such rays. Suitable filters are calc spar, "uvioi glass," water, or aqueous solutions, whilst arc lamps with long drawn arcs may be used without filters.—F. C. T.

*Hides and skins; Treatment of* — prior to and during tannage. L. A. Groth, Purley, Surrey. Eng. Pat. 116,334, June 4, 1917. (Appl. No. 7979 of 1917.)

HIDES or skins are suspended in tanks containing the solutions used in leather manufacture, and are kept in horizontal reversing motion by means of electric power. At the same time an electric current is passed through the liquors and hides in order to accelerate and perfect the various processes. The system is said to effect great saving in time and cost of production.—F. C. T.

## XVI.—SOILS; FERTILISERS.

*Soil acidity. I. Its relation to the growth of plants.* E. Truog. Soil Sci., 1918, 5, 169–195.

THE first of a series of papers on soil acidity, in which a general discussion of the literature on the subject is given. The conclusions of the author are that the amount of lime which should be applied to a soil depends upon three factors:—the lime requirement of the crop or crops to be grown, the degree of acidity of the soil, and the fertility of the soil. The lime requirement of a plant refers to the actual lime needs of the plant itself, especially as to the ease and rate at which lime must be secured from the soil by the plant for normal growth. This is dependent on the lime content and the rate of growth of the plant and the feeding power of the plant for lime as governed by the extent and character of its root system, the internal acidity of the roots, and their excretion of carbon dioxide.

—W. G.

*Soil acidity as influenced by green manures.* J. W. White. J. Agric. Res., 1918, 13, 171–197.

EXPERIMENTS were undertaken to determine the cause of the production of acidity in soils continuously manured with ammonium sulphate. Portions of the acid soil were taken and finely divided young green plants, of leguminous and non-leguminous varieties usually ploughed in for green manure, mixed with them and the mixture maintained at a constant moisture content for nine months. During the first two weeks the lime requirement of the mixture decreased nearly one half, although no change took place in the control portion of soil alone. Thereafter there was an increase in the lime requirement until it exceeded that of the soil alone. Nitrates increased continuously during the period and the amount became twice as much as that in the soil alone, although the latter amount had doubled during the period. The nitrification was insufficient to account for the increased lime requirement. A similar series of experiments was carried out with the same plants first air-dried before being used. The results were similar, although the changes occurred more slowly. Analyses were made to determine the effect of the green manuring on the organic matter in the soil. In every case the amount was larger at the end of

the period than that in the soil alone. Three-fifths of the added organic matter was destroyed during the period; of that left, one-third was present as humus soluble in dilute alkali. The air-dried plants were not so largely decomposed as those applied green.—J. H. J.

*Soil solution in different classes of soil; Relation between biological activities in the presence of various salts and the concentration of the —.*

C. E. Millar. J. Agric. Res., 1918, 13, 213—223.

A SANDY loam and a clay loam soil respectively were mixed with 2% of dried blood, and varying amounts, 0.001—0.7%, of magnesium sulphate, calcium nitrate, calcium chloride, and potassium chloride were added in solution. The depression of the freezing point was determined, and after incubation for 4 days at 30° C. the amount of ammonia produced. It was found that with the larger amounts of the salts, ammonification fell off. In the sandy loam there was a point in the concentration of the salt where this reduction was marked, namely, 0.061% with magnesium sulphate, calcium chloride, and potassium chloride, and 0.2% with calcium nitrate. The increase in osmotic pressure calculated from the lowering of the freezing point, occurred at concentrations of 0.016% and 0.032% respectively. With the clay loam, no point of marked depression of ammonification occurred except with potassium chloride. Thus the effect of salts on ammonification is modified by the nature of the soil. Dried blood alone causes an increase in osmotic pressure varying with the soil used.

—J. H. J.

*Nitrogenous constituents of soil; Effect of heat on some —.* R. S. Potter and R. S. Snyder. Soil Sci., 1918, 5, 197—212.

THREE silt loams and a peat soil were examined, and the heat treatment consisted of two hours at 100° C., dry heat, 2 hours at 200° C., dry heat, 3 hours in an autoclave at 10 lb. per sq. in. pressure on each of three successive days, or 3 hours in an autoclave at 15 lb. per sq. in. once, followed in each case by inoculation with fresh soil emulsion and incubation for 10 or 20 weeks. A blank series without inoculation was also carried out. The amount of ammonia in the soil was increased by all the heat treatments alone, greater increases being, in general, obtained at the higher temperatures. In the case of the peat soil heated at 200° C., however, there was excessive volatilisation of ammonia. The amino-acid nitrogen results followed those for ammonia, but the peat soil was again an exception, there being very little change by any of the treatments. Dry heat at 100° C. caused very little change in the nitrate content of the soil, but the steam heat in the autoclave in each case caused a considerable increase in the nitrate content of all the soils. At 200° C. dry heat there was almost total disappearance of nitrates. In every case there was an increase in the soluble non-protein nitrogen, the lowest results being obtained with the peat soil. In the three loam soils the subsequent incubation for ten hours after dry heat at 100° and 200° C. caused an increase in ammonia, but in one of these cases the 20 hours' incubation caused a decrease. Incubation had very little effect on the ammonia content of the treated peat soil. Ten weeks' incubation in all cases caused a drop in the amino-acid nitrogen, the value rising again, however, during the next ten weeks. Nitrates were increased during both periods of incubation. In the peat soil there was always a decrease in the soluble non-protein nitrogen after incubation, but in the mineral soils there was an increase due to incubation after dry heat at 200° C.

—W. G.

*Peat soils; Determination of lime and phosphoric acid in —. Comparison of the Jönköping with the Bremen method.* C. O. Rost and F. C. Clapp. Soil Sci., 1918, 5, 213—218.

IN the Bremen method the soil sample is incinerated, the ash extracted with *aqua regia*, and the lime and phosphoric acid in the extract determined by the usual methods. In the Jönköping method a quantity of air-dried soil equivalent to 70 grms. of water-free material is digested with 700 c.c. of 12% hydrochloric acid for 48 hours. Higher results are obtained for iron and aluminum oxides, lime, and phosphoric acid by the Bremen than by the Jönköping method. This lower value for lime in the second method is due to a portion of the extracted lime being rendered insoluble in water during the course of the analysis and hence not included in the final precipitation. This loss varies with the amount of iron present.—W. G.

*Soil organisms; Correlation between the production of carbon dioxide and the accumulation of ammonia by —.* J. R. Neller. Soil Sci., 1918, 5, 225—241.

A STUDY of the decomposition of organic matter in soils by pure cultures or by mixtures of pure cultures of micro-organisms. Over a 12-day period the production of carbon dioxide was more uniform than the accumulation of ammonia; but, in general, a high production of carbon dioxide by pure cultures of bacteria was accompanied by a high ammonia accumulation with 1% of cottonseed meal or of alfalfa in sandy loam. Pure cultures of fungi caused more oxidation of carbon but much less accumulation of ammonia than the bacteria. Soil infusions resembled the fungi cultures with respect to ammonia accumulation but were more active in production of carbon dioxide. Mixtures of pure cultures of bacteria showed no antagonism, but in some cases a symbiotic relation seemed to exist.—W. G.

*Sulphur phosphate composts; Experiments with — conducted under field conditions.* J. G. Lipman and H. C. McLean. Soil Sci., 1918, 5, 243—250.

EXPERIMENTS designed to determine the best conditions of preparing sulphur and "floats" (rock phosphate) soil composts for rendering available the phosphorus of "floats," showed that temperature is an important factor in sulphur oxidation and hence in formation of available phosphorus, and consequently for maximum efficiency composts should be made up in warm weather.—W. G.

PATENTS.

*Fertiliser.* J. N. Hoff, New York. U.S. Pat. 1,261,025, Apr. 2, 1918. Date of appl., Sep. 29, 1917.

HUMUS is digested with phosphate rock and acid phosphate (superphosphate) in order to produce reverted phosphate and to render the nitrogen of the humus more available. The mass is neutralised and digested again. It is then inoculated with bacteria and dried till the moisture is reduced below 30%, whereby many of the bacteria are converted into the spore state. The mass is again inoculated with suitable bacteria.—J. H. J.

*Fertiliser; Process of making a mixed potash, nitrogenous, and phosphatic —. Process for producing a mixed potash and nitrogen fertiliser.* R. F. Gardiner, Clarendon, Va. U.S. Pats. (A) 1,261,116 and (n) 1,261,117, Apr. 2, 1918. Date of appl., Aug. 6, 1917.

(A) Rock phosphate, pebble phosphate, apatite, alunite, muscovite, orthoclase, and leucite are



ground separately so as to pass a sieve with 136 meshes to the inch, and are then mixed. Ammonium sulphate is added to the mixture which is heated below 150° C. and then up to 400° C. until the gaseous decomposition products of the ammonium sulphate are copiously evolved. (B) Alunite, muscovite, orthoclase, and leucite are treated as in (A) to produce a potash and nitrogenous fertiliser. —J. H. J.

*Fertilisers; Manufacture of* — J. R. Robinson, Stockton-on-Tees. Eng. Pat. 116,758. (Appl. Nos. 8858, June 20, and 18,802, Dec. 18, 1917.)

A MIXTURE of cellulosic material, e.g., peat, peat moss, rape meal, castor meal, seed husks or refuse, sweepings from maltings, residue from beet sugar refineries, and the like, and soil, or soil-like material, e.g., decayed vegetable matter, cesspool sludge, road sweepings, destructor residues, and the like in a finely divided condition, either alone or together with phosphatic material such as steamed bone flour, basic slag, ground copralites or other mineral phosphates, and/or potassium sulphate, potassium chloride, kainite, flue-dust, etc., is treated with a lime compound, e.g., quicklime, limestone, dolomite, etc. Common salt, especially when kainite is not used, and magnesium sulphate may also be added. When the chemical action has ceased, yeast, mixed to a thin paste with water, is added and thoroughly incorporated with the mass and the whole allowed to remain at a suitable temperature to permit the yeast to multiply and act on the cellulosic matter, etc., converting it into plant-food. —L. A. C.

*Ammonium sulphate; Process of preventing agglomeration and hardening of* — during storage. F. Dahl, Hamborn. Ger. Pat. 305,715, Nov. 25, 1915.

A SALT of potassium or sodium, such as the sulphate or chloride, is added to ammonium sulphate soon after its manufacture, if desired in the proportion required by plant life. The preparation remains permanently dry, and may be stored in paper bags. —C. A. M.

## XVII.—SUGARS ; STARCHES ; GUMS.

*Selivanoff reaction [for detection of ketoses or sucrose]; A modification of the* —. F. Weehuizen. Rec. trav. chim. Pays-Bas, 1918, 37, 302—303.

For the detection of a ketose or sucrose in a mixture of sugars, the mixture is added to a saturated solution of hydrogen chloride in alcohol containing a little resorcinol. A cherry-red coloration appears in the cold in three minutes if a ketose is present. The acid solution must be saturated with respect to the acid and free from water. For the detection of sucrose in milk, 10 c.c. of the milk is shaken with 30 c.c. of absolute alcohol, filtered, and 10 c.c. of the filtrate evaporated to a syrup, cooled, treated with 2—4 c.c. of the alcoholic acid solution and 50 mgrms. of resorcinol, and the mixture triturated. A red colour is developed in three minutes if the milk contains 0.5% of sucrose. —W. G.

### PATENT.

*Honey; Preparation of artificial* — containing iron. W. Schmitz, Berlin, and E. Jansen, Berlin-Wilmersdorf. Ger. Pat. 303,754, Mar. 28, 1915.

THE hydroxide or other suitable compound of iron is added to sugar during inversion whereby salts of iron are produced with the acid used for inversion and saccharates of iron with the sugar.

—H. J. H.

## XVIII.—FERMENTATION INDUSTRIES.

*Yeast; Influence which the plant function of — exercises on the yield of alcohol: new interpretation of fermenting power.* L. Lindet. Comptes rend., 1918, 166, 910—913.

THE author considers that fermenting power must be considered as a function of the plant power, as measured by the amount of sugar consumed by unit quantity of yeast for its plant life, and the zymase power, as measured by the sugar consumed by unit quantity of yeast in accomplishing its zymatic function. The experimental results show that the less yeast there is and the more the fermentation is prolonged, the greater is the proportion of sugar consumed by the plant function and the lower is the yield of alcohol. Lowering of the temperature has a greater effect on the duration of the fermentation than on the weight of yeast produced. —W. G.

*Yeast foods; Carbohydrates other than sugar [e.g., waste liquors from cellulose manufacture] which are suitable as* —. T. Bokorny. Chem.-Zeit., 1918, 42, 260.

THE waste liquors from cellulose manufacture may be used for growing yeast; these liquors contain mannose, levulose, galactose, and xylose. Pentoses and dextrins may also be used for the purpose, but arabinose, rhamnose, and methylpentoses are of little value. —W. P. S.

*Lactic acid formation; Influence of lactic acid on* —. W. van Dam. Biochem. Zeits., 1918, 87, 107—122.

INVESTIGATIONS were made to ascertain whether the stoppage of fermentation by the coccal form of bacteria is due to the action of the hydriion of the lactic acid produced or to the undissociated molecule. The conclusion is drawn that the stoppage is due to both. If sodium lactate is added to the whey, further acid formation ceases when the concentration of the undissociated molecule reaches 0.02N. In such cases, the final hydriion concentration fails to reach the toxic amount of about  $10 \times 10^{-5}$ . If hydrochloric acid is added to the whey before inoculation, so much lactic acid is produced as to bring the final hydriion concentration to  $10 \times 10^{-5}$  —  $20 \times 10^{-5}$ . In this case the amount of undissociated lactate remains below the toxic amount. Attention is called to the fact that certain conclusions of other authors as to the value of nitrogenous nutrients is vitiated by their failing to take into account their buffer action. (See also J. Chem. Soc., Aug., 1918.) —S. B. S.

*Potassium requirements of bacteria.* G. P. Koch. Soil Sci., 1918, 5, 219—224.

WORKING with cultures of *B. subtilis* it was found that potassium is essential to the development and activities of the organisms. In its absence, magnesium sulphate and calcium phosphate cannot be utilised by the organism. Measuring the activity of the *B. subtilis* from the amount of ammonia formed by it from dialysed peptone, the maximum activity and development was obtained by supplying 0.24 mgrm. K<sub>2</sub>O, in addition to the 0.1 mgrm. present in the peptone, in 100 c.c. of solution. Any amount above this did not further increase the activity of the bacteria. —W. G.

*Enzymes and bacteria; Action of cyanhydrins on* —. M. Jacoby. Biochem. Zeits., 1918, 87, 129—134.

THE action of the urease of soya bean is strengthened by the presence of the cyanhydrin of

acetaldehyde, but inhibited by the corresponding propaldehyde derivative. Both compounds inhibit strongly the formation of enzymes by bacteria, whilst they exert a very moderate deleterious action on the bacteria themselves. (See also J. Chem. Soc., Sept., 1918.)—S. B. S.

*Concentration of sulphite waste liquors.* Oman. See V.

*Nucleic acid and its analytical examination.* Chapman. See XX.

#### PATENTS.

*Proteolytic enzymes: Process for producing an extract of —.* Kancaguchi Boseki Kabushiki Kwaisha, Tokyo-Fu, Japan. Eng. Pat. 106,501, May 16, 1917. (Appl. No. 7051 of 1917.) Under Int. Conv., May 16, 1916.

PROTEOLYTIC enzymes capable of dissolving sericin and therefore of value in the manufacture of silk, as well as for medicinal purposes, are extracted from cocooning insects of the family *Bombycidae*, or their discharges or secretions (cast-off skins, etc.), obtained as waste products in sericulture and filature), by digesting with water, e.g., for 6–18 hours at 5°–10° C., or for 5–15 mins. at 40° C., separating the extract by pressure and concentrating at a low temperature, e.g., *in vacuo* at 45° C. The products thus obtained may be used, or if necessary the enzymes may be precipitated therefrom by known means.—J. H. L.

*Wine; Non-alcoholic — and process of making same.* J. Russell, New York. U.S. Pat. 1,264,564, Apr. 30, 1918. Date of appl., July 5, 1917.

WINE is heated and the alcoholic and other vapours are condensed, and the condensed liquid is intimately mixed with melted vaseline in order to absorb the ethereal components and separate them from the alcohol. The unabsorbed liquid is removed and the other components are recovered from the vaseline by distillation and returned to the wine. —J. F. B.

*Process of treating the residual liquor obtained in the hydrolysis of cellulosic materials.* U.S. Pat. 1,261,328. See V.

*Densimetric methods and appliances.* Eng. Pat. 115,662. See XXIII.

### XIXA.—FOODS.

*Food substitutes; Directions for the examination and licensing of —.* German Food Substitutes Commission. Z. anal. Chem., 1918, 57, 170–176.

GENERAL directions are given for deciding as to the use of various food substitutes, the points considered being the nature of the substance, its keeping qualities, cost, raw materials required for its preparation, etc. Baking powders must yield 2.35 to 2.85 grms. of carbon dioxide from the amount of the powder used per 0.5 kilo. of flour; the excess of sodium bicarbonate present must not exceed 0.8 gm.; the use of bisulphates, bisulphites, alum, etc., is not permitted, but the powder may contain 20% of calcium carbonate. All powders having the general composition of baking powder must be sold as such and not as egg-powder, etc. Egg powders must have a food value equal to that of eggs, otherwise the word "egg" cannot be used in their description; they must not contain glue or gelatin. Vanillin powder must contain at least 1%, and vanillin salt at least 2% of vanillin, and the latter

may not be substituted by piperonal or coumarin. Spice substitutes must have a strength equal to that of the spices they replace; the sand content must not exceed 2.5%. Tea substitutes containing a large proportion of valueless vegetable substance are not permitted; the description of tea substitutes must not be such as would indicate that they consist of real tea. Other substitutes mentioned are those for pudding powder, jellies, seasonings, honey, etc. —W. P. S.

*Fat in milk; Additive factors for the calculation of — from the specific gravity and total solids.* L. J. Harris. Analyst, 1918, 43, 263–267.

The percentage of fat in milk may be rapidly calculated by adding together two factors respectively corresponding with the sp. gr. and total solids. The factors are obtained as follows:—

$$t = \frac{T}{1.2} - 8; s = 8 - \left( \frac{G}{4.8} + 0.116 \right),$$

where  $t$  represents the factor for total solids,  $s$  that for the sp. gr., and  $T$  and  $G$  have the same meaning as in Richmond's formula. Determination of the total solids by direct evaporation of 5 grms. of milk in a porcelain dish gives concordant results. The two sets of factors for sp. gr. from 1.0220 to 1.0370, and for total solids 10.00 to 13.99% have been embodied in tables, and the percentage of fat calculated by means of them almost invariably agrees within 0.1% with that determined by the Werner-Schmidt method, and frequently the agreement is as close as 0.01%.—C. A. M.

*Gerber test [for milk]; Conditions affecting accuracy in the —.* F. E. Day and M. Grimes. Analyst, 1918, 43, 215.

THE most concordant results are obtained when the centrifuging is maintained for three mins. at 1000 revs. per min. In the case of skimmed milks, re-heating followed by a second centrifuging is necessary, but with new milks this procedure is liable to give irregular results. It is important to pay attention to the temperature of the fat at the moment of reading its volume, and it is recommended that the tube be calibrated with an oil which is liquid at the ordinary temperature. (See also this J., 1918, 278A.)—W. P. S.

*"Cocoa teas"; Analysis of —.* J. L. Baker and H. F. E. Hulton. Analyst, 1918, 43, 189–197.

"Cocoa teas" consist of cacao shell or of cacao shell mixed with full-fatted cacao nib. Results of analyses of four samples of the product are given in the following table:—

	A.	B.	C.	D.
	%	%	%	%
Moisture .. .. .	11.0	10.8	8.76	10.44
Fat .. .. .	4.0	20.05	12.65	4.7
Total ash .. .. .	15.0	8.0	7.48	10.04
Ash soluble in water .. .. .	2.2	5.78	5.14	6.68
Ash insoluble in hydrochloric acid .. .. .	5.14	0.36	0.31	0.5
Alkalinity of soluble ash, as K <sub>2</sub> O .. .. .	0.33	3.85	3.08	4.28
Nitrogen .. .. .	2.03	2.38	2.38	2.66
Crude fibre .. .. .	12.8	14.87	14.87	16.62
Substances soluble in cold water .. .. .	21.7	17.5	20.2	22.0

Sample A evidently consists of shell without any admixture of nib; the high ash is due to the shell having been obtained from beans which had been "clayed" or "earthed." B consists of a mixture of shell, 61%, and nib, 36%, which has been alkalised. Sample C has also been alkalised and contains shell, 86%, and nib, 14%. The sample D consists wholly of shell.—W. P. S.



*Cocoa and cacao products; Estimation of shell in —.* J. L. Baker and H. F. E. Hulton. Analyst, 1918, 43, 197—201.

THE quantity of shell in cocoa is usually estimated from the levigation residue, the nitrogen content, or the crude fibre content. The authors have investigated the accuracy of these methods, mainly with the purpose of ascertaining the possibility of distinguishing between Grade A and Grade B cocoas mentioned in the Cocoa Powder Order, 1918; this Order provides that Grade A cocoa shall not contain more than 2% of shell, whilst Grade B cocoa shall not contain more than 5% of shell and not less than 22.5% or more than 30% of cacao butter. In the levigation process 10 grms. of the powder is extracted for 24 hrs. with ether in a Soxhlet apparatus, then dried, mixed into a paste with water, transferred to a 500 c.c. cylinder, and diluted to 400 c.c.; after mixing and settling for 15 mins., the supernatant liquid is drawn off, the sediment diluted to 400 c.c., the liquid drawn off after 10 mins., and this operation twice repeated with 5 mins.' settling. The sediment is then rinsed into a platinum basin, evaporated, dried, and weighed, an allowance being made for the ash present. The figures adopted for sediment are 3% for dry, fat-free nib and 30% for dry, fat-free shell. The percentage of shell in the original sample is calculated from the formula:

$$S\% = \left( \frac{100 M}{100 - (F + W)} \right) - 3 \left( \frac{100 - (F + W)}{27} \right), \text{ where } M =$$

percentage of levigation sediment,  $F$  = percentage of fat, and  $W$  = percentage of water in the sample. If the percentage of shell is calculated from the crude fibre content the formula used is:

$$S\% = \left( \frac{K \times 100}{100 - (F + W)} - 5.7 \right) \times \frac{100 - (F + W)}{11.1}, \text{ where } K =$$

percentage of crude fibre. The fibre contents of dry, fat-free nib and shell are 5.7 and 16.8%, respectively. The percentage of shell is calculated from the nitrogen content by the formula:

$$S\% = 2.17(100 - F - W) - \frac{100N}{2.26}, \text{ where } N = \text{per-}$$

centage of nitrogen; the nitrogen contents of dry, fat-free nib and shell are 4.9 and 2.64%, respectively. The results of analyses of various samples by the three methods were as under:—

	Moisture.	Fat.	Levigation residue	Nitrogen.	Crude fibre.	Percentage of shell calculated from		
						Levigation.	Nitrogen.	Crude fibre.
	%	%	%	%	%			
A	9.3	12.0	17.37	2.66	9.65	55.7	53.0	46.8
B	2.0	28.0	2.57	3.57	4.04	1.7	0.0	7.6
C	1.7	28.6	4.23	3.29	—	7.9	6.3	—
D	4.2	32.6	3.70	3.22	—	6.6	0.0	—
E	2.0	25.5	0.75	3.29	—	0.0	11.7	—
F	4.7	21.9	3.06	3.50	—	3.2	4.3	—

The methods are suitable for the estimation of considerable proportions of shell but appear to be inadequate for distinguishing between cocoas of Grades A and B where the difference in shell content is small. In the case of cocoa powder, the fat content cannot be used as a basis in calculating the percentage of shell since the cocoa has been partially de-fatted.—W. P. S.

*Cacao "germ."* P. A. E. Richards. Analyst, 1918, 43, 214.

A SPECIMEN of cacao "germ" obtained from roasted cacao bean, and free from nib and shell, contained fat, 3.58, and moisture, 7.2%. The fat-free, dry

material contained: nitrogen, 5.5; crude fibre, 3.65; total ash, 7.3; soluble ash, 4.0; cold-water extractives, 28.7%. The alkalinity of the soluble ash was 1.69% (as  $K_2O$ ) and the butyro-refractometer value of the fat was 66 at 35° C. The levigation method yielded a residue of 38.3%.—W. P. S.

*Chemical study of nuts of sanga-sanga (Ricino-dendron africanum.)* Pieraerts. See XII.

*A modification of the Seliwanoff reaction [for the detection of sucrose in milk].* Weehuizen. See XVII.

*Influence of lactic acid on lactic acid formation.* Van Dam. See XVIII.

#### PATENTS.

*Bread and ingredients therefor; Manufacture of —.* J. Chandler, London. Eng. Pat. 115,556, Aug. 16, 1917. (Appl. No. 11,788 of 1917.)

LINSEED is heated with water in such proportion that the filtered extract sets to a thin jelly on cooling; for convenience in handling and weighing it may be stiffened by addition of a small quantity of gelatin. The product is used in bread-making, e.g., 1—4 lb. to 280 lb. of flour, to improve the edible and keeping qualities of the bread, especially when a large proportion of flour other than wheat flour is used.—J. H. L.

*Bran; Treatment of — for incorporation in bread.* H. Feddersen, Berlin. Ger. Pat. 303,577, July 18, 1911.

THE bran is made into a dough, left for a time under conditions suitable for enzyme action, and then, while warm, subjected to wet grinding prior to incorporation with dough for bread. Palatability and availability of cell contents are increased by the treatment, which also shortens the time necessary for reducing the bran to the required degree of fineness, and therefore reduces the risk of infection with harmful ferments.—H. J. H.

*Butter-fat; Sterile — and method of producing the same.* C. E. Rogers, Detroit, Mich. U.S. Pat. 1,264,336, Apr. 30, 1918. Date of appl., Jan. 17, 1912.

BUTTER is melted at a temperature not exceeding 180° F. (82° C.), and the fat is separated from the casein, etc., freed from moisture at about 212° F. (100° C.), and sterilised, under exclusion of air, at about 240° F. (115.5° C.).—C. A. M.

*Margarine; Apparatus for the manufacture of —.* C. J. A. Rydberg, Copenhagen. Eng. Pat. 116,603, Aug. 8, 1917. (Appl. No. 11,397 of 1917.)

THE constituents of the margarine are emulsified in a rotating container, and the emulsion crystallised on hollow rollers arranged in pairs within the container, and provided with means for interior cooling. The emulsion may also be kneaded by the cooling rollers arranged eccentrically within the container.—C. A. M.

*Fruits, vegetables, and similar articles; Method of and apparatus for dehydrating —.* G. H. Benjamin, New York. U.S. Pat. 1,264,600, Apr. 30, 1918. Date of appl., July 10, 1917.

A CURRENT of air at definite temperature and humidity is passed over a portion of the material to be dried, then heated to its original temperature, mixed with a fresh quantity of air proportional to

the increased humidity, and passed over a second portion of the material, this process being repeated over successive portions until the whole of the material has been subjected to air currents. Apparatus for this process comprises a pump, an inlet and outlet for the air, with a series of cells between the inlet and outlet, in which removable trays are arranged in superposed zig-zag order, means for heating the air between each series of trays, and for regulating the volume and temperature of the air during its passage through the cells.—C. A. M.

*Food product and process of producing same.*  
R. Graham, Cupar, Scotland. U.S. Pat. 1,264,876,  
Apr. 30, 1918. Date of appl., May 28, 1917.

Whole wheat bread is prepared by treating wheat bran and middlings or other bran-containing material with a diastatic and peptic agent, such as malted wheat flour, keeping the mixture warm for about 12 to 36 hours, adding white flour to the product, making it into dough, and baking for at least  $3\frac{1}{2}$  hours at a relatively low oven temperature. The wheat malted for this purpose is dried and kept for about 3 months at temperatures gradually falling from about 200° F. to 100° F. (93° to 38° C.).—C. A. M.

## XIXB.—WATER PURIFICATION; SANITATION.

*Drinking water; Purification of —.* C. Kippenberger. Ber. deuts. Pharm. Ges., 1918, 28, 230—240.

A short general account of processes of purification and sterilisation of drinking water used in Germany, covering the application of bleaching powder, chlorine gas, permanganate, vegetable and animal charcoal, ultra-violet light, and ozone.—J. H. J.

*Drinking water; Removal of iron from —.*  
O. Beck. Z. angew. Chem., 1918, 31, 112, 115—116.

The water in the district of Antwerp, in so far as it is not derived from deep wells, has to a large extent the character of moorland water, and, owing to the deposits of iron pyrites in the soil, contains a high proportion of iron as bicarbonate and as ferrous sulphate. Treatment of the water with a mixture of bleaching powder and sodium chloride, and then with Schäffer's aluminium sulphate-charcoal (blood charcoal containing 16.6% of aluminium sulphate), and filtration through a Dunbar filter, removed the iron and gave a sterile water; but it was found that after standing, the water gave a deposit of calcium sulphate, and acquired a bitter taste due to aluminium compounds, whilst the filter rapidly became choked by the sediment. These drawbacks were obviated by a method in which 250 c.c. of the water is agitated with 400 c.c. of 3% hydrogen peroxide solution, which is added in successive small quantities, this being followed by 100 grms. of finely powdered anhydrous sodium carbonate. The precipitate subsides within 30 mins., and the water can be readily filtered, yielding a filtrate free from iron and practically sterile. The method is suitable for the removal of not too excessive quantities of iron from water, but frequent renewal and disinfection of the filter is advisable.—C. A. M.

*Sewage disposal; The Miles acid process for —.*  
Met. and Chem. Eng., 1918, 13, 591—594.

A small portion of the sewage to be treated is pumped through an absorption tower, where it

meets a counter current of sulphur dioxide. After saturation, the sewage is returned to the main sewer to be mixed with the bulk of the sewage. The whole then flows through two settling tanks in series, where four hours' sedimentation is allowed. According to R. S. Weston, about a ton of sulphur dioxide per million gallons of sewage is used, or 382 tons of nitre cake with 200 lb. of sulphur dioxide may be used instead. The sludge collects at the bottom of the tanks, and when pumped out contains 80—92% of water. The drying of the sludge is difficult owing to the large amount of crude fibre (up to 40%), but this may be screened out, pressed down to 50% of water, and extracted separately. The dried sludge is extracted, and the grease obtained consists of glycerides and fatty acids in about equal proportions, and may amount to 430 lb. from a million gallons of sewage. The residue left contains 45%  $\text{NH}_3$  and 1—2%  $\text{P}_2\text{O}_5$ . The effluent from the settling tanks contains 800—1200 lb.  $\text{K}_2\text{O}$  and 50 lb.  $\text{P}_2\text{O}_5$  per million gallons, and has an acidity of 0.066—0.01% as  $\text{SO}_2$ . It is nearly sterile, and is without odour. The process is in operation in a 10,000-gallon test plant at New Haven, Connecticut.—J. H. J.

*Albuminoid ammonia; Determination of — in liquids containing gas liquor.* H. F. Stephenson. Analyst, 1918, 43, 215—216.

THIOCYANATES must be removed before albuminoid ammonia can be determined in liquids containing gas liquor, since thiocyanate yields ammonia when boiled with alkaline permanganate solution. A definite volume of the sample (which should be alkaline) is treated with a quantity of aluminium sulphate sufficient to give a decided precipitate, filtered through an asbestos filter, the filtrate acidified with nitric acid, the thiocyanate precipitated by the addition of silver nitrate, and the mixture again filtered. The filtrate is then distilled in the usual way for the determination of the albuminoid ammonia. Owing to the slight solubility of silver thiocyanate, a control determination should be made on distilled water to which a small quantity of thiocyanate has been added and a corresponding correction applied to the results obtained; this correction is practically a constant.—W. P. S.

*Disinfection; Application of the mass law to the process of —.* R. E. Lee and C. A. Gilbert. J. Phys. Chem., 1918, 22, 348—372.

From a study of the velocity of disinfection in the cases of *B. typhosus* with 0.2% phenol at 37.5°, anthrax spores with mercuric chloride at 20°, and *Staphylococcus pyogenes aureus* with 0.2% phenol at 20° C., it is shown that disinfection is an orderly time process closely analogous to a chemical reaction. A definite logarithmic relationship between velocity and concentration has been established in all cases examined. (See also J. Chem. Soc., Aug., 1918.)—J. F. S.

*Arsenates; Calcium — [as insecticides].* R. H. Robinson. J. Agric. Res., 1918, 13, 281—294.

WHEN commercial calcium arsenates have been used in orchards as a spray for fruit trees, it has led to burning of the foliage owing to their solubility in water, and experiments were made to determine whether an improved product could be produced. Pure calcium arsenate and calcium hydrogen arsenate were prepared. When tested for solubility by shaking with water at 25° C., calcium hydrogen arsenate yielded a solution containing 0.37 grm. per 100 grms. of water, which would be sufficient to render it unsuitable as a spray. Calcium arsenate, however, was so sparingly soluble (0.013 grm. at 25° C.) as to render it safe for use, and it was also



sufficiently stable in presence of acid and alkali. The addition of 1 part of lime to every 2 parts of either arsenate renders it more nearly insoluble, and would make the calcium hydrogen arsenate available for spraying. It was found that either arsenate could be mixed with wet and dry lime-sulphur without the ingredients losing their stability. (See also J. Chem. Soc., 1918, ii., 232.)—J. H. J.

*Effluents from ammonia-recovery plant of coke ovens.* Marsson and Weldert. See IIA.

*Recovery of grease from waste materials by the solvent extraction process.* Garner and Carmichael. See XII.

#### PATENTS.

*Water softener; Process for producing a* —. G. H. Widner. Eng. Pat. 107,197, May 17, 1917. (Appl. No. 7070 of 1917.) Under Int. Conv., May 20, 1916.

SEE U.S. Pat. 1,207,826 of 1916; this J., 1917, 161. Instead of heating the aluminium silicate mineral with salt, it may be heated alone and then steeped in salt water.

*Water-softening material; Process for producing a* —. G. H. Widner, Des Moines, Iowa. Eng. Pat. 116,023, May 17, 1917. (Appl. No. 15,744 of 1917.)

THE source of the material is natural clay containing preferably 20% of alumina and 53% of silica. It is reduced to a fine mud in presence of sodium chloride, then concentrated and forced under pressure through a screen which delivers the material in fine strings; these are cut off in suitable lengths, dried, and baked at 600° C. until hard enough to resist the powdering action of water. (See also U.S. Pat. 1,207,826; this J., 1917, 161.)—J. H. J.

*Sterilising water by means of ozone; Apparatus for* —. L. H. E. Régot, Paris, Assignor to Steynis Ozone Co. U.S. Pat. 1,264,360, Apr. 30, 1918. Date of appl., Aug. 3, 1916.

THE water to be sterilised operates an injector which draws air through an ozone generator. An electric circuit supplies current to the generator. In the circuit is a rotating contact arranged to be driven by a motor operated by the water to be sterilised.—J. H. J.

*Water; Sterilisation of — and removal of iron and manganese therefrom by electrolytic means.* F. Tiemann, Berlin. Ger. Pat. 301,585, Apr. 23, 1916.

THE water is caused to flow through the anode compartment of a diaphragm electrolytic cell, and a conductor of the first order, indifferent to oxygen, e.g., carbon or pyrolusite, is added to it continuously, in the form of fine powder or granules, which will remain in suspension in the flowing water. The ionised oxygen produced oxidises and precipitates iron and manganese and destroys bacteria. After settling, the sludge is used again, and the water is filtered through sand.

*Water; Sterilisation of — and removal of iron and manganese therefrom by electrolytic means.* F. Tiemann, Berlin. Ger. Pat. 302,226, Sept. 30, 1916. Addition to Ger. Pat. 301,585 (see preceding abstract).

Economy of apparatus and current are effected by subjecting only a small portion of the water to

electrolysis with the addition of conducting material—preferably carbon grains—in suspension. The ionised oxygen is adsorbed or absorbed by the carbon grains and exerts its purifying action when the treated water is mixed with the main bulk of untreated water.—H. J. H.

*Water; Sterilisation of — and removal of iron and manganese therefrom by electrolytic means.* F. Tiemann, Berlin. Ger. Pat. 302,227, Oct. 11, 1916. Addition to Ger. Pat. 301,585 (see preceding abstracts).

INSTEAD of suspending the conducting material in the water, it is used as a filling material, in the form of hollow balls or the like, in the anode chamber, being retained in place by screens. The water flows over and through the material (e.g. carbon, platinum, pyrolusite).—B. V. S.

*Boiler-feed water; Process for removing gases from, softening, and preheating* —. I. P. Winer, Warsaw. Ger. Pat. 302,643, Oct. 8, 1913.

IN the upper part of the boiler is a receptacle having an elongated tubular portion, similar to a smoker's pipe; the tube passes through the wall of the boiler, extends downwards, and then re-enters the boiler near the bottom. The feed water is forced through a set of nozzles arranged within the bowl of the receptacle, and is sprayed against the wall of the boiler, whence it is deflected by means of guide-plates into the receptacle, and thence into the boiler. The tubular extension of the receptacle, outside the boiler, is connected to a gauge-glass.

*Separation and settlement of solids and semi-solids from sewage liquids and analogous liquids.* W. Clifford, Wolverhampton, and Jones & Attwood, Ltd., Amblecote, Staffs. Eng. Pat. 115,872, May 17, 1917. (Appl. No. 7064 of 1917.)

A TANK in the form of an inverted cone is used, into which the sewage is brought by a horizontal pipe, the mouth of which directs the current into a much wider bucket suspended in the upper part of the tank. This has the effect of breaking up the current and causing eddies which pass upwards out of the bucket. These eddies are prevented from spreading by a guard wall of much wider diameter surrounding the bucket and carried upwards above the level of the liquid in the tank. The guard wall is open at the bottom, and the liquid passes from it with a low velocity and without any disturbing effect upon the deposited sludge in the bottom of the tank. The liquid finally passes out of the tank over a weir at the circumference. The sludge is withdrawn through a pipe in the apex of the cone.

—J. H. J.

*Sewage and other foul liquids; Apparatus for aerating* —. R. Ames, Brighton. Eng. Pat. 115,933, June 19, 1917. (Appl. No. 8785 of 1917.)

THE sewage enters a channel situated alongside a treatment tank, from which it is siphoned into a trough which travels over the tank. The sewage flows into the tank through vertical pipes with bell-mouths, which depend from the trough to the bottom of the tank. Hollow pipes pass down the centres of the vertical pipes and carry horizontal fans at their lower ends, revolving inside the bell-mouths, whereby vortices are created and air is drawn down the pipes. The propellers of the fans are hinged in order to permit of the fans being drawn up the pipes when they require to be cleaned. A further supply of air is obtained through a pair of tubes from the surface to the inside of each bell-mouth.—J. H. J.

*Sewage sludge; Process for removing fat from —.* C. Stiepel, Berllu-Steglitz. Ger. Pat. 305,768, Aug. 6, 1915.

The fat is converted into calcium soap which is then extracted with a halogen compound of a hydrocarbon, such as carbon tetrachloride, trichloroethylene, etc. The difficulty of using an acid process in presence of much calcium carbonate is thus obviated.—C. A. M.

*Process of making exchange silicates. Process of making exchange bodies.* U.S. Pats. 1,263,706 and 1,263,707. See VII.

*Utilisation of nitre-cake [for preparation of an insecticide].* Eng. Pat. 116,181. See X.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Morphine in opium; Determining —.* C. A. Hill. Analyst, 1918, 43, 214.

The use of a Gooch crucible furnished with a filter paper is recommended for collecting the precipitated morphine obtained in the B.P. method for the determination of this alkaloid; the precipitate is washed with morphinated water in the usual way. —W. P. S.

*Morphine; Effect of codeine in hindering the precipitation of — by ammonia from a solution of its lime compound.* H. E. Annett and H. Singh. Analyst, 1918, 43, 205–215.

The presence of codeine decreases the amount of morphine precipitated from its calcium hydroxide solution by ammonia in the presence of alcohol and ether (B.P. method), the effect increasing as the ratio of codeine to morphine increases. Considerably higher results for morphine in opium are obtained if the codeine is removed by extracting the solution with toluene previous to the precipitation with ammonia. This applies particularly to Indian opiums with a high codeine content. (See also J. Chem. Soc., Aug., 1918.)—W. P. S.

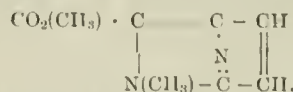
*Brucine; Colorimetric determination of — in presence of strychnine.* A. Wüber. Z. angew. Chem., 1918, 31, 124.

Dowzako's method of determining brucine by means of the colour reaction with nitric and sulphuric acids (this J., 1902, 1560) has the drawback that the original red coloration decreases with varying velocity according to the concentration of the alkaloidal solution, whilst strong nitric acid gives a yellow coloration with strychnine, and the brucine reaction is not sensitive with dilute nitric acid. Trustworthy results may be obtained by using as the reagent a mixture of equal vols. of strong nitric acid (sp. gr. 1.4) and 20% sulphuric acid, and adding immediately after the reaction, 2 c.c. of a saturated aqueous solution of potassium chlorate. The reaction is capable of detecting 0.1 mgrm. of brucine in 50 c.c. of a solution containing 0.1 gm. of strychnine. The standard solution of brucine used for the colorimetric comparison should also contain 0.1 gm. of strychnine, the effect of this alkaloid on the reaction being largely independent of the concentration. (See also J. Chem. Soc., Aug., 1918.)

—C. A. M.

*Ricinine.* B. Böttcher. Ber., 1918, 51, 673–687.

CASOR-OIL seed contains about 0.15% of the alkaloid, ricinine, which is the methyl ester of ricininic acid. This and its degradation products show the reactions of pyridine-carboxylic and glyoxaline-carboxylic (c.g. histidine) acids. Combining these facts with what was previously known about ricinine, the following formula is proposed:—



(See further J. Chem. Soc., 1918, 1, 301.)

—J. C. W.

*Choline; Microchemical tests for —.* N. Schoorl. Pharm. Weekblad, 1918, 55, 363–369.

An aid to the identification of choline is furnished by the formation of double salts by its hydrochloride with the chlorides of platinum and of gold, and with mercuric and bismuth iodides. The microchemical characteristics of these salts and of the picrate and picrolonate are described.—A. J. W.

*Nucleic acid and its analytical examination.* A. C. Chapman. Analyst, 1918, 43, 259–263.

NUCLEIC acid derived from yeast, frequently described as "plant-nucleic acid," is a white friable substance with the composition



The commercial product should be white or nearly so, and should dissolve in an aqueous solution of sodium acetate, or in dilute solutions of ammonia or of sodium or potassium hydroxides, to form bright and nearly colourless solutions. The acid should be precipitated as a white curd-like substance from an aqueous solution of the sodium salt, and the precipitate should dissolve on further addition of a large excess of hydrochloric acid. Sodium nucleate gives a bulky greenish-blue precipitate with a solution of copper acetate acidified with acetic acid; a white flocculent precipitate, becoming granular, with calcium chloride in presence of a few drops of acetic acid; and a white gelatinous precipitate with silver nitrate. In the biuret test for proteins it should give, at most, only a slight indication of purple. Inorganic phosphate is detected by adding to a solution of the acid in considerable excess of ammonium or sodium acetate a few drops of acetic acid and a little uranium acetate. In the absence of inorganic phosphate the flocculent precipitate should dissolve completely on boiling. Nitrogen may be determined by Kjeldahl's method, and phosphorus by fusing the sample with six times its weight of sodium carbonate containing 10% of potassium nitrate, and determining the phosphoric acid by the molybdate or magnesia methods. Commercial nucleic acid of good quality should contain not much less than 15% of nitrogen (theory 16.1%) and 9% of organic phosphorus calculated on the moisture-free substance (theory 9.5%). If the ratio of organic phosphorus to nitrogen (1:17) is approximately correct, the presence of material quantities of the products of hydrolysis is excluded. Nucleic acid may be identified by the identification of guanine and adenine in the products of hydrolysis. For this purpose 10 grms. of the acid is heated in a boiling water-bath for 2 hours with 40 c.c. of 10% sulphuric acid in a small flask fitted with an air condenser. Strong ammonia is then added until present in an excess of about 2%, and the precipitated guanine is filtered off, washed with 1% ammonia solution, dissolved in the smallest possible amount of dilute sulphuric acid, and the base reprecipitated by means of ammonia, and converted into its hydrochloride.



which may be identified by well-known tests. The ammoniacal filtrate is acidified with sulphuric acid, and the adenine precipitated as a cuprous compound by means of 10% copper sulphate solution. The adenine copper compound is suspended in water, decomposed with hydrogen sulphide, the filtrate evaporated to dryness, the residue dissolved in dilute sulphuric acid, and the adenine sulphate allowed to crystallise, and identification tests applied. (See also J. Chem. Soc., Aug., 1918.)—C. A. M.

*Acetylsalicylic esters.* A. Astruc. J. Pharm. Chim., 1918, 17, 386—388.

In the analysis of methyl and ethyl acetylsalicylates, determinations should be made of the ester content by saponification. In addition to giving correct saponification values, the substances should have a neutral reaction, since mixtures of 92.78% of acetylsalicylic acid and 7.22% of methyl alcohol, or 86.53% of acetylsalicylic acid and 13.47% of ethyl alcohol, have the same saponification values as pure methyl and ethyl acetylsalicylates, respectively.—W. P. S.

*Chloroform; Preparation of — from ethyl alcohol.* K. Ukita. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 219—226.

Pure chloroform can be obtained in good yield from alcohol by Besson's process; the cost is considerably less than when acetone and bleaching powder are used as raw materials, especially under present conditions, and the method affords a useful means of utilising waste chlorine in bleaching powder and electrolytic alkali works. Chlorine is passed into alcohol until the density of the liquid reaches 35° B. (sp. gr. 1.32), or until two layers which are formed at first become united. A mixture of bleaching powder, milk of lime, and water (500 grms., 100 grms., and 2 litres respectively per 100 grms. of alcohol) is then added, and the chloroform is recovered by distillation. Yield, 95—98 grms. per 100 grms. of alcohol.

*Urotropine and formaldehyde; Behaviour of — in the organism.* E. Salkowski. Biochem. Zeits., 1918, 87, 143—162.

The most satisfactory test for detecting formaldehyde in the presence of urotropine is that of Jorissen as modified by Vanino (use of phloroglucinol and sodium hydroxide). Solutions of urotropine cannot be kept for any length of time without decomposition except in the presence of small amounts of sodium carbonate (0.1—1%). (See also J. Chem. Soc., Sept., 1918.)—S. B. S.

*Organic halogen compounds; Catalytic reduction of —.* K. W. Rosenmund and F. Zetzsche. Ber., 1918, 51, 578—585.

The removal of halogen atoms from organic compounds by the action of hydrogen in the presence of a catalyst has recently been developed into methods for estimating halogens (see Kehler, this J., 1917, 568). The authors give an account, with examples, of their experience with palladinised barium sulphate and colloidal palladium as catalysts.

—J. C. W.

*Aldehydes; New method for the preparation of —.* K. W. Rosenmund. Ber., 1918, 51, 585—594.

One of the best methods for the preparation of aldehydes yet discovered consists in the hydrogenation of the acid chlorides in boiling xylene or cumene with palladinised barium sulphate (5% of metal) or nickel as the catalyst. Examples are given, including the preparation of *p*-methylcarbonatobenzaldehyde,  $\text{CH}_3\text{O-CO-O-C}_6\text{H}_4\text{-CHO}$ , m.pt. 18.5° C., in 95% yield. (See further J. Chem. Soc., 1918, i., 300.)—J. C. W.

*Aldehydes; New method for the preparation of —. II. Synthesis of gallaldehyde.* K. W. Rosenmund and F. Zetzsche. Ber., 1918, 51, 594—602.

The carbomethoxy- and acetyl-derivatives of gallic acid are converted into the acid chlorides, these are reduced to the aldehydes by the new method (see preceding abstract), and then hydrolysed to gallaldehyde. This dissolves freely in water and alkalis, and is even more acidic than acetic acid. (See further J. Chem. Soc., 1918, i., 300.)—J. C. W.

*Mustard oil; Iodine value of —.* E. G. Raynes. Analyst, 1918, 43, 216—217.

The iodine values of various mustard oils were found to be as follows: Expressed mustard oil, 119.6 to 121.0; extracted black mustard oil, 114.4; extracted white mustard oil, 104.7 to 108.6. The values recorded previously by certain authors appear to be too low.—W. P. S.

*Volumetric determination of chlorine, bromine, cyanogen, and mercuric ions.* Votocek. See XXIII.

#### PATENTS.

*Aluminium; Preparing water-soluble compounds of — with organic acids.* H. Sefton-Jones, London. From J. A. von Wülfing, Berlin. Eng. Pat. 115,463, May 4, 1917. (Appl. No. 6379 of 1917.)

PREPARATIONS of aluminium acetate or formate, directly soluble in water, for pharmaceutical and technical use, are produced by evaporating to dryness solutions containing the salts named together with alkali or alkaline-earth salts of the same acids; e.g., a mixture of 224 parts of aluminium formate solution of 23° B. (sp. gr. 1.19) with a solution of 40 parts of magnesium formate, or 34 parts of sodium acetate, yields readily soluble products when evaporated to dryness *in vacuo*.  
—J. H. L.

*Metal albumin combinations; Manufacture of —.* O. Imray, London. From Soc. of Chemical Industry in Basle, Switzerland. Eng. Pat. 115,686, May 15, 1917. (Appl. No. 6943 of 1917.)

ALBUMIN obtained from a normal human or animal organ or tissue, or from the juices expressed therefrom, is so far freed from constituents, such as degradation products, soluble in water, that it no longer gives the ninhydrin reaction, and is then converted into metal combinations as described in Eng. Pat. 110,936 (this J., 1917, 1289) for albumin from modified or infected tissues. The preliminary extraction of soluble matters greatly enhances the therapeutic value of the products, enabling the latter to exert their specific action without injurious secondary effects. Details are given of the process as applied to normal lung, brain, liver, and thyroid tissues.—J. H. L.

*Halogen calcium compounds; Process of preparing non-deliquescent water-soluble —.* E. Ritsert, Frankfurt. Ger. Pat. 305,367, Sept. 16, 1915. Addition to Ger. Pat. 288,966 (this J., 1916, 423).

INSTEAD of combining calcium chloride with lactose, as in the original patent, calcium bromide or iodide is used, or halogen compounds of calcium are made to react with sucrose or levulose to obtain therapeutic preparations.—C. A. M.

*Dimethylaminophenyldimethylpyrazolone; Process for manufacturing readily soluble preparations of —.* Farbw. vorm. Meister, Lucius, u. Brühlung. Ger. Pat. 305,369, July 8, 1915.

*DIMETHYLAMINOPHENYLDIMETHYLPYRAZOLONE* (dimethylaminoantipyrine) is soluble in 18 parts of water, but by adding to it an equivalent quantity of ethylurethane a preparation soluble in an equal weight of water is obtained. In this way it is possible to obtain a preparation suitable for subcutaneous injection, the urethane being harmless and only increasing the sedative action.—C. A. M.

*Ethanoltrialkylarsonium hydroxides and their salts; Process of preparing —.* Chem. Werke Grenzach A.-G., Grenzach. Ger. Pat. 305,772, Jan. 25, 1916.

THERAPEUTICALLY active compounds are obtained by the interaction of glycolhalogen-hydrins with trialkylarsines.—C. A. M.

*Acetic aldehyde; Manufacture of —.* H. Dreyfus, Basle, Switzerland. Eng. Pat. 115,899, May 22, 1917. (Appl. No. 7378 of 1917.)

A SILICON-IRON alloy (containing *c.g.* 12 to 17% Si) is resistant to boiling dilute sulphuric acid and does not amalgamate with mercury, and is therefore suitable for the construction of apparatus for the manufacture of acetaldehyde as described in Eng. Pat. 105,064 (this J., 1918, 222 A).—L. A. C.

*Quinine group; Therapeutically-valuable compounds of the —.* P. Hüssy, Assignor to Soc. of Chem. Industry in Basle, Switzerland. U.S. Pat. 1,261,235, Apr. 2, 1918. Date of appl., May 23, 1917.

SEE Eng. Pat. 112,974 of 1917; this J., 1918, 222 A. The compound from quinine and diallylbarbituric acid melts at 128° C., is sparingly soluble in water, easily soluble in ethyl and methyl alcohols and in ethyl acetate, and insoluble in petroleum spirit.

*Paraffinum liquidum; Process of making —.* H. G. C. Fairweather, London. From H. V. Dunham, Brattleboro, Vermont, U.S.A. Eng. Pat. 116,418, Sept. 4, 1917. (Appl. No. 12,672 of 1917.)

SEE U.S. Pat. 1,240,792 of 1917; this J., 1917, 1125.

*Halogen products of hydrocarbons; Apparatus for manufacturing —.* B. S. Lacy, Seward, N.J., Assignor to The Roessler & Hasselacher Chemical Co., New York. U.S. Pat. 1,263,906, Apr. 23, 1918. Date of appl., Mar. 31, 1917.

SEE Eng. Pat. 101,708 of 1916; this J., 1917, 1063.

*Electrolysis of chlorides [and production of chlorohydrocarbons]. Manufacture of alkalis [and chlorohydrocarbons].* U.S. Pats. 1,264,535 and 1,264,536. See VII.

*Method of producing catalysts for reduction or hydrogenation purposes and apparatus therefor.* Eng. Pat. 107,004. See XII.

*Surgical dressing [from beeswax]. Product of beeswax and process of manufacturing the same.* U.S. Pats. 1,264,185 and 1,264,186. See XII.

*Process for obtaining an extract of proteolytic enzymes.* Eng. Pat. 106,504. See XVIII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### PATENT.

*Colour photography.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,261,542, Apr. 2, 1918. Date of appl., July 19, 1916.

A FILM pack for use in three-colour photography comprises three films sensitised to different parts of the spectrum, the outer ones facing one another and the centre one being on a thin support; the first film is coloured to act as a filter for the other two and the second one acts as an additional filter for the third one. For example, the first film is blue-sensitive and is stained yellow, the second film is green-sensitive on a celluloid support and faces the third, red-sensitive film. The centre film may have a transparent coating on its back to give adherent contact with the first film.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

### PATENT.

*Pyrotechnic article and method of making same.* A. Jedel, New York. U.S. Pat. 1,265,205, May 7, 1918. Date of appl., Apr. 23, 1915.

FINELY powdered potassium chlorate, strontium nitrate, and a fusible gum insoluble in water, are mixed, placed in a mould, and heated to melt the gum and agglomerate the material into a briquette.—W. F. F.

## XXIII.—ANALYSIS.

*Thermo-regulator for apparatus filled with a constant water-level.* J. Milbauer. Z. anal. Chem., 1918, 57, 162—164.

THE overflow water passes into one limb of a U-tube containing mercury, the other limb containing a floating valve controlling the gas supply. Should the supply of water be interrupted, the float falls and cuts off the supply of gas to the burner. (See also J. Chem. Soc., Aug., 1918.)

—W. P. S.

*Gas-holder; Universal —.* J. Milbauer. Z. anal. Chem., 1918, 57, 161—162.

THE gas-holder described is constructed so that the gas contained therein is delivered at any desired constant rate and pressure. (See also J. Chem. Soc., Aug., 1918.)—W. P. S.

*Refractometer; A differential —.* G. A. Shook. J. Ind. Eng. Chem., 1918, 10, 553—554.

AN instrument of the Abbé type, so constructed that two liquids may be examined simultaneously.

*Acidimetry of coloured solutions: an application of the pocket spectroscope.* A. Tingle. J. Amer. Chem. Soc., 1918, 40, 873—879.

IN using the spectroscopic method of determining the end-point in acidimetric titrations (this J., 117 T), the edge of the absorption band nearer the red end of the spectrum is usually the sharper. With cochineal (25 c.c. of a saturated solution in 50% alcohol) the band extends over nearly the whole of the green on neutralisation. With methyl orange the end-point must always be reached by adding alkali to acid and not *vice versa*, and the indicator must be neutral. As usually prepared it



contains free acid. A neutral indicator is prepared by moistening with dilute hydrochloric acid, evaporating, moistening with dilute ammonia, again evaporating, and dissolving in water; 1 c.c. of a 0.1% solution is used. Lacmoid does not give a very sharp end-point, neutrality being marked by a total suppression of the spectrum. With phenolphthalein (0.2 c.c. of a 1% solution), a new band is developed in the green, closely bordering on the yellow, one edge almost coinciding with the "D" line. Rosolic acid (0.2 c.c. of a 1% solution) is well suited to spectroscopic observation, the absorption band broadening abruptly after neutrality is reached. The end-point in the titration of sulphuric acid in presence of copper sulphate, *e.g.*, in electrolytic baths, can be easily determined by the spectroscopic method.

*Anions; Detection of* —. F. Feigl. Z. anal. Chem., 1918, 57, 135—138.

THE following procedure is proposed for the identification of various acids in a mixture. The substance is boiled with concentrated sodium carbonate solution, or fused with a mixture of sodium and potassium carbonates, the solution is filtered, nearly neutralised with nitric acid, warmed with an excess of solid zinc nitrate, and filtered. The precipitate may contain zinc sulphide, sulphite, phosphate, borate, fluoride, ferrocyanide, ferricyanide, cyanide, molybdate, vanadate, and tungstate. These are identified by drop reactions with various reagents, portions of the precipitate being transferred to a piece of filter paper and treated with a drop of the following reagents:—Acid lead nitrate solution, brown coloration (sulphide); sodium nitroprusside, red coloration (sulphite); acid ferrous sulphate solution, blue-green coloration (ferrocyanide); ferric chloride solution, blue coloration (ferricyanide); stannous chloride solution, yellow or brown coloration (molybdate or tungstate); if sulphides and sulphites are absent, a black coloration with mercurous nitrate indicates cyanide, but if sulphide or sulphite is present, a portion of the precipitate is treated with dilute sulphuric acid in a crucible, the latter is covered with a piece of filter paper moistened with ammonium sulphide, and heated; the filter paper is then treated with a drop of acid ferric chloride solution, when a red coloration indicates the presence of cyanides. Borate is detected by the tumeric reaction and fluoride by the etching test. The filtrate may contain zinc thiocyanate, chloride, bromide, iodide, sulphate, thiosulphate, and sulphite. Portions of the filtrate are tested with the usual reagents for thiocyanate, iodide, bromide, sulphate, and sulphite. Another portion of the solution is then treated with lead nitrate, boiled, and filtered; the precipitate gives a brown coloration with silver nitrate if thiosulphate is present. The filtrate is then treated with nitric acid and silver nitrate, and filtered; chloride and bromide may be identified in the precipitate, and chlorate in the filtrate.—W. P. S.

*Chlorine, bromine, cyanogen, and mercuric ions; Volumetric determination of* —. E. Votocek. Chem.-Zeit., 1918, 42, 257—260, 271—272.

CHLORIDES in a solution rendered slightly acid with nitric acid may be titrated with standardised mercuric nitrate solution, using 0.06 gm. of sodium nitroprusside as indicator; the end-point of the titration is denoted by the formation of a white turbidity of mercuric nitroprusside. Sulphates, phosphates, and chlorates do not interfere, but the solution must not contain sulphites or nitrites. The results obtained are accurate and the method may be used for the titration of very small quantities of chlorides. Bromides, but not iodides or

fluorides, may also be titrated in the same way with mercuric nitrate solution, and mercury salts and mercury in organic combination, after these have been converted into mercuric sulphate, can be titrated with sodium chloride solution. The sodium nitroprusside is added to the solution of the mercury sulphate and the sodium chloride solution is run in until the turbid solution becomes clear. Cyanides may also be titrated with mercuric chloride solution. Mercury may be separated from copper by means of sodium nitroprusside. A solution containing cupric and mercuric salts is treated with an excess of sodium chloride and sodium nitroprusside is added; the copper is precipitated as cupric nitroprusside whilst the mercury compound remains in solution. If the sodium chloride is added in the form of a standard solution, the mercury may be determined by titration after the copper compound has been removed by filtration. —W. P. S.

*Carbon; Determination of* — by the wet method. W. Stepp. Biochem. Zeits., 1918, 87, 135—142.

THE flask in which decomposition takes place is connected with a condenser to hold back the acid. Purification of the sulphuric acid is accomplished by heating the strong acid first with potassium bichromate and afterwards with permanganate. It is difficult to obtain the potassium bichromate, used in destroying the organic matter, free from carbonate. The author prefers a mixture of chromic and sulphuric acids. Finally, when oxidation with the mixture of sulphuric and chromic acids has proceeded for four hours, the apparatus is allowed to cool in a current of air free from carbon dioxide, 10 c.c. of 5% solution of potassium permanganate is added, and the mixture is heated for another hour. (See also J. Chem. Soc., Sept., 1918).—S. B. S.

*Carbonates and bicarbonates; Determination of* —. Use of litmus and phenolphthalein as indicators. W. Mestrezat. Bull. Soc. Chim., 1918, [iv.], 23, 250—254.

IN the titration of carbonates in cold solutions, using phenolphthalein as indicator, a satisfactory and correct end-point is obtained when the whole of the carbonate is just converted into bicarbonate, if the alkaline liquid is diluted so as not to contain more than 0.07% of sodium carbonate. Similarly total alkalinity may be determined by titration of the cold solution, using litmus as indicator, if the following procedure is adopted. The standard acid is added 1—2 c.c. at a time until a change in the tint of the indicator is just noticeable; then between each successive addition of 0.2—0.3 c.c. of standard acid the liquid is thoroughly mixed and divided into two parts, one part being used as a control and the standard acid added to the other. The end-point is attained when the addition of acid no longer causes a change in tint.

—W. G.

*Strontium; Detection of* — in the presence of barium by means of calcium sulphate. T. P. Raikow. Z. anal. Chem., 1918, 57, 164—170.

THE solution containing strontium and barium is shaken for 15 secs. with an excess of calcium sulphate solution and then filtered. If strontium is present a turbidity develops in the filtrate; with small quantities of strontium the filtrate may have to be boiled before the turbidity is observed. In cases where only very small quantities of strontium are present the latter may be precipitated together with the barium sulphate; the precipitate should, therefore, be washed several times with boiling water and the filtrate thus obtained tested with barium chloride solution to detect the presence of strontium sulphate dissolved out by the hot water. —W. P. S.

*Hydrofluoric acid; Use of — in analysis.* N. H. Furman. J. Amer. Chem. Soc., 1918, 40, 895—906.

By the use of an acid fluoride solution, copper may be quantitatively separated from vanadium either by electro-deposition or by the action of hydrogen sulphide. The behaviour of vanadium under these circumstances closely resembles that of tin, tungsten, uranium, and titanium. Acid fluoride solutions of stannic tin give none of the characteristic reactions of stannic tin (see page 413 A) and this fact may be utilised in the analysis of tin alloys. The alloy is dissolved in hot concentrated sulphuric acid and the cold solution poured into water containing hydrofluoric acid. Lead sulphate is precipitated and the filtrate contains copper, antimony (trivalent), stannic tin, and a trace of lead. This is treated with hydrogen sulphide when copper, antimony, and lead are completely precipitated. The filtrate containing the tin is evaporated in a platinum dish until the hydrofluoric acid is removed and the sulphuric acid has begun to fume. The sulphuric acid solution is then poured into a large volume of water, the stannic hydroxide salted out, filtered off, and weighed as stannic oxide. By the addition of boric acid to an acid fluoride solution of stannic tin, the ordinary chemical reactions of the metal are developed, and from such a solution the tin may be quantitatively separated either by the action of hydrogen sulphide or by electro-deposition. Silica, in the form of sodium silicate, produces the same effect as boric acid. The change in the behaviour of acid fluoride solutions of stannic tin on the addition of excess of boric acid is made use of in the separation of the metals of the tin group. A further effect produced by the addition of boric acid is that the acid fluoride solutions have a greatly reduced action on the glass of the containing vessels. (See also J. Chem. Soc., Aug., 1918.)—H. M. D.

*Copper; Determination of — by potassium thiocyanate, potassium iodide, and thiosulphate.* G. Bruhns. Chem.-Zeit., 1918, 42, 301—302.

The neutral or slightly sulphuric acid solution of the copper salt is treated with potassium iodide and then titrated with thiosulphate solution containing an excess of potassium thiocyanate, using starch solution as indicator, or the copper solution may be titrated with thiosulphate solution containing both potassium iodide and thiocyanate. The use of the latter enables a smaller quantity of potassium iodide to be used, since the cuprous iodide formed reacts with the thiocyanate, yielding cuprous thiocyanate and potassium iodide which reacts with a further quantity of cupric salt. Lead does not interfere with the reaction, but silver and mercury must not be present. The influence of ferric salts, if present in small quantity, may be prevented by the addition of oxalic acid; ferrous salts have no effect.—W. P. S.

*Copper; Titration of — with potassium cyanide.* M. P. Appleby and K. W. Lane. Analyst, 1918, 43, 268.

In the usual method of titrating copper with potassium cyanide, a sharp end-point is not obtained when the concentration of the copper is much less than 1 gram. per litre. Smaller quantities may be accurately titrated in the form of double carbonates of alkali and copper in presence of excess of sodium carbonate. The copper solution is added to a warm solution containing about 70 grms. of anhydrous sodium carbonate and 50 grms. of sodium bicarbonate per 500 c.c. A standard solution of copper is prepared by dissolving about

0.1 gram. of copper in dilute acid, and adding it slowly, and with constant stirring, to 500 c.c. of the solution of carbonate and bicarbonate. This solution is used for standardising the cyanide solution, as in the ammonia method. The end-point of the reaction is shown by the colour of the solution changing from deep blue, through bluish-purple to grey. (See also J. Chem. Soc., Aug., 1918.)—C. A. M.

*Mercury; New method of estimating — by means of zinc filings.* M. François. Comptes rend., 1918, 166, 950—952.

The mercury salt, in a finely divided state, mixed with 0.5 gram. of potassium iodide, is treated with 1 gram. of zinc filings and 10 c.c. of 2 N sulphuric acid. At the end of half-an-hour a further quantity of zinc and acid is added, and this is repeated at the end of the hour. After 24 hours the liquid is decanted through a filter and the residue washed four times by decantation with water. To it is then added, in portions of 5 c.c. each, 25 c.c. of hydrochloric acid, diluted with its own volume of water, and after 24 hours this is decanted and 25 c.c. of fuming hydrochloric acid is added. After 24 hours the zinc has all dissolved and the mercury has collected into one large globule, which is washed by decantation with water, transferred to a capsule, dried first with filter paper and finally over sulphuric acid in the cold, and weighed. The method is accurate and applicable to all mercury salts, whether soluble or insoluble, except the sulphide. It can be applied to cinnabar if this is first oxidised by 10 c.c. of a solution of bromine (50 c.c.) in a mixture of fuming hydrobromic acid (50 c.c.) and water (50 c.c.), and after 24 hours diluted with 30 c.c. of water, and the free bromine removed by the addition of three lots of zinc filings (1 gram. each) at intervals of half-an-hour, prior to the ordinary treatment with zinc and sulphuric acid.—W. G.

*Nickel; Determination of — with  $\alpha$ -benzildioxime.* R. Strebing. Chem.-Zeit., 1918, 42, 242—243.

Precipitation of nickel with  $\alpha$ -benzildioxime and weighing of the dried precipitate (Atack, this J., 1913, 769) gives trustworthy results when dealing with small quantities of the metal. If, however, the amount of the latter exceeds 0.025 gram., the results obtained are too high; a larger quantity of the reagent is required, and owing to its small solubility a portion is thrown out of solution and occluded in the nickel precipitate. To determine relatively large quantities of nickel, the slightly ammoniacal solution of the nickel salt is treated with a small excess of a 0.05% alcoholic  $\alpha$ -benzildioxime solution, the mixture heated on a water-bath for 10 mins., filtered while hot, the precipitate washed with hot water, and ignited. The residue of oxide obtained is moistened with nitric acid, again ignited, and weighed.—W. P. S.

*Chromium; Estimation of — in presence of iron.* K. Schorlemmer. Collegium, 1918, 145—149.

The presence of iron in solutions of chromium salts causes error in the ordinary determination of chromium by thiosulphate. To obviate this, the solutions of chromium salts are oxidised with hydrogen peroxide, allowed to cool, and re-oxidised with a fresh addition of peroxide. The ferric hydroxide is filtered off, dissolved in acid, twice oxidised with peroxide, again filtered off, the filtrate added to the first filtrate, and the total chromium determined in the mixture by means of thiosulphate. If much iron is present, the treatment of the ferric hydroxide must be repeated and the filtrate added to the other two.—D. W.



*Manganese; Influence of impure zinc oxide on the determination of —.* M. Muller. Stahl u. Eisen, 1917, 37, 287. Rev. Mét., 1918, 15, Extr., 172.

THE purity of the zinc oxide used may appreciably affect the estimation of manganese. The following tests are necessary to reveal the impurities contained. To detect heavy metals, lime and magnesia, 1–2 grms. of the zinc oxide is dissolved in 10–20 c.c. of dilute acetic acid (sp. gr. 1.040). The solution should be quite clear and should still remain clear and colourless after the addition of 20 c.c. of dilute ammonia. It should not change on the addition of ammonium oxalate or sodium phosphate, and any precipitate formed by hydrogen sulphide should be pure white. To detect nitrates 1 gm. is dissolved in 10 c.c. of dilute acetic acid. 2 or 3 drops of an indigo solution (1:1000) and 10 c.c. of concentrated sulphuric acid are added, and the solution shaken well. The blue coloration should not be changed. Arsenic is detected by treating 1 gm. of the oxide with 5 c.c. of stannous chloride, the mixture being well agitated. The colour should not deepen after waiting for 1 hour. To test the indifference of the oxide to potassium permanganate, 30 c.c. of dilute nitric acid is concentrated to 5–10 c.c. and cooled. A paste of the zinc oxide made with cold distilled water is then added until a small excess remains. The mixture is made up to 300 c.c., shaken, and filtered. 160 c.c. of the filtrate is heated to boiling and titrated with permanganate.—F. C. Th.

*Arsenic; Simple process for the determination of small quantities of — in dead bodies.* H. Führer. Ber. deuts. Pharm. Ges., 1918, 23, 221–229.

THE organic matter is destroyed by mixing 10 grms. of the material with 5 grms. of potassium permanganate and 10 c.c. of sulphuric acid (1:5), and evaporating to dryness on the water-bath. Then 25 c.c. of concentrated sulphuric acid is added, and also hydrogen peroxide solution, until the mixture is decolorised. It is then transferred to a Kjeldahl flask, 30 c.c. of sulphuric acid is added, and the liquid is heated to boiling over a free flame and then reduced with 5 grms. of ferrous sulphate. When cold, 50 grms. of sodium chloride is added, and the liquid is distilled into 50 c.c. of 1% potassium iodide solution in a conical flask, the mouth of which is protected by a tube filled with glass beads moistened with some of the potassium iodide solution. After distillation, 0.5 gm. of crystallised zinc chloride is added to the distillate and then 10 grms. of zinc. To the mouth of the flask a long upright tube is fitted, packed in the lower part with lead acetate paper and provided at the upper end with a long strip of paper which has been immersed in a solution of mercuric bromide and dried. At the end of an hour, the strip of paper is taken out, and the colour produced compared with a series of standard strips corresponding to from 0.005 mgrm. to 0.5 mgrm. of  $As_2O_3$ .—J. H. J.

*Marsh's apparatus [for detection of arsenic].* W. Kirkby. Pharm. J., 1918, 100, 286.

IN using Marsh's apparatus for testing for arsenic risk of explosion may be obviated by interposing a tube loosely packed with cotton-wool between the bottle and the hydrogen jet.—C. A. M.

*Molybdenum; Determination of —.* O. Binder. Chem.-Zeit., 1918, 42, 255.

IN order to convert molybdenum sulphide completely into molybdic acid, so that the latter can be weighed, the ignited sulphide should be treated with a few drops of concentrated nitric acid, then dissolved in ammonia, re-precipitated with nitric

acid, evaporated, and ignited. The pure white molybdic acid thus obtained is weighed, dissolved in ammonia, and any small quantity of insoluble matter separated and deducted from the weight.

—W. P. S.

*Plant ashes; Factors affecting the composition of — with special reference to tobacco.* O. D. Roberts. Analyst, 1918, 43, 254–259.

ASH obtained by the usual methods of determining the mineral matter in plants is, as a rule, deficient in carbon dioxide, and to obtain comparable results it is necessary to determine carbon dioxide in the ash and to calculate the results upon an ash free from that constituent and from unburnt carbon. Part of the inorganic chlorine in tobacco is lost during the ignition, whilst the amount of sulphate left in the ash is in excess of that originally present as sulphate in the plant. Hence it is inaccurate to assume that the amounts of sulphate and chlorine in the ash represent the total quantity of sulphur and chlorine present in the plant. To determine the inorganic salts the plant should be extracted with water or dilute acids and the salts determined in the extracts, whilst organic chlorine, sulphur, and phosphorus may be obtained by the difference between the results for the total and the inorganic constituents.—C. A. M.

*Gas analysis with very small quantities.* L. Hamburger and W. Koopman. Z. anal. Chem., 1918, 57, 121–135.

AN apparatus is described by means of which very small quantities of gaseous mixtures may be analysed, and in which, during the analysis, the gas comes in contact only with glass, platinum, and mercury. The gas is freed from moisture and carbon dioxide by cooling with a mixture of solid carbon dioxide and alcohol and with liquid air respectively, then burnt by means of a platinum spiral, after addition of oxygen or of hydrogen if necessary; excess of unburnt oxygen is determined by adding hydrogen and again burning, the use of an iron spiral having proved unsatisfactory. A capillary tube is used for the combustion, as if this be effected at a low pressure combination of nitrogen and oxygen and of oxygen and mercury vapour may occur and lead to incorrect results. The use of automatic pumps of the Sprengel type for transferring the gas from one part of the apparatus to another is also inadmissible, as gas occluded by the mercury is liberated when the pressure falls very low and may pass into the measuring tube. The use of Töpler pumps overcomes this difficulty. Results obtained with quantities of gas of the order of 10 cb.mm. are given. In the case of a mixture of  $H_2$  26.7, CO 22.7,  $CH_4$  22.9, and  $N_2$  27.8%, the values found were  $H_2$  26.4, CO 21.1,  $CH_4$  24.2, and  $N_2$  28.3%.

*Determination of unsaturated hydrocarbons in gasoline.* Dean and Hill. See IIa.

*Differentiation of soda and sulphite pulps.* Klemm. See V.

*Gasometric determination of nitrates.* Hill. See VII.

*Analysis of spent oxide.* Hottenroth and others. See VII.

*Detection of iodides in presence of cyanides.* Curtman and Kaufman. See VII.

*Reaction between the alkali phosphates and magnesium chloride.* Balareff. See VII.

*Rapid determination of carbon in steel.* Jackson. See X.

*Determination of sulphur in iron and steel.* Pinsl. See X.

*New thermo-electric effect [in metals].* Benedicks. See X.

*Approximate determination of the minerals in [ore] concentrates.* Thomas and Apgar. See X.

*Identification of molybdenite.* Fuchs. See X.

*Determination of oil in seeds.* Gray. See XII.

*Analysis of sulphonated oils.* Schultz. See XII.

*Acidity of chrome tanning liquors.* Thomas and Baldwin. See XV.

*Determination of lime and phosphoric acid in peat soils. Comparison of Jönköping with Bremen method.* Rost and Clapp. See XVI.

*A modification of the Selivanoff reaction [for the detection of ketoses and sucrose.]* Weehuizen. See XVII.

*Additive factors for the calculation of fat in milk from the specific gravity and total solids.* Harris. See XIXa.

*Conditions affecting accuracy in the Gerber test [for milk].* Day and Grimes. See XIXa.

*Determination of shell in cocoa and cocoa products.* Baker and Hulton. See XIXa.

*Determination of albuminoid ammonia in liquids containing gas liquor.* Stephenson. See XIXa.

*Determining morphine in opium.* Hill. See XX.

*Effect of codeine in hindering precipitation of morphine by ammonia from a solution of its lime compound.* Annett and Sligh. See XX.

*Colorimetric determination of brucine in presence of strychnine.* Wöber. See XX.

*Nucleic acid and its analytical examination.* Chapman. See XX.

*Acetylsalicylic esters.* Astruc. See XX.

*Microchemical tests for choline.* Schoorl. See XX.

*Catalytic reduction of organic halogen compounds.* Rosenmund and Zetzsche. See XX.

#### PATENTS.

*Thermo-elements having copper sulphide as the positive constituent; Process for the manufacture of —.* J. Marshall, Dresden, Germany. Eng. Pat. 103,671, Jan. 25, 1917. (Appl. No. 1277 of 1917.) Under Int. Conv., Dec. 9, 1915.

A SMALL proportion (1–3%) of sulphur is added to the copper sulphide, before casting, to increase the mechanical strength and reduce the specific resistance without appreciably affecting the E.M.F. of the material. The contact points of the casting are surrounded by a layer of electro-deposited metal which serves as a support for the attachment of other metallic parts of the element.—W. E. F. P.

*Gas; Apparatus for analysing —.* Aktiebolaget Ingenjörssfirma F. Egnell, Stockholm. Eng. Pats. (A) 103,812, Sept. 27, 1916, and (B) 104,160, Sept. 28, 1916. (Appl. Nos. 13,749 and 13,800 of 1916.) Under Int. Conv., Jan. 29 and Feb. 12, 1916.

(A) In an apparatus applicable for analysing flue gases, the gas is drawn through a liquid seal into a measuring vessel by the withdrawal of liquid from the latter, and is forced out through another liquid

seal as the liquid again rises, to a pipe having two branches, one of which dips into an absorption receptacle containing caustic potash for absorbing carbon dioxide, and the other passes to a receptacle containing pyrophoric iron for absorbing oxygen. The outlet pipes from these two receptacles are combined and discharge into another measuring vessel. When a valve on the pipe leading to the absorbing receptacle for oxygen is closed, the gas passes only through the caustic potash to the second measuring vessel, so that the volume of carbon dioxide is obtained. When the valve is opened the caustic potash acts as a liquid seal, and the gas passes only through the oxygen-absorbing receptacle to the second measuring vessel thus indicating the volume of oxygen. In a modification, the outlet pipes from the two absorbing receptacles are not connected to the second measuring vessel, but an auxiliary receptacle is provided communicating with the receptacle containing caustic potash into which the liquid is forced, and the increase of pressure is measured to give an indication of the volume of gas absorbed. The first measuring vessel may also be divided into two similar vessels, from which the gas is passed to the two absorbing vessels respectively. In another modification, the control valve is provided on the pipe leading from the oxygen-absorbing vessel and controls the passage of gas from this receptacle to the measuring vessel, or to the absorbing vessel for carbon dioxide. Several other combinations of two or more absorbing receptacles are also described. (B) In apparatus of the type described in (A) in which the gas has to be chemically or physically treated before analysis, a constriction is provided in the gas inlet piping, or in the discharge pipe of the medium which causes the flow of gas into the first measuring vessel, so as to retard the flow of gas.—W. E. F. P.

*Gas calorimeter.* J. F. Simmance, London. Eng. Pat. 115,915, May 31, 1917. (Appl. No. 7813 of 1917.)

In a recording, constant flow gas calorimeter, the container through which the gas passes on its way to the burner is so constructed and adjusted that the gas issues from the burner orifice at a standard volume per unit time, irrespective of changes due to variations in temperature, pressure, and specific gravity. The gas on its way to the calorimeter may be by-passed through a wet meter, and the recording pen adjusted to correspond with the calorific value determined in the usual manner, the gauge-glass of the meter being connected, through a tap, with a water reservoir adapted to be raised or lowered, to facilitate adjustment of the water level. To ensure regularity of flow and constant pressure of water at the entrance to the calorimeter, the water supplied at constant head is consecutively exposed to the atmosphere in three or more vessels at successively lower levels, each forming a constant head, and the last being within the calorimeter. Provision is also made for saturating with aqueous vapour the air supplied for combustion, and for cutting off the gas supply automatically in the event of the flow of water being interrupted.

—W. E. F. P.

*Distillation; [Laboratory] apparatus for fractional —.* S. E. Bowrey, London. Eng. Pat. 115,504, May 15, 1917. (Appl. No. 6939 of 1917.)

In an apparatus for fractional distillation the supply of gas for heating is controlled by a thermo-regulator of known type containing mercury, actuated directly by the pressure of the vapour in the distillation column. The mercury tube of the regulator is sealed through the wall of the column and bent upwards inside, below the level of the side tube leading to the condenser. To ensure a sufficient pressure of vapour to actuate the regulator



properly, the passage of vapour up the column may be obstructed by a constriction above the level of the regulator tube but below that of the condenser tube; the portions of the column above and below the constriction may be connected externally by a reflux tube of suitable length, to allow any liquid condensed above the constriction to flow back into the lower part of the column.—J. H. L.

*Densimetric methods and appliances.* A. F. C. Pollard, Ardrossan. Eng. Pat. 115,662. (Appl. Nos. 6480, May 7, and 18,174, Dec. 7, 1917.)

IN order to indicate or record the density or temperature of a flowing liquid or to separate a flowing liquid into fractions of varying densities, the liquid is caused to flow through a movable system supported by hydraulic means on a fixed axis of rotation in such a manner that the supply and discharge of the liquid have no turning effect on the movable system, but the latter assumes a definite angular position of stable equilibrium which varies with and corresponds to the density and/or temperature of the liquid. A liquid of standard density of the same kind as, or having a coefficient of expansion approximately the same as, that of the other liquid, is caused to flow through the system in a similar manner in order to eliminate the effect of temperature on the density. The apparatus may be used in conjunction with a spirit still, and the effect of the varying density of the distillate on the angular position of the movable system may be utilised to collect fractions of different densities in separate receivers. Various forms of the apparatus are described and illustrated in detail.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Barron and Barron. Impact grinders. 11,059. July 5.  
 British Dyes, Ltd., Turner, and West. Tanks for transporting hydrochloric acid or other corrosive liquids. 11,781. July 18.  
 Fraser. Machines for disintegrating and screening pulverulent material. 11,730. July 17.  
 Hall. Grinding machinery. 11,970. July 23.  
 Hayhurst. Drying machines. 11,797. July 18.  
 Hill and Tuckett. Grinding apparatus. 12,009. July 23.  
 Levinstein, Oxley, and Levinstein, Ltd. Process for carrying out catalytic reactions. 12,027. July 23.  
 Lynde. Digesters. 12,034. July 24.  
 Mansfield. Filters for liquids. 11,155. July 8.  
 Morison. Condensing-plant. 11,242. July 9.  
 Preisig and Steiger. Drying finely-divided material. 10,998. July 4.  
 Stuart. Reclaiming material. 11,462. July 12. (U.S., Nov. 13, 1917.)  
 Sturgeon. Centrifugal separators. 11,818. July 19.  
 Techno-Chemical Laboratories, Ltd., and Testrup. Conducting high temperature reactions. 10,930. July 3.  
 Topham. Furnaces. 11,911. July 20.  
 Ward and Ward. Centrifugal separators. 11,392. July 11.  
 COMPLETE SPECIFICATIONS ACCEPTED.

18,409 (1916). Trotter. Apparatus for sorting or separating solid substances. (117,274.) July 24.

- 5004 (1917). Hamilton (Allan). See II.  
 5263 (1917). Carr. Method of drying liquid materials. (117,469.) July 31.  
 7467 (1917). Clifford, and Jones and Attwood. Separation and settlement of solids and semi-solids from chemical and like manufacturing liquids. (117,472.) July 31.  
 7589 (1917). Miller and Lloyd. Grinding or crushing machines. (117,281.) July 24.  
 7941 (1917). Roy, and Morson and Son. Method and means for bringing about chemical reactions between gases and/or vapours by means of catalysts. (117,103.) July 17.  
 9466 (1917). Barbet et Fils et Cie. See XVIII.  
 10,114 (1917). Ges. f. Linde's Eismaschinen A.-G. See VII.  
 11,059 (1917). British Dyes, Ltd., Turner, and Crosland. Grinding mills, disintegrators, mixers, etc. (116,958.) July 10.  
 12,072 (1917). Fritzweiler and others. See X.  
 12,475 (1917). Wood. Centrifugal apparatus for extracting dust from air or gases. (117,173.) July 17.  
 12,592 (1917). Brand. Furnace rakes for mechanical roasting-furnaces and the like. (116,974.) July 10.  
 16,592 (1917). Benjamin. Continuous tunnel kilns. (117,383.) July 24.  
 16,976 (1917). Leitch. Apparatus for solidifying or concentrating liquid or semi-liquid materials on a revolving cylinder. (117,560.) July 31.  
 18,239 (1917). Wurth. Charging shaft-furnaces. (117,024.) July 10.  
 1734 and 4993 (1918). Simon-Carves, Ltd., and Hunter. Settling-tanks for clarifying liquids. (117,576 and 117,586.) July 31.  
 3385 (1918). Stokes. Incinerating-furnaces. (117,417.) July 24.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

- American Linseed Co. Artificial fuel and methods of making same. 11,741. July 17. (U.S., July 17, 1917.)  
 American Linseed Co. Apparatus for producing artificial fuel, etc., from plastic or viscous material. 11,742. July 17. (U.S., July 17, 1917.)  
 Campion. Gas-producers. 11,969. July 23.  
 Coke Oven Construction Co., Colquhoun, and Marr. Coke ovens, etc. 11,256 and 11,257. July 9.  
 Davies. Distillation, gasification, etc., of carbonaceous material. 10,895. July 3.  
 Davies. Carbonisation of wood, etc. 11,898. July 20.  
 Davies. Carbonisation of carbonaceous materials. 11,602. July 16.  
 Dempster and Sons, and Toogood. Distillation of coal. 11,610. July 16.  
 Flood and Flood. Quenching coke. 10,914. July 3.  
 Holt. Manufacturing gas from coal, etc. 10,972. July 4.  
 James. Burning-fuel. 11,012. July 4.  
 Johnson (Houghton and Co.). Manufacture of cutting oils. 11,581. July 15.  
 Lamplough. Converting heavier hydrocarbons into lighter hydrocarbons. 11,808. July 18.  
 Leadbeater. Treating peat to render it more adaptable as fuel. 10,823. July 2.  
 Lloyd. Preparation of fuel oil from dehydrated tar. 12,102. July 25.  
 McCaffery. Generating gaseous fuel for heating or firing furnaces. 11,989. July 23.  
 Macrae and Topp. Manufacture of fuel from peat, lignite, etc. 12,040 and 12,041. July 24.  
 Milbourne. Gas purifiers. 11,453. July 12.

Mitchell. Furnaces for treating briquetted fuel. 10,813. July 2.  
 Oddy and Smith. Distillation of coal, shale, peat, wood, etc. 11,356. July 11.  
 Oddy and Smith. Purifying or refining hydrocarbons. 11,357. July 11.  
 Oddy and Smith. Light hydrocarbons for motor and lighting purposes. 11,358. July 11.  
 Oré. Purification of coal gas and manufacture of iron salts used in same. 11,677. July 16.  
 Parkes and Pearson. 11,397. *See XX.*  
 Pease. Heat treatment of coal to obtain oils, gas, ammonia, etc. 12,207. July 26.  
 Rew. Manufacture of gas. 11,318. July 10.  
 Spicer (Wells and Wabba). Gas-producing and gas-purifying devices. 11,309. July 10.  
 Wade (Pittsburgh Testing Laboratory). Process of regenerating lubricants. 11,402 and 11,407. July 11.  
 Wallace. Utilisation of oil, etc., shales. 12,250. July 27.  
 West and Wild. Retorts for destructive distillation of coal, etc. 11,738. July 17.  
 Wolstencroft. Utilisation of waste material for artificial fuel. 10,953. July 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9178 (1916). Cassal, Cassal, and Gerrans. Treatment of liquid hydrocarbon. (117,087.) July 17.  
 5004 (1917). Hamilton (Allan). Continuous fractionation and dephlegmation of petroleum or other hydrocarbon distillates or mixtures of volatile liquids. (117,277.) July 24.  
 9485 and 9487 (1917). Jackson (Seaman Waste Wood Chemical Co.). Distillation of wood, etc. (116,939 and 116,940.) July 10.  
 10,224 and 12,013 (1917). Helps. Manufacture and treatment of coal or like gas. (117,308.) July 24.  
 15,654 (1917). Forward. Production of motor fuel oil by distilling petroleum. (117,372.) July 24.  
 17,165 (1917). Hemingway. *See VII.*

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Ambler, Gibbs, and Selden Co. Production of sulphonic acids. 11,196. July 8.  
 Cooper. Preparation of  $\beta$ -naphthol. 11,896. July 20.  
 Gibbs, Lewis, and Selden Co. Purification of anthraquinone. 11,072. July 5.  
 Lewis. Production of monochlorobenzol. 11,706. July 17.  
 Lloyd. 12,102. *See II.*  
 Lord. Obtaining products from blast-furnace pitch. 11,745 and 12,193. July 17 and 26.  
 Valentine. Apparatus for dehydration and fractional distillation of coal tar, etc. 11,163. July 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

4430 (1917). Cooper and Edwards. Production of high-boiling phenols. (116,905.) July 10.  
 5004 (1917). Hamilton (Allan). *See II.*  
 9101 (1917). Imray (Soc. Chem. Ind. in Basle). Manufacture of derivatives of *p*-aminophenol. (116,920.) July 10.  
 16,653 (1917). Ellis and others. *See XIII.*

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Brotherton and Co., Ehrhardt, and Kay. Manufacture of black sulphur colours. 11,215. July 9.  
 Holliday and Co., and Law. Chlorination of purpurin, etc. 10,787. July 2.  
 Holliday and Co., and Law. Chlorination of allizarin and its derivatives. 10,788. July 2.  
 Imray (Soc. Chem. Industry in Basle). Orthoxy-azo dyestuffs, chromium compounds, and process of dyeing therewith. 11,458. July 12.

Marks (Sunbeam Chemical Co.). Universal dyes and method of producing same. 11,078. July 5.  
 Soc. L'Air Liquide. Manufacture of a blue sulphurised dye. 11,949. July 22. (Fr., July 31, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

200 (1917). Oman. Preparation of azo colouring matters from lignosulphonic acid or its salts. (103,480.) July 31.  
 4856 (1917). Malcolmson and Fitzgerald. Manufacture of dyestuffs and process of dyeing therewith. (117,095.) July 17.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Auchinachie. Treatment of cardboard to render it impermeable. 12,052. July 24.  
 Bacon and Sindall. Treatment of straw, bracken, etc., to manufacture paper pulp. 11,945. July 22.  
 Barr. 10,862. *See XIII.*  
 Bindley and Duleken. 11,759. *See XIII.*  
 Dreyfus. Manufacture of cellulose derivatives. 11,872 and 11,873. July 20.  
 Dreyfus. Manufacture of dopes, solutions, or preparations having a basis of cellulose acetate. 12,085. July 24.  
 Dunville and Co., and Gilmour. Production of solvents for cellulose acetates and nitrates, including celluloid, and their use in the production of varnishes, lacquers, and dopes. 10,949. July 4.  
 Flatters. Rendering fabrics gas- and water-proof. 11,607. July 16.  
 Flatters. Process for doping and fireproofing fabrics. 11,608. July 16.  
 Hlggins. Manufacture of paper from seaweed. 11,561. July 15.  
 Johnston and Sutherland. Degumming and cleansing or bleaching leaf and stalk fibres. 11,506. July 13.  
 Lancaster. Treating paper or cloth articles with paraffin waxes, etc. 11,767. July 18.  
 McEwen. Treatment of flax, etc. 12,113. July 25.  
 Milne. Paper-making machines. 10,985. July 4.  
 Partington and Roberts. Pulp for making paper, etc. 11,282. July 10.  
 Peachey. 11,267. *See XIII.*  
 Schweizerische Versuchsanstalt für Textilindustrie. Testing textile, etc., materials. 11,303. July 10. (Switz., Mar. 13.)  
 Soc. Nauton Frères et de Marsac, and Tesse. 11,007. *See XIII.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

200 (1917). Oman. *See IV.*  
 7569 (1917). Davies. Fourdrinier paper machines. (107,579.) July 31.  
 805 (1918). Schmid. *See XII.*  
 3235 (1918). Rafsky. Coated paper. (117,414.) July 24.  
 5958 (1918.) Galloway and Stratton. Apparatus for colouring paper. (117,428.) July 21.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Almsworth and Mather. Winding and dyeing machines. 11,427. July 12.  
 Clavel. Dyeing, bleaching, etc., cops. 11,665. July 16. (Ger., July 20, 1917.)  
 Imray (Soc. Chem. Ind. in Basle). 11,458. *See IV.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

4856 (1917). Malcolmson and Fitzgerald. *See IV.*  
 566 (1918). Glaister and Hoyle. Dyeing textile fabrics, and apparatus therefor. (117,039.) July 10.



# VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Bousfield, and Nobel's Explosives Co. Manufacture of liquid nitrogen peroxide and nitric acid. 10,869. July 2.

Bracq. Pyrites furnaces. 11,020. July 4. (Fr., Feb. 22, 1917.)

British Dyes, Ltd., Turner, and West. 11,781. See I.

British Thomson-Houston Co. (General Electric Co.). 12,195. See X.

Dutt and Dutt. Manufacture of sulphur, alkaline earth carbonates, and alumina. 11,293. July 10.

Fitzgerald. Preparation and use of dense poisonous gases other than chlorine for military purposes. 11,570. July 15.

Jephcott. 11,637. See XX.

Norsk Hydro-Elektrisk Kvaestofaktieselskab. Production of ammonia from cyanamide. 12,000. July 23. (Norway, Oct. 4, 1917.)

Norsk Hydro-Elektrisk Kvaestofaktieselskab. Production of alumina compounds from clay, argillite, etc. 12,001. July 23. (Norway, Sep. 25, 1917.)

Norton Co. Purifying aluminous materials. 11,378. July 11. (U.S., Aug. 18, 1917.)

Norton Co. Producing crystalline alumina. 11,379. July 11. (U.S., Aug. 18, 1917.)

Norton Co. 11,380. See VIII.

Oré. 11,677. See II.

Parkes and Pearson. Fixation of nitrogen of atmosphere. 11,192. July 8.

Patentaktiebolaget Jungner. 11,369. See IX.

Pease. 12,207. See II.

Perrin. Concentration of sulphuric acid. 11,021. July 4. (Fr., May 24, 1917.)

Pratt Engineering and Machine Co. Sulphur burning. 11,531. July 13. (U.S., July 14, 1917.)

Purex, Ltd., and Wilson. 10,978. See XIII.

Roge. Recovery of acid from acid sulphate. 11,253. July 9.

Silberrad. Manufacture of nitrates. 11,292. July 10.

Techno-Chemical Laboratories, Ltd., and Testrup. Production of cyanides. 10,931. July 3.

Welford. 12,232. See X.

## COMPLETE SPECIFICATIONS ACCEPTED.

15,385 (1916). Riedel. Production of ammonium chloride during the working of blast furnaces. (102,146.) July 24.

10,114 (1917). Ges. f. Linde's Eismaschinen A.-G. Separating the constituents of air or other gaseous mixtures. (109,789.) July 24.

10,202 (1917). Annable. See X.

12,179 (1917). Andreucci. Production of nitrogen from the air. (117,333.) July 24.

1344 (1917). Kimura. See XI.

13,487 (1917). Soc. Industrielle de Produits Chimiques. Manufacture of sodium carbonate and ammonium sulphate from sodium bisulphate. (109,814.) July 24.

17,165 (1917). Hemingway. Desulphurisation of gases and other fluids containing sulphuretted hydrogen. (117,387.) July 24.

# VIII.—GLASS; CERAMICS.

## APPLICATIONS.

Forster. Feeding or controlling feed of molten glass from glass furnaces or tanks. 12,093. July 25.

Hill and Manby. Polishing or grinding composition. 11,802. July 18.

Norton Co. 11,379. See VII.

Norton Co. Aluminous abrasive. 11,380. July 11. (U.S., Dec. 26, 1917.)

Roberts, Thomas, and Williams. Manufacture of tiles, bricks, and coarse ware. 11,889. July 20.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,257 (1917). Bennett. Kilns for firing earthenware, tiles, and other articles. (117,147.) July 17.

15,735 (1917). McCoy. Apparatus for manufacture of sheet glass. (117,194.) July 17.

16,592 (1917). Benjamin. See I.

2579 (1918). Edwards, Edwards, and Mollart. Glaze used in manufacture of pottery and like articles. (117,047.) July 10.

8003 (1918). Kettel, Gasch, and Dean. Method for platinising earthenware, pottery, etc. (117,432.) July 24.

# IX.—BUILDING MATERIALS.

## APPLICATIONS.

Akerman. Manufacture of Portland cement. 10,801. July 2.

Gastaldo and Minc. Artificial stone. 11,957. July 22.

Hoare. Production of building-material. 10,937. July 3.

Otto. Seasoning wood. 11,904. July 20.

Patentaktiebolaget Jungners Kali-Cement. Simultaneous manufacture of hydraulic cement and alkali. 11,369. July 11. (Sweden, July 11, 1917.)

Revello. Cementing substance. 10,878. July 2.

Roberts and others. 11,889. See VIII.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,690 (1917). Stewart. Concrete and the like. (117,504.) July 31.

12,116 (1917). Kiefer. Bricks or building blocks. (110,155.) July 24.

# X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Aktiebol. Svenska Kullagerfabriken. Hardening process. 10,886. July 2. (Sweden, June 18, 1917.)

Asheroft. Manufacture of magnesium and calcium metals and alloys. 10,808. July 2.

Ballantine and Sulman. Production of ferro-alloys. 10,819. July 2.

Berglund. Extraction of zinc in electric furnaces. 12,219. July 26. (Sweden, Oct. 22, 1917.)

British Thomson-Houston Co. (General Electric Co.). Methods of producing pure elements. 12,195. July 26.

Broadley. Machines for separating metals, ores, minerals, etc., by settlement and flotation. 10,828. July 2.

Brown, and Simon-Carves, Ltd. Crucible and melting-pot furnaces. 10,893. July 3.

Brunskill. Coating duralumen and aluminium sheets, wires, etc., with non-corrodible layer of copper or compound of copper with a sodium aluminate or both. 10,820. July 2.

Bullen. Concentration of ores. 11,140. July 6.

Cliff. Puddling, reheating, and like metallurgical furnaces. 11,553. July 15.

Elmore. Extraction of lead from its ores. 11,126. July 6.

Evans. Means for separating magnetic material. 11,940. July 22.

Hadfield. Manufacture of iron-silicon alloys. 10,924. July 3.

Hadfield. Manufacture of low-carbon ferro-manganese. 11,198. July 8.

Lalbin. Obtaining by a cold process a galvanic deposit of aluminium. 11,115. July 5.

Lord. 11,745 and 12,193. See III.

Marino. Process for electroplating metals, particularly for coppering iron and steel. 12,125. July 25.

Mooney. Treating steel for aircraft. 11,148. July 8.

Moysey. Prolongs used in the production of zinc or spelter. 11,678. July 17.

O'Gorman and Thomas. Indicating the condition of steel, etc., under heat treatment. 11,002 and 11,003. July 4.

Pope and Thomas. Indicating the condition of steel and other magnetisable metal under heat treatment. 11,104. July 4.

Rigg. Desulphurisation of zinc sulphide ores. 11,588. July 15. (Australia, Sep. 18, 1917.)

Rouvier. Uniting aluminium or aluminium alloy members to other members. 11,249. July 9.

See, Herme et Bulre. Treatment of iron ore and ferri ferrous materials in a pulverulent condition. 11,332. July 10. (Fr., July 10, 1917.)

Stabilimentl Biak Ing. Pouchain. Light alloy for parts subjected to high temperatures. 11,946. July 22. (Ital., Mar. 2.)

Stabilimentl Biak Ing. A. Pouchain. Manufacture of aluminium alloy. 11,947. July 22. (Ital., Mar. 22.)

Sumitomo Chukosho, Ltd. Magnet steel. 10,834. July 2. (Japan, July 10, 1917.)

Turner. Granulating and disintegrating aluminium, etc. 12,037. July 24.

Welford. Treatment of aluminium compounds to produce aluminium, metals, salts, etc. 12,232. July 27.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,385 (1916.) Riedel. *See* VII.

5298 (1917.) Herreshoff. Recovery of values from metalliferous slags. (117,470.) July 31.

8175 (1917.) Greaves and Etchells. Manufacture of steel. (117,286.) July 24.

9674 (1917.) Queneau. Separation of metals from their ores. (116,943.) July 10.

9736 (1917.) Wust. Method of improving the Thomas process. (107,770.) July 10.

9756 (1917.) Lancey. Crucible furnaces. (116,946.) July 10.

10,064 (1917.) Inman. Gas-heated and similar furnaces for heat-treating metals and the like. (117,301.) July 24.

10,073 (1917.) Electrolytic Zinc Co. Refining metallic zinc-bearing materials by the electrolytic process. (108,312.) July 24.

10,202 (1917.) Annable. Separation of magnesium from compounds and mixtures of magnesium and calcium carbonates. (117,483.) July 31.

10,990 (1917.) Wardle. Recovery of waste metal. (117,321.) July 24.

11,497 (1917.) Jones. Apparatus for electrically distilling ores, etc. (111,835.) July 10.

12,072 (1917.) Fritzweiler, Stuer, and Chem. Fabr. Rhenania. Preparation of catalytic substances from natural ores containing hydrated iron oxide. (109,056.) July 10.

13,306 (1917.) Aitken. Crucible furnaces for melting metals. (117,180.) July 17.

17,660 (1917.) Alldays and Onions Pneumatic Engineering Co., and others. Tilting crucible furnaces. (117,212.) July 17.

5152 (1918.) Gaunt, Brookfield, and Tylor and Sons. Tilting crucible furnaces. (117,054.) July 10.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

Berglund. 12,219. *See* X.

Hamblet. 12,236. *See* X.

Horll. Resuscitating spent dry cells. 11,464. July 12.

Lalbin. 11,115. *See* X.

Marino. 12,125. *See* X.

Marks (Stuart). Electrolytic cells. 11,739. July 17.

Mond and Rabinovitch. Electric batteries. 11,181. July 8.

Price. Electric batteries. 10,877. July 2.

#### COMPLETE SPECIFICATIONS ACCEPTED.

10,073 (1917.) Electrolytic Zinc Co. *See* X.

10,843 (1917.) Syndicat Gramans' Patent. Electric batteries. (108,482.) July 31.

11,497 (1917.) Jones. *See* X.

13,411 (1917.) Kimura. Apparatus for the electrolysis of water. (117,533.) July 31.

17,292 (1917.) Price. Electric batteries. (117,209.) July 17.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Betjemann. Toilet soaps. 11,733. July 17.

Carmichael and Dixon. Apparatus for extracting grease, oils, fats, resins, etc., from seeds, animal matter, sewage sludge, etc. 12,046. July 24.

Eoff. Manufacture of glycerol, etc. 11,948. July 22. (U.S., July 21, 1917.)

Gaudart. Extraction of coconut butter. 12,254. July 27. (Fr., Apr. 28, 1917.)

Spence. Linseed-oil substitute. 12,107. July 25.

Spensley. Manufacture of dry soaps. 11,699. July 17.

Wade. 11,407. *See* II.

#### COMPLETE SPECIFICATIONS ACCEPTED.

7944 (1917.) Townsend. *See* XIX.

805 (1918.) Schmid. Manufacture of substitute for sericin soap. (117,042.) July 10.

7249 (1918.) Tanaka. Manufacture of soap containing sulphur. (117,060.) July 10.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

##### APPLICATIONS.

Barr. Dopes. 10,862. July 2.

Bindley and Dulcken. Dopes. 11,759. July 17.

Carmichael and Dixon. 12,046. *See* XII.

Damard Lacquer Co., and Potter. Phenol-formaldehyde condensation products. 11,601. July 15.

Dreyfus. 12,085. *See* V.

Dunville and Co., and Gilmour. 10,949. *See* V.

Eaton. Plastic compositions. 11,060. July 5.

Peachey. Manufacture of a plastic or solid material, applicable also as varnish, coating, dope, or lacquer. 11,267. July 10.

Purex, Ltd., and Wilson. Production of basic lead sulphate. 10,978. July 4.

Soc. Anon. l'Hélice Intégrale. Process of lacquering. 12,002. July 23. (Fr., Aug. 30, 1917.)

Soc. Nauton Frères et de Marsac, and Tesse (Hagerstown Cap Co.). Transparent dope, and manufacture of same. 11,007. July 4. (Fr., Feb. 7.)

Spence. 12,107. *See* XII.

#### COMPLETE SPECIFICATIONS ACCEPTED.

7214 (1917.) Kilburn (Norske Aktieselskab for Elektrokemisk Industri). Manufacture of white or light-coloured pigments. (110,535.) July 31.

9534 (1917.) Shinozaki. Writing-inks. (117,117.) July 17.

16,653 (1917.) Ellis, Foster, and Ellis-Foster Co. Coumarone resin and process of making same. (117,016.) July 10.

18,156 (1917.) Hope. Manufacture of ink for printing on paper. (117,023.) July 10.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### APPLICATIONS.

Dagnall. Mineral rubber composition. 11,415. July 12.

Dunlop Rubber Co., and Worthington. Coagulation of rubber latex. 11,473. July 12.



Fol and Varenhorst. Regenerating vulcanised rubber. 11,193. July 8. (Ger., Mar. 18, 1914.)  
 North British Rubber Co., and Porritt. Vulcanisation of rubber, etc. 11,686. July 17.  
 Peachey. Vulcanisation of caoutchouc. 12,179. July 26.

COMPLETE SPECIFICATION ACCEPTED.

5012 (1917). Flint. Compound for use in lieu of rubber. (105,912.) July 24.

XV.—LEATHER; BONE; HORN; GLUE.

APPLICATIONS.

Gregory and Johnson. Tanning. 10,792. July 2.  
 Rüttimann. Waterproof leather substitute and process of making same. 11,375. July 11. (Switz., July 18, 1917.)

COMPLETE SPECIFICATIONS ACCEPTED.

9380 (1917). Johnson (Badische Anilin und Soda Fabrik). Manufacture of substances capable of precipitating glue or gelatin. (116,933.) July 10.  
 9381 (1917). Johnson (Badische Anilin und Soda Fabrik). Tanning, and manufacture of agents to be used therein. (116,934.) July 10.  
 9382 (1917). Johnson (Badische Anilin und Soda Fabrik). Tanning. (116,935.) July 10.  
 9420 (1917). Wade (Deutsch-Koloniale Gerb- u. Farbstoff-Ges.). Manufacture of products adapted to precipitate glue, etc. (116,936.) July 10.  
 14,621 (1917). Hall. Machines for treating hides, skins, and leather. (117,358.) July 24.  
 2054 and 4015 (1918). Tiltson and Melbourne. Treatment of hides, skins, etc. (117,581.) July 31.

XVI.—SOILS; FERTILISERS.

APPLICATIONS.

Conder. Apparatus for excavating superphosphates, etc. 12,160. July 25.  
 Stanley. Phosphatic fertilisers. 11,909. July 20.

XVII.—SUGARS; STARCHES; GUMS.

APPLICATION.

Dupire. 12,205 and 12,255. See XVIII.

XVIII.—FERMENTATION INDUSTRIES.

APPLICATIONS.

Dupire. Extraction of yeast from fermented musts of molasses. 12,205 and 12,255. July 26 and 27. (Fr., Feb. 23 and Mar. 22.)  
 Reeve. 11,824. See XIX.

COMPLETE SPECIFICATIONS ACCEPTED.

17,596 (1916). Soc. d'Exploit. des Proc. Boulard. Manufacture of alcohol from grain in open vats with the use of saccharifying mucors. (102,945.) July 17.

9466 (1917). Barbet et Fils et Cie. Apparatus for distilling in multiple effect alcoholic liquors and the like. (107,600.) July 10.

11,671 (1917). Gaebel. Rotary malt-drum. (116,966.) July 10.

XIX.—FOODS; WATER PURIFICATION; SANITATION.

APPLICATIONS.

Brooke. Apparatus for drying farina and other meal or grain. 11,920. July 22.

Carmichael and Dixon. 12,046. See XII.

Ferguson and Peyton. Food preparations. 10,974. July 4.

Gaudart. 12,254. See XII.

Greaves. Treating vegetable and animal waste materials to make them fit for food. 12,100. July 25.

Reeve. Vinegar substitute. 11,824. July 19.

Wallace. Garbage disposal. 11,010. July 4. (U.S., July 5, 1917.)

COMPLETE SPECIFICATIONS ACCEPTED.

4967 (1917). Campbell. Desiccated milk product and its manufacture. (117,276.) July 24.

7944 (1917). Townsend. Treatment of edible oils and fats and production of butter substitutes. (117,104.) July 17.

8085 and 16,624 (1917). Barry. Preservation of food. (117,107.) July 17.

9298 (1917). Doran. Curing meats. (107,593.) July 17.

17,597 (1917). Cutler. Egg food preparations. (117,022.) July 10.

XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

APPLICATIONS.

Ambler and others. 11,196. See III.

Imray (Soc. Chem. Industry in Basle). Manufacture of acetaldehyde. 12,008. July 23.

Jephcott. Obtaining nicotine, potash, etc., from tobacco, etc. 11,637. July 16.

Levinstein, Bader, and Levinstein, Ltd. Production of benzyl chloride and benzal chloride. 12,227. July 27.

McKee. 1,2,4-Methylhydroxyisopropylbenzene. 11,629. July 16.

Mead. Preparation of aromatic hydrazines. 10,900. July 3.

Napp (Preiswerk). Manufacture of isobutyl ester of oleic acid. 11,785. July 18.

Parkes and Pearson. Treatment of methane. 11,397. July 11.

Quinan. Production of carbon compounds. 11,851. July 19.

Silberrad. Manufacture of ethylene. 12,166. July 26.

COMPLETE SPECIFICATION ACCEPTED.

10,237 (1917). Jones (Nagai). Preparation of local anesthetics. (117,486.) July 31.

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

APPLICATIONS.

Hamblët. Fireproof, flexible, and unbreakable kinematograph films, photographic plates, coatings for telegraphic and telephonic wires, etc. 12,236. July 27.

Wilcockson. Iron photographic printing process. 11,900. July 20.

COMPLETE SPECIFICATION ACCEPTED.

9771 (1917). Shiels. Photographic prints and the like. (117,135.) July 17.

XXII.—EXPLOSIVES; MATCHES.

APPLICATIONS.

Chanard. Incendiary material. 10,884. July 2. (Fr., Mar. 22, 1917.)

Cunliffe. Matches. 12,032. July 24.

Garroni. Manufacture of explosives. 12,243. July 27.

Warden. Matches. 10,863. July 2.

XXIII.—ANALYSIS.

COMPLETE SPECIFICATIONS ACCEPTED.

12,453 (1917). Avery, Ltd., and Dobson. Hardness-testing machines. (117,526.) July 31.

3124 (1918). Michell. Methods and means for determining the viscosity of fluids. (117,234.) July 17.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Filters; New* — R. Zsigmondy and W. Bachmann. *Z. anorg. Chem.*, 1918, **103**, 119—128.

A new type of filter, termed a membrane filter, is prepared by drying solutions of certain colloids, under conditions not specified. The filters have a parchment-like appearance with a smooth surface, and it is claimed for them that they are strong, durable, and rapid. The size of the pores can be varied, and suitable membranes chosen for any desired purpose. The filters are specially suitable for filtering under reduced pressure, and an advantage is that after use they can be cleaned and used again with undiminished filtration velocity. They have been used successfully for filtering gold and graphite sols, and for separating mixed colloidal solutions containing particles of different sizes. (See also *J. Chem. Soc.*, Sept., 1918.)

—E. H. R.

**PATENTS.**

*Purifying or otherwise conditioning air or other gases; Apparatus suitable for* — and for other purposes. C. E. Jeffcock and W. H. Yardley. *Sheffield. Eng. Pat.* 116,512, Mar. 20, 1917. (Appl. No. 4055 of 1917.)

A SERIES of vertical parallel plates with horizontal corrugations is arranged in a casing with the crests of adjacent plates opposed, and liquid is allowed to drip on to the plates from a superposed chamber with a perforated bottom. The liquid flows in thin films over the plates and is collected in a sump from which it may be drawn by a pump for further circulation. The gas passes horizontally through the casing, and blades twisted helically are clamped between the corrugations to cause rotation of the streams of gas and ensure better contact with the wet plates. The diameter of the twisted blades is such that the opposed crests of the corrugated plates are spaced a short distance apart. The helical blades are arranged in two sets, separated by a free vertical space, and the liquid is caused to flow only over the plates at the air inlet end, covering one of the sets of blades. The treated gas expands on reaching the intermediate space and then passes through the spaces containing the other set of helical blades so that entrained moisture is deflected on to the plates and drains into the bottom of the casing. On leaving the plates, the treated gas may impinge on a layer of asbestos, felt, or other absorbent material to eliminate moisture before passing from the apparatus. In a modification, the suction pipe from the sump is provided with a strainer, and the superposed liquid-supply chamber is provided with a series of horizontal superposed perforated plates to distribute the liquid.—W. F. F.

*Mixing gases and liquids; Process for* — M. Bruman. *Berlin. Ger. Pat.* 304,042, Jan. 6, 1915.

The gas and liquid are forced separately into a chamber in which two turbine wheels rotate, one fixed and the other loose on the same axle, the former being coupled to a motor. The fluids are thoroughly mixed first in the inner and then in the outer turbine wheel and impelled by centrifugal force to the periphery, where they are collected by a pipe of trumpet shape, which delivers them into a receiving tank. The contents of the latter can be drawn through the apparatus again.

—H. J. H.

*Spraying liquids into gases; Apparatus for* — E. Enke. *Breslau. Ger. Pat.* 304,050, May 31, 1916.

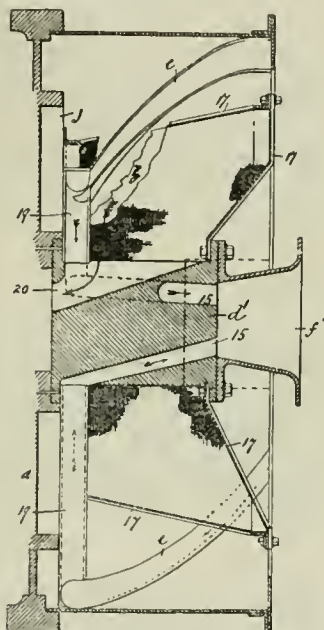
A JET is employed consisting of two cylindrical concentric tubes, the outer projecting beyond the

inner. The liquid is supplied through the inner tube and breaks into spray in consequence of the partial vacuum induced by the gas forced through the outer tube. No high pressures are required.

—H. J. H.

*Grinding ores, minerals, stones and the like; Machines for* — J. R. Broadley, *London. Eng. Pat.* 116,583, July 13, 1917. (Appl. No. 10,145 of 1917.)

THE material is ground in the ordinary way in a horizontal cylinder, *a*, provided with grinding balls, and then passes through a grid plate, *j*, into the compartment, 17, bounded by a perforated screen.



The finished material which passes through is collected by the shoots, *c*, which are partly radial and partly spiral, and is discharged through the passages, 19, to the conduits, 15, and outlet, *f''*, in the hollow trunnion, *d'*. The material which does not pass through the screen, 17, is returned by the radial guides, *z*, and passages, 20, to the cylinder, *a*, for re-grinding. In a modification two concentric screens are used; the material retained by the inner screen is returned by radial guides through the hollow trunnion to the grinding cylinder; the material retained by the outer screen is discharged axially at its open end into a hopper; and the fine material passing through both screens falls into another hopper. In a modification of the latter machine, a single screen only may be used.

—W. F. F.

*Drying apparatus for use in drying fruit, herbs, vegetables, seeds, chemicals, fibre, and the like.* C. S. Boulton, *Broughton, Northants. Eng. Pat.* 116,606, Aug. 16, 1917. (Appl. No. 11,795 of 1917.)

HOT air is passed over the material contained in shallow trays, which are arranged one above the other on platforms in a frame, and are provided with gauze bottoms. Two opposite sides of each tray project below the gauze bottom in the form of an obtuse-angled V, so that the tray may be tilted to one side or the other and the under and upper sides exposed alternately to the current of hot air. The air passes upwards at one side of



the tier of trays, and then horizontally over and through the trays. The inlet of each compartment containing a tray is provided with a baffle extending downwards from the upper edge so that by moving the tray to or from the opening and retaining it by means of a wedge, and by tilting it, the admission of air may be varied or stopped. The outlet from any compartment may be controlled or closed by a board or flap when a tray is or is not in use in that compartment.—W. F. F.

*Desiccating liquid substances; Process and apparatus for* —. C. E. Gray, Eureka, Cal. U.S. Pat. 1,266,013, May 14, 1918. Date of appl., Sept. 30, 1915.

THE drying chamber has the form of an inverted cone, and the liquid is sprayed on to the inner wall of the chamber by a rotary sprayer. A cyclonic air current is created through the chamber and over the drying surface for causing the particles of substance to move toward and remain near the drying surface when detached from it. The moisture is carried away by the air current, and the solid substance is mechanically detached from the surface and moved along to the discharge outlet by a scraper which travels in advance of the sprayer.—J. H. P.

*Dryer; Revolving cylindrical* —. L. F. Baier, Ravensburg, and F. Baier, Kempen. Ger. Pat. 303,016, Sept. 4, 1915.

THE dryer consists of a revolving cylinder fitted with longitudinal ribs bearing attachments arranged to throw the material backwards. Between the ribs are the blades of a screw conveyor which carries the material forward. The ribs are connected to a plate at the end of the cylinder, which is geared to move backwards and forwards and which communicates this motion to the ribs themselves.—H. J. H.

*Dryer; Tubular* — for lignite, etc. Zeitzer Eisengiesserei und Maschinenbau A.-G., Zeitz. Ger. Pat. 304,081, Dec. 31, 1913.

THE dryer consists of a horizontal cylinder containing a perforated central tube of large diameter, surrounded by other tubes, and the heating is effected by steam blown into the central tube and escaping by an outlet at the periphery. The outlet is connected by a system of pipes fitted with steam traps to the fresh steam injector, whereby the exhaust steam, after separation of condensed water, is returned to the dryer. The distribution of steam through the perforations of the inner tube can be controlled.—H. J. H.

*Evaporator*. W. G. Abbott, jun., Wilton, N.H. U.S. Pat. 1,265,863, May 14, 1918. Date of appl., Jan. 13, 1915.

THE apparatus comprises a series of pairs of concentric tubes, with means for circulating a fluid through the annular spaces between the tubes. A member fitting over the end of each inner tube has an outwardly flaring surface in order to deflect outwardly the fluid issuing from the annular space. Radial spacing fingers are arranged between the tubes.—J. H. P.

*Evaporator*. A. E. Hammond, Portland, Oreg. U.S. Pat. 1,266,461, May 14, 1918. Date of appl., May 21, 1917.

A LONG rectangular housing is provided with an air inlet and means for heating the air at one end.

At the opposite end the end walls converge and means are provided for circulating the air through the housing. Near the ends are curved partitions to distribute and retard the flow of the air. The material to be dried is carried through the housing in cars, which travel on longitudinal and transverse tracks, with turntables at the junctions.

—J. H. P.

*Filter; Continuous rotary pressure* —. J. A. McCaskell, Salt Lake City, Utah. U.S. Pat. 1,266,133, May 14, 1918. Date of appl., Apr. 18, 1917.

THE material to be filtered is admitted to a closed pressure chamber within which rotates a shaft having a series of hollow filtering elements attached to it. These filtering elements and the shaft have connecting filtrate passages. Fixed scrapers extend transversely to the shaft and remove solids deposited on the filters. The material scraped off the filters is collected in transverse troughs which are connected with a main trough inside the casing, parallel to the shaft. This latter trough is provided with a conveyor for removing the material.

—J. H. P.

*Emulsifier*. M. Leitch, Poughkeepsie, N.Y., Assignor to The De Laval Separator Co., New York. U.S. Pat. 1,266,501, May 14, 1918. Date of appl., June 14, 1915.

A ROTARY emulsifier comprises a central receiving chamber in the upper end of a vertical shaft and communicating by passages with an annular sediment collecting chamber. Above the latter and surrounding the receiving chamber are one or more emulsifying elements. The emulsifying elements have inlet passages leading from the sediment chamber and outwardly extending distributing passages terminating in emulsifying discharge orifices.—J. H. P.

*Stopper for carboys or the like*. British Dyes, Ltd., J. Turner, J. Bruce, and J. D. Eastwood, Huddersfield. Eng. Pat. 116,798, July 30, 1917. (Appl. No. 10,908 of 1917.)

*Heating apparatus [furnace]*. A. B. Chantraine, Marcinelle, Belgium. U.S. Pat. 1,265,746, May 14, 1918. Date of appl., Aug. 18, 1914.

SEE Fr. Pat. 475,649 of 1914; this J., 1916, 53.

*Furnace; Liquid-fuel* —. H. E. Yarrow, Glasgow. U.S. Pat. 1,266,372, May 14, 1918. Date of appl., Aug. 1, 1916.

SEE Eng. Pat. 17,450 of 1915; this J., 1916, 821.

*Separating fluids of different specific gravities; Apparatus for* —. J. S. Gander, London. U.S. Pat. 1,266,008, May 14, 1918. Date of appl., Jan. 13, 1916.

SEE Eng. Pats. 12,635 and 14,223 of 1915; this J., 1916, 1206.

*Filters; Outlet or extraction pipe of* —. J. J. Lassen and V. F. Hjort, London. U.S. Pat. 1,266,132, May 14, 1918. Date of appl., Mar. 12, 1917.

SEE Eng. Pat. 106,512 of 1916; this J., 1917, 904.

*Extraction of water from animal, vegetable and mineral substances; Electro-osmotic* —. B. Schwerin, and Ges. für Elektro-Osmose, Frankfurt, Germany. U.S. Pat. 1,236,329, May 14, 1918. Date of appl., Nov. 20, 1912. Renewed Oct. 24, 1917.

See Eng. Pat. 23,545 of 1912; this J., 1913, 200.

*Regenerative gas-furnaces.* Eng. Pat. 116,870.  
See IIa.

*Boiling-pans and the like.* Eng. Pat. 116,486.  
See XVIII.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Errors in ultimate analysis of — due to the presence of carbonates.* Knublauch. J. Gasbeleucht., 1918, 61, 183—186.

A study of twelve analyses shows that the error due to carbon dioxide liberated from alkaline-earth carbonates (this J., 1916, 295) may be considerable, e.g., 1% in the percentage of carbon, and also that it bears no relation to the ash content. In the proximate analysis of coal this carbon dioxide alters the character of the volatile matter, and consequently should be allowed for in the value of the factor, V, in Goutal's formula for calculating the calorific value.—H. J. H.

*Coal; Comparative study of the thermal decomposition of — and of some of the products of its carbonisation.* M. C. Whitaker and J. R. Suydam, jun. J. Ind. Eng. Chem., 1918, 10, 431—432.

COMPARATIVE experiments were made by decomposing powdered Pennsylvania gas coal, gasoline, kerosene, gas oil, benzene, and naphthalene at different temperatures, ranging from 650° to 900° C., in a vertical, electrically heated furnace consisting of an iron tube, 6 ft. long and 4 in. in diameter, and conducting the gases from the bottom of the furnace to a gas-holder. The coal was dropped from a hopper through the heated tube, whilst the other substances were vaporised by allowing them to trickle down a length of chain suspended in the upper part of the furnace. It was found that the paraffin hydrocarbons when thus "cracked" at the same temperature yielded gases of practically the same composition, whereas the aromatic hydrocarbons without side chains only began to decompose at a higher temperature than the paraffins and apparently yielded only hydrogen and methane on decomposition. Eliminating constituents containing nitrogen and oxygen, the coal gas was similar to that obtained from the paraffin oils and unlike that produced from the aromatic substances. Hence the conclusion is drawn that in general coal is composed of straight-chain compounds.—C. A. M.

*Wood and peat; Manufacture of gas from — in the municipal gasworks at Horsens, Denmark.* J. Qvist. J. Gasbeleucht., 1918, 61, 193—195.

THE shortage of coal in 1917 necessitated the carbonisation of wood and peat—the latter after a certain degree of drying in stacks. Horizontal band-fired retorts were used and a certain amount of coal was carbonised, but separately from the substitutes. The charge of wood or peat was 55–60 kilos. per retort and the make of gas 30 cub. metres per 100 kilos. Trouble occurred owing to accumulations of pitch in the hydraulic main. The gas contained 27–25% CO<sub>2</sub> and had a gross calorific value of 3700–4000 kilogram-calories per cub.

metre. On distribution of the gas there was an increase of trouble due to naphthalene stoppages. The yield of ammonia was small, but the admixture of a proportion of coal gas sufficed to neutralise the acetic acid present. The air supply of gas appliances required adjustment, but apart from an increased consumption the gas behaved satisfactorily in most of its applications. The pitch from wood and peat was valueless, but the brown tar found a ready market. The wood charcoal proved to be well adapted to use in suction gas producers. Peat charcoal was less satisfactory, as much of it was small and had to be employed as an indifferent domestic and industrial fuel.—H. J. H.

*Petroleum industry in Assam; The development of the —.* H. S. M. Jack. J. Inst. Petroleum Tech., 1917, 3, 263—276.

AN historical account is given of the progress made up to the present time in opening up and working the oilfields of Assam. The crude petroleum is found in intermittent deposits situated in loose sands. Considerable gas pressures are present. The crude oil has sp. gr. 0.856 and is very dark brown without the fluorescent greenish tint common to many American oils. It is perfectly fluid at 82° F. (27.5° C.), begins to deposit crystals of paraffin at 81° F. (27° C.), and becomes semi-solid at 77° F. (25° C.). The present practice is to subject the crude oil to continuous distillation in boiler stills arranged in groups of three. Crude benzine is taken off from the first still, intermediate kerosene from the second, and the bulk of ordinary kerosene from the third. The residue is distilled to dryness in pot stills, the distillate being passed successively through an anhydrous ammonia refrigerating machine and a carbonic acid machine. From the former is obtained the bulk of wax of high melting point—140° F. (60° C.) normally—and from the latter the basis of the batching oil, used for softening jute fibre. The coke remaining contains 97–98% pure carbon and is used as fuel under the stills. The non-condensable vapours from the boiler stills are used as fuel. The crude benzine is redistilled in closed steam coils, and two qualities of petroleum spirit are produced of sp. gr. 0.710 and 0.750 respectively.—T. St.

*Toluene; Determination of — in crude petroleum.* S. E. Bowrey. J. Inst. Petroleum Tech., 1917, 3, 287—305.

IN the examination of a crude oil the first operation is the elimination by distillation of all constituents boiling above 150° C., and the removal of unsaturated hydrocarbons from the distilled spirit by washing with sulphuric acid, alkali, and water. 500 c.c. of the prepared sample is then placed in a 1-litre Dewar flask, and washed several times with liquid sulphur dioxide at –35° C., this temperature being obtained by connecting the flask, after the admission of the sulphur dioxide, to a vacuum pump for a short time. After each washing, the sulphur dioxide with dissolved hydrocarbons is drawn over into a separating funnel and the solvent evaporated under reduced pressure. Light paraffins are also volatilised at this stage, together with a small amount of aromatic hydrocarbons. For very accurate determinations of benzene, therefore, it is desirable to use a special form of distilling apparatus for removing sulphur dioxide. Suitable apparatus is described. A 10% solution of sodium hydroxide is next admitted to the separating funnel to absorb sulphur dioxide vapours, drawn off again, and the hydrocarbons run off and measured. This fraction contains substantially all the aromatic hydrocarbons, and consists of about 80% of these substances and 20% of paraffins. A portion is then fractionated very slowly (six drops



per minute), the receivers being changed at 75°, 85°, 105°, 115°, and 130° C. A self-acting device for regulating the speed of distillation is described. This depends for its action on slight changes of pressure within the distillation flask being made to operate a mercury cut-off, acting upon the gas supply. The specific gravity of each fraction is determined by means of a Sprengel tube and the content of benzene, toluene, and xylene, as the case may be, calculated. The specific gravity of the paraffins is deduced from the range of distillation by simply determining the specific gravity of similar fractions from pure paraffin spirit. The aromatic hydrocarbons in intermediate fractions are divided equally between the benzene and toluene, and toluene and xylene, and evaporation loss during distillation is reckoned as made up of  $\frac{2}{3}$  benzene and  $\frac{1}{3}$  toluene. The total aromatic hydrocarbons left unextracted by sulphur dioxide are determined by nitration, and divided *pro rata* among the benzene, toluene, and xylene as determined in the extracted portion. In an additional note the author states that by adding to the sulphur dioxide extract five times its volume of kerosene (previously washed with sulphur dioxide if necessary), and again extracting with sulphur dioxide, an almost quantitative extraction of pure benzene, toluene, and xylene is obtained. Any paraffins present in the second extract, with the exception of a negligible proportion, boil above 150° C. and are eliminated in the course of fractionation.—T. St.

*Method for measuring the coefficient of expansion of pitch, asphaltum, and similar substances.* Kawakami. See III.

*Analysis of spent oxide.* See VII.

*Use of crude gas liquor as fertiliser.* Kayser. See XVI.

*Use of spent oxide as a fertiliser.* See XVI.

#### PATENTS.

*Gas furnaces; Regenerative* —. The E. W. Harvey Gas Furnace Co., Ltd., and E. W. Harvey, London. Eng. Pat. 116,870, Mar. 5, 1918. (Appl. No. 3857 of 1918.)

Is a "uni-directional" regenerative gas furnace having a preheating hearth and a finishing hearth, such as that described in Eng. Pat. 10,824 of 1912 (this J., 1912, 972), the gas is supplied continuously from a horizontal row of orifices at the end of the finishing hearth, and air is supplied from every alternate orifice of a row above the gas orifices. Part of the combustion products is withdrawn through the other alternate orifices, and passes through one of two regenerators. The gas is preheated by passing through the casing close to the regenerators and the hot gas passages. The remainder of the combustion products passes forwards over the preheating hearth. The ports for the air and combustion products may then be reversed. In a modification, the air ports may be divided into two series, above and below the gas ports. An oxidising atmosphere may be produced by placing the gas ports above the air ports or between two series of air ports, and a reducing atmosphere may be produced by placing a series of air ports between two series of gas ports. A low construction of heating chamber may be obtained by arranging the air ports to open through the floor at right angles to the gas ports, or if a clear way is required through the heating chamber, the gas and air ports may be arranged on both sides of the finishing hearth.—W. F. F.

*Gas producers, cupola furnaces, and the like; Construction of* —. H. A. Bartlett, London. Eng. Pat. 116,185, July 23, 1917. (Appl. No. 10,544 of 1917.)

A LAYER of heat-resisting resilient material is spread on a ferro-concrete base and a firebrick lining built up on it. The outside of the brickwork is covered with a layer of non-conducting resilient material which may be in the form of a rope wound round the brickwork. This material forms the core on which an outer casing of ferro-concrete is built up.—W. F. F.

*Vertical retorts for production of illuminating gas.* A. Birkholz, Zürich. Ger. Pat. 299,546, Dec. 24, 1915.

THE flue system of vertical retorts is divided into two sections—upper vertical and lower horizontal flues. The producer gas used for heating enters the system at a point about half-way up the retort, meeting the secondary air. The burning mixture travels up the vertical flues to a horizontal collecting channel, whence it is conveyed by a passage down the front of the retort into the top of the lower section of horizontal flues. The gases descend these by a zig-zag course and pass into the recuperators at the bottom. The heating is thereby more intense at the top of the retorts where distillation proceeds more vigorously.

—H. J. H.

*Gas producers or other chambers; Apparatus for feeding and distributing fuel or other material to* —. F. Thuman, London. Eng. Pat. 116,445, Oct. 25, 1917. (Appl. No. 15,537 of 1917.)

Is an apparatus for feeding fuel into gas producers, gas ovens, cupolas, or retorts without opening the fuel chamber to the air, an air-tight feeding chamber is mounted at the top of the producer or the like, and is provided with a slide-valve for admitting fuel, and a conical valve at the bottom for discharging fuel into the retort. The two valves are operated at the proper intervals by a single rotating cam or by interconnected cams. A rotating guiding spout is provided to deposit the fuel uniformly around the circumference of the retort. A vertical prodding or poking device is caused to act on the material in the feeding chamber during the time the outlet valve is open. In a modification, a stationary circular spreader may be provided below the outlet of the feeding chamber to distribute the material.—W. F. F.

*Water-gas containing varying proportions of the component gases; Adaption of twin generator gas plants for the manufacture of* —. H. E. Smith, and Perry and Co. (Bow), Ltd., London. Eng. Pat. 116,582, July 12, 1917. (Appl. No. 10,059 of 1917.)

To produce a higher proportion of carbon monoxide in water-gas, two generators are used, through which air is blown in parallel to heat them. A separate chamber lined with fireclay and containing limestone is provided between the two generators and is heated by the combustion products. When steam is passed through the generators in series, the water-gas from the first generator in passing through the limestone liberates carbon dioxide, which is reduced to monoxide in passing through the second generator. The intermediate chamber may be provided with a dividing wall, so that the gas passes through the two compartments in series, and dampers may be provided in this wall to vary the contact with the limestone. To reduce the proportion of carbon monoxide, a secondary jet of steam is passed through the second generator and oxidises part of the carbon monoxide to dioxide. The proportion of carbon monoxide may be reduced in this way to 15%.—W. F. F.

*Methane; Process for the synthetic production of* — E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 116,302, June 28, 1917. (Appl. No. 3192 of 1917.)

One part of coke, charcoal, or graphite in finely divided form is intimately mixed with three parts of one or more of the hydroxides of sodium, potassium, aluminium, manganese, iron, titanium, calcium, barium, or strontium and with about 1 part per 1000 of titanium, uranium, copper, nickel, or their compounds to act as a catalyst. The mixture is placed in a firebrick retort and heated to 400°—1000° C. according to its composition. Methane is liberated and collected.—W. F. F.

*Producer; Suction gas* — K. Schneidewind. Leipzig. Ger. Pat. 304,094, May 28, 1915.

The supply of air below the grate is furnished partly through a pipe of fixed aperture and partly through a valve which is automatically controlled by the suction produced by the engine in the delivery pipe of the producer. Above the seat of the valve an inverted cone is suspended from one arm of a balance governor, and from the other a piston working in a cylinder which is connected through two chambers, inserted to damp pulsations, to the delivery pipe. Variations of pressure in the latter raise or lower the cone and thus open or close the valve.—H. J. H.

*Gas producer with arrangement for removing coke.* Dr. North Kommandit-Ges., Hanover. Ger. Pat. 304,401, Nov. 4, 1916.

The producer is of section resembling the iron blast furnace, the zone of gasification being the lower portion tapering downwards. The wall of this portion, at about the middle of its height, is pierced by a ring of openings communicating with a closed annular chamber into which coke can fall, and where it cools prior to removal. A slight constriction at the widest place, at the top of the gasification zone, insures regularity of descent of the charge. A portion of the coke can be extracted without interruption and cooling of the producer.—H. J. H.

*Washer scrubbers for extracting ammonia, naphtha, and other impurities from coal gas.* J. A. Spencer, Southall. Eng. Pat. 116,319, Nov. 24, 1917. (Appl. No. 7674 of 1917.)

The scrubber consists of a large number of rods or tubes of hard wood or metal assembled in a rotating drum. The ends of the rods are mounted in perforated plates and held in position by outer non-perforated plates secured to the perforated plates by bolts.—W. F. F.

*Hydrocarbon oils and the like; Process and apparatus for the cracking of* — J. Nelson, Glasgow. Eng. Pat. 116,304, Mar. 13, 1917. (Appl. No. 3668 of 1917.)

Hydrocarbon oil or vapour is forced through a bath of liquid material, such as molten metal, maintained at a suitable temperature. For working at moderate temperatures, molten lead is a suitable contact substance; for higher temperatures, metals or alloys of higher melting point may be used. The extent of cracking may be controlled by varying the depth at which the oil or vapour is delivered below the level of the liquid, and the whole or a part of the product, e.g., the heavier fraction, may be again treated in a similar manner. If the hydrocarbon contains tarry matter, or other substances which cannot be volatilised, it may be distilled from an auxiliary vessel, and the distillate or vapours then subjected to the cracking

process. The passage of the vapours through the liquid keeps it thoroughly agitated, thus maintaining a uniform temperature throughout the contact mass and preventing accumulation of carbonaceous material. A simple form of apparatus for the process consists of a vertical cast-iron cylinder about 2 ft. 6 in. high and 2½ in. internal diameter, heated at its lower end, and provided at the upper end with a lateral exit pipe and a gas-tight cover through which passes an adjustable inlet pipe, the lower end of which is immersed in a quantity of molten lead. Kerosene of sp. gr. 0.803 after one treatment in this apparatus, the lead being maintained at about 700° C., and the inlet pipe dipping 4 in. below the surface of the lead, yields about 20% of light spirit of sp. gr. 0.757 and boiling at or below 160° C., no carbonaceous deposits being formed. Other forms of apparatus are described in which the vapours are caused to pass several times through a bath of the molten metal, e.g., by means of vertical internal partitions, open alternately above and below the liquid, the apparatus being essentially free from constrictions or long highly-heated channels.—L. A. C.

*Mineral oils; Apparatus for refining* — C. E. Lapp, Los Angeles, Cal. U.S. Pat. 1,266,281, May 14, 1918. Date of appl., Nov. 5, 1917.

A HELICAL pipe coil is mounted vertically within a cylindrical wall mounted on a base. The convolutions of the coil are held spaced apart by supporting columns which are made up of units fitted together end to end, and have seats in their ends to receive the convolutions of the coils. Draw-off pipes leading upwards from various parts of the coil are provided, and means for securing the units together.—T. St.

*Montan wax; Extraction of* — from lignite. A. Riebeck'sche Montanwerke Akt.-Ges., Halle. Ger. Pat. 305,349, Aug. 9, 1916.

LIGNITE is dried until its moisture content does not exceed 25% and then extracted with a mixture of alcohol and benzene.—L. A. C.

*Naphthenic acids; Process for treating [deodorising]* — Erdöl-Verwertungs-Akt.-Ges., Berlin. Ger. Pat. 305,771, Sept. 17, 1916.

NAPHTHENIC acids, either alone or mixed with fatty acids, are treated with nitrous acid, halogens, or hypohalogenous acids, preferably by blowing a stream of air charged with the reagent through the oil, or else by the action of substances which liberate such reagents, with or without the addition of acids, the reagents being used in small quantities just sufficient to overcome the odour of the naphthenic acids.—J. F. B.

*Coke discharged from retorts or furnaces; Apparatus for removing* — Drakes, Ltd., and J. W. Drake, Halifax. Eng. Pat. 116,567, June 27, 1917. (Appl. No. 9188 of 1917.)

*Coke-ovens; Storage bunkers and compressing apparatus used in connection with* — Simon-Carves, Ltd., and S. Hunter, Manchester. Eng. Pat. 116,680, Mar. 21, 1918. (Appl. No. 4994 of 1918.)

*Coke-oven; Regenerator* — E. Lecocq, Brussels. U.S. Pat. 1,265,684, May 7, 1918. Date of appl., May 6, 1913.

SEE Fr. Pat. 457,674 of 1913; this J., 1913, 1000.



*Ammonia [by the direct recovery process from coal gas]; Manufacture of —.* E. Oré, Caen, France. U.S. Pat. 1,266,147, May 14, 1918. Date of appl., Oct. 12, 1917.

SEE Eng. Pat. 115,449 of 1917; this J., 1918, 405 A.

*Tubular dryer for lignite, etc.* Ger. Pat. 304,081. See I.

*Treatment of producer-gas tar from coal.* Ger. Pat. 302,398. See III.

*Process for increasing the decolorising action of fullers' earth.* Ger. Pat. 304,076. See VII.

*Apparatus for distilling oils, fats, and the like.* Eng. Pat. 116,309. See XII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Retorts; Vertical — for distilling, carbonising, roasting and the like of coal, carbonaceous, and other material.* H. R. L. Walker and W. R. Bates, London. Eng. Pat. 116,458, Nov. 17, 1917. (Appl. No. 16,951 of 1917.)

In a vertical retort for the destructive distillation of coal, shale, peat, or wood, gas burners are placed in inclined radial passages in the walls, and secondary air is drawn in through segmental, vertical passages in the walls. The combustion products pass upwards through zig-zag flues surrounding the central carbonising chamber, and then downwards through segmental flues alternating with the secondary air passages, to a transverse horizontal flue passing through the retort. The gases then pass upwards through a central concentric heating flue to the discharge flue. The volatile distillation products are withdrawn from the top of the annular retort. The charge is fed from a hopper and is moved downwards by an annular ram operated hydraulically. To facilitate removal of the coke, the upper side of the transverse flue is constructed as a knife edge.—W. F. F.

*Leading-in conductor [e.g., for electric lamps].* R. Jacoby, Berlin, Assignor to General Electric Co. U.S. Pat. 1,265,665, May 7, 1918. Date of appl., Apr. 8, 1914.

SEE Eng. Pat. 1056 of 1914; this J., 1915, 906.

*Preparation of active carbon from sewage sludge.* Ger. Pat. 305,895. See XIX.

## III.—TAR AND TAR PRODUCTS.

*Naphthalene; Determination of — in tars and tar oils.* Knublauch. J. Gasbeleucht., 1918, 61, 134—137, 145—149.

THE author's method of estimating naphthalene by direct titration of the picrate with standard alkali (this J., 1917, 702) has been adapted to the analysis of tars and tar oils. The precipitation is effected with both the sample and the picric acid in alcoholic solution. After mixing, the solution is diluted with water, and the precipitation is complete if the resulting solution contains not more than 10% of alcohol and at least 0.2% of picric acid or less than 12.5% of alcohol and at

least 0.3% of picric acid. The quantity of picric acid must be chosen to ensure these end concentrations without the precipitation of free picric acid. *Crude naphthalene and tar oils.* A quantity containing 0.1—0.2 gm. of naphthalene is weighed into a 200 c.c. conical flask; 15 c.c. of absolute alcohol is added and picric acid equal to 2 to 2½ times the weight of naphthalene. The flask and contents are heated to 60°—65° C. until all is dissolved, cooled, and the volume made up to 120 c.c. with a 0.3% aqueous solution of picric acid. After 1 hour the precipitate is collected on a 10 cm. smooth filter and washed with a 0.2% picric acid solution. The paper and contents are transferred to a small beaker with 50—80 c.c. of water and titrated with N/10 caustic soda, using methyl orange as indicator, a deduction of 0.2 c.c. being made for the picric acid retained by the paper and precipitate. Phenol does not interfere. The results with pure naphthalene were quantitative. *Tar.* The quantity taken should contain about 0.07 gm. of naphthalene, the proportion of picric acid as above but in no case more than 0.6 gm. so as to avoid precipitation at the end. In the case of viscous tars, the sample is weighed into a small beaker and the picric acid dissolved in 15 c.c. of alcohol added. After heating the beaker and contents to 60°—65° C., the tar is carefully extracted by stirring and manipulation with a glass rod for at least 30 minutes. When cold, 15 c.c. of water and 120 c.c. of 0.3% aqueous picric acid solution are added. Leaving the tar residue in the beaker, the picrate precipitate is transferred to a filter and the determination continued as above. Tars containing much light oil and little naphthalene give unsatisfactory end points, and in such cases the picrate precipitate is decomposed by an excess of N/10 caustic soda, the liquid is washed with hot water into a small beaker, and then filtered into a conical flask. 5—6 c.c. of a neutral solution of an ammonium salt is added and the ammonia liberated is determined by distillation into a measured volume of N/10 sulphuric acid. When the filtration of the picrate is difficult, the tar is extracted with 15 c.c. of a solution of picric acid in alcohol, the extract being poured into a conical flask. Further extractions are made with 35 c.c. of alcohol, and the washings added to the first, leaving the tarry residue behind. 35 c.c. of the alcohol is removed by distillation and the determination of the picrate continued as described. *Light oils or tar oils.* It is necessary to remove a portion of the light oil before precipitation. The oil taken is treated with the picric acid dissolved in 25 c.c. of alcohol and a portion of this is distilled off. After cooling, the 0.3% solution of picric acid is added so as to dilute the alcohol ten times and the determination is completed by one of the methods described. The volume of alcohol distilled and the weight of picric acid taken must be chosen so that the end concentrations are appropriate.—H. J. H.

*Pitch, asphaltum and similar substances; Method for measuring the coefficient of expansion of —.* K. Kawakami. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 24, 107—112.

THE apparatus consists of a glass cylinder, 2 cm. in diam. and 11.5 cm. long, provided with a glass stopper carrying a graduated capillary tube about 13 cm. long. The substance to be tested is melted and poured into the cylinder, leaving a small space about 1 cm. deep on the top, and allowed to cool. The space above the substance is next filled with coloured water and the stopper is placed in the cylinder. The level of the water in the capillary is adjusted to zero and the whole apparatus is placed in a water thermostat kept at the required temperature until the rise of the water in the

capillary ceases. The capillary is calibrated by filling with mercury, and the volumes of the whole apparatus and of the water are also measured. From the above data the volume expansion of the substance is calculated, correction being made for the expansion of the water and of the glass.

—J. F. B.

*p*-Aminophenol; *Electrolytic preparation of* —. T. Shōji. *Kōgyō-Kwagaku-Zasshi* (J. Chem. Ind., Tokyo), 1918, 21, 117—136.

THE author has studied the conditions for the preparation of *p*-aminophenol by the electrolytic reduction of nitrobenzene in presence of sulphuric acid. Platinum electrodes are most satisfactory, but a carbon cathode may be used. The concentration of the sulphuric acid must not exceed 80%. In order to avoid sulphonation of the aminophenol. The quantity of sulphuric acid should be five times that of the nitrobenzene. The temperature must be kept at about 80° C., as a higher temperature accelerates sulphonation. The current density should be 8—9 amp. per sq. dm. and the E.M.F. 6—8 volts. Vigorous agitation of the electrolyte is most important from the point of view of efficiency, and with good agitation the quantity of electricity consumed is nearly theoretical. The cathode chamber must be kept closed to avoid loss of nitrobenzene by evaporation. Under these conditions the yield of *p*-aminophenol is 43% of the theoretical. A portion of the sulphuric acid may be used again after making up with strong acid; another portion may be used for neutralisations, e.g., of alkali fusions, etc. In spite of the low yield the method is capable of competing with the chemical method on account of its simplicity and because nitrobenzene is a cheaper raw material than phenol.—J. F. B.

*Anthraquinone; Determination of* —. II. F. Lewis. *J. Ind. Eng. Chem.*, 1918, 10, 425—426.

A METHOD for the separation of anthraquinone from anthracene and phenanthraquinone consists in boiling the mixture with an excess of 5% sodium hydroxide solution and zinc dust; the anthraquinone is reduced to oxanthranol (Graebe and Liebermann, *Annalen*, 1871, 160, 126) and goes into solution. The mixture is filtered and the insoluble portion treated with further quantities of alkali and zinc dust. When the filtrate is shaken with air, the anthraquinone is re-oxidised, and may be collected, dried, and weighed. (See also *J. Chem. Soc.*, Sep., 1918.)—W. P. S.

*Organic sulphur compound; Preparation of a new type of* —. G. N. White. *Chem. Soc. Trans.*, 1918, 113, 608—609.

THE crystalline sodium salt of  $\beta$ -naphthylthiosulphoxylic acid,  $C_{10}H_7S.S.H$ , can be obtained by heating a mixture of  $\beta$ -naphthyl disulphide, sulphur, and sodium sulphide crystals. The corresponding lead salt, obtained by double decomposition, gradually decomposes when kept, changing from its original deep orange colour through scarlet and brown to black, whilst when heated with alcohol it yields  $\beta$ -naphthyl disulphide. It is probable that analogous compounds are formed as intermediate products in the manufacture of sulphide dyes.

—D. F. T.

*Comparative study of the thermal decomposition of coal and of some of the products of its carbonisation.* Whitaker and Suydam, jun. See IIa.

*Determination of toluene in crude petroleum.* Bowrey. See IIa.

## PATENTS.

*Bituminous binder and process of making same from coal-tar pitch.* G. A. Henderson, St. Albans, W. Va. U.S. Pat. 1,266,261, May 14, 1918. Date of appl., July 30, 1917.

COAL-TAR pitch is mixed with sulphur and petroleum, and then heated to drive off the petroleum, whereby a bituminous binder is produced consisting of sulphur 6 to 14, and coal-tar pitch 86 to 94%.—T. St.

*Producer-gas tar from coal; Treatment of* —. A. Riebeck'sche Montanwerke A.-G., Halle. Ger. Pat. 302,398, Dec. 14, 1916.

THE tar or tar oil is washed with a limited quantity of alcohol or wood spirit at a moderate temperature. Air is blown through, forming oxygenated products which are dissolved by the alcohol. The alcohol layer and the undissolved portion are separated and both freed from spirit. The oxygenated products are left as heavy viscous liquid suitable for lubricating waggon axles. The undissolved portions are lighter and can be fractionated into motor spirit and lubricating oil. The necessity for washing with sulphuric acid, with its attendant losses and inconveniences, is avoided.

—H. J. H.

*Cresols; Oxidation of* —. U. Pomilio, Turin, Italy. U.S. Pat. 1,265,378, May 7, 1918. Date of appl., Aug. 20, 1917.

SEE Eng. Pat. 103,739 of 1916; this J., 1917, 382.

*Electrical insulating materials [from pitch].* Eng. Pat. 116,532. See XI.

*Apparatus for distilling oils, fats, and the like.* Eng. Pat. 116,309. See XII.

*Process and apparatus for the cracking of hydrocarbon oils.* Eng. Pat. 116,304. See IIa.

*Process for tanning animal hides.* Ger. Pat. 304,859. See XV.

## IV.—COLOURING MATTERS AND DYES.

*Naphthal- and naphthylamine-sulphonic acids; Action of tetrazodi-o-tolylmethane chloride on* —. I. J. H. Stebbins, jun. *J. Ind. Eng. Chem.*, 1918, 10, 445—448.

TWO red dyes are obtained by the action of tetrazodi-o-tolylmethane chloride on 1,4-naphtholsulphonic acid; one, which is formed in aqueous solution, contains one mol. of 1,4-naphtholsulphonic acid and is insoluble in water, the other, containing two mols. of the acid, is produced when the reacting solution contains alcohol; it is soluble in water. These two products dye wool but have no affinity for cotton. In aqueous sodium carbonate solution tetrazodi-o-tolylmethane chloride combines directly with two mols. of 2,3,6-naphtholdisulphonic acid. (See also *J. Chem. Soc.*, 1918, 1., 353.)—W. P. S.

## PATENTS.

*Acid wool dyestuffs; Manufacture of* — indifferent to copper. O. Imray, London. From Soc. Chem. Ind. in Basle, Switzerland. Eng. Pat. 116,530, June 11, 1917. (Appl. No. 8329 of 1917.) Addition to Eng. Pat. 15,127, Oct. 26, 1915.

DYESTUFFS containing copper and soluble in water are prepared by treating mordant-dyeing azo-



dyestuffs derived from diazocarboxylic acids of the benzene or naphthalene series or from nitro-, sulpho-, or halogen derivatives of such acids, which contain no hydroxyl group in *o*-position to the diazo-group, with copper or a copper compound, as described in Eng. Pat. 1611 of 1915 (this J., 1916, 301), or by producing the dyestuffs in presence of copper or a copper compound as described in Eng. Pat. 15,127 of 1915 (this J., 1916, 1104; see also Eng. Pats. 12,249, 13,204, and 16,916 of 1915; this J., 1916, 419, 922, and 1917, 78).—L. A. C.

*Azo-dyestuffs; Manufacture of chromium compounds of —.* O. Imray, London. From Soc. Chem. Ind. in Basle, Switzerland. Eng. Pat. 116,535, June 12, 1917. (Appl. No. 8420 of 1917.) Addition to Eng. Pat. 104,045, Mar. 13, 1916.

ACCORDING to the present process, which is a modification of that described in the chief patent (see U.S. Pat. 1,221,849; this J., 1917, 542), chromium compounds of azo-dyestuffs, used for producing fast tints in an acid bath, as described in Eng. Pat. 15,456 of 1915 (this J., 1916, 1255), may be prepared by treating the dyestuff in presence of a solvent (*e.g.*, water) at a raised temperature with chromium hydroxide or a hydrated chromium oxide, preferably in freshly precipitated or colloidal condition, or with a normal chromate, with or without the addition of a reducing agent or an alkali-fixing agent, or both.—L. A. C.

*Mono-azo dyes; Preparation of mordant —.* Ges. für Chem. Ind. in Basel. Ger. Pat. 304,674, Dec. 29, 1915.

DIAZO compounds, especially *o*-hydroxydiazo compounds of the benzene or naphthalene series, are coupled with the condensation product from isatin and resorcinol prepared as described in Ger. Pat. 290,599 (this J., 1916, 530).—L. A. C.

[*Sulphur dyes.*] *Method for preparing blue colouring materials. Method for preparing direct-dyeing black colouring materials.* R. Vidal, Asnières, France. U.S. Pats. 1,265,836 and 1,265,837, May 14, 1918. Date of appl. Aug. 18, 1916. SEE Fr. Pat. 468,526 of 1913; this J., 1914, 957

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER

*Textile fibres; Investigations on —.* W. Harrison. Proc. Roy. Soc., 1918, A, 94, 460—469.

DOUBLE refraction in textile fibres is due to the presence of internal stresses. It may be increased by compressing the fibres, for instance, between a thick glass plate and a grooved celluloid film placed on a second glass plate, the whole being examined on the stage of a microscope in polarised light between crossed nicols and the difference between the compressed and uncompressed portions noted. Examined in this way under a pressure of 5 tons per sq. in., wool fibres showed interference figures indicating spreading of the fibre substance in all directions away from the centre of pressure; cotton fibres offer greater resistance to deformation than wool fibres and show no interference figures. After removal of the pressure, in both cases, the fibres did not return to their original shape; the increased double refraction of the compressed portions remains until the stresses are relieved by immersion in water. The return to the original shape in cold water is much more rapid in the case of wool than in that of cotton. Similar results were obtained when fibres which had been bent

or twisted were placed in cold water. Fibres subjected to a limited amount of extension when dry do not return to their original length when kept loose in a dry atmosphere, but do so rapidly when placed in water; the effect of a humid atmosphere is the same as that of cold water but much less rapid. At high temperatures, water renders fibres truly plastic; deformation is produced by compression, but this causes little or no internal stresses. These experiments have a bearing on the "feel" and finish of textile fabrics. The double refraction shown by cotton fibres in the natural state is due to permanent strain produced by internal stresses; it disappears when the fibre is swollen by cuprammonium solution, except in those bands where swelling has not occurred. The direction of strain is parallel to the axis of the fibre and the natural condition of cotton fibres corresponds with that produced by tension on an elastic body; all the other natural fibres appear to be subject to similar stresses. The shrinkage in length which takes place on mercerisation appears to be due to the balancing of the internal stresses. The condition of a highly nitrated cotton fibre containing more than 12.5% of nitrogen is just the reverse and corresponds to that produced in an elastic body by compression in the direction of the axis. In both cotton and wool the distribution of the internal stresses is irregular. The modification of the internal stress produced by boiling wool in water is similar to that produced by treating cotton fibres with concentrated alkalis. Cold water has no influence in relieving the natural internal stresses, probably because these are due to changes in volume, whereas stresses caused by external compression are only due to changes in shape. Artificial fibres are generally produced with internal stresses which cause double refraction, similar to those occurring in natural cotton, and the physical forces which are operative in the formation of artificial fibres from viscous fluids are analogous to those acting in the formation of natural fibres from plastic cell materials.—J. F. B.

*Cotton plant; Chemistry and histology of the glands of the — with notes on the occurrence of similar glands in related plants.* E. E. Stanford and A. Viehoever. J. Agric. Research, 1918, 13, 419—435.

INTERNAL glands occur in the cortex, foliage, flower and seed of the different species of cotton plants, and to some extent in plants of allied genera (*Thespesia*, *Eriarylon*, etc.). The glands in portions of the plant exposed to light contain quercetin, probably in the form of its glucosides, quercimeritrin or isoquercitrin, ethereal oil, resins, and possibly tannins. They are surrounded by flattened cells which contain a red anthocyan pigment. The glands not normally exposed to light contain gossypol and are surrounded by a layer of flattened cells which do not contain anthocyan. On exposure to the light the gossypol is replaced by quercimeritrin. —C. A. M.

*Hemp; Korean —.* K. Ueda. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 314—319.

HEMP bast fibre is produced in large quantities in Korea; it is prepared by an artificial retting process and the scutching is done by hand. The strands average 1.2—1.3 m. in length and 0.6—0.7 mm. in breadth; the fibre has a pale yellow colour and appears of excellent quality. An analysis of a typical specimen showed: moisture, 9.77; ash, 2.07; oil and wax, 1.26; aqueous extract, 7.30; cellulose, 71.03; incrusting matter, 10.64%. The cellulose was determined by Cross and Bevan's method; lignocellulose was present in the raw fibre.—J. F. B.

*Chemistry of the cotton plant, with special reference to Upland cotton.* Vlehoever and others. See XX.

#### PATENTS.

*Cotton; Process for imparting a transparent appearance to —.* E. Heberlein, Wattwil, Switzerland. U.S. Pat. 1,265,082, May 7, 1918. Date of appl., July 20, 1917.

TRANSPARENT effects on cotton fabrics are produced by subjecting the goods to the action of a caustic alkali solution of at least 15° B. (sp. gr. 1.109), washing and then treating with sulphuric acid of at least 50.5° B. (sp. gr. 1.498), both treatments being carried out at a temperature below 0° C. The order of the treatments may be reversed, and the two treatments may be repeated alternately; one of the reagents may be caused to react only in places to produce pattern effects, and the fabrics may be treated under tension.—J. F. B.

*Cellulose; Manufacture of coverings or masses of structureless —.* C. F. Cross, London, and Viscose Development Co., Ltd., Bromley, Kent. Eng. Pat. 116,366, June 12, 1917. (Appl. No. 8418 of 1917.)

CELLULOSE regenerated from viscose is deposited in the form of a covering or mass and washed; the shrinkage which takes place during drying is regulated by subjecting the covering or mass during the period of dehydration to an intermittent or continuous rolling pressure, which is increased as the dehydration proceeds. For instance, annular cylinders of structureless cellulose are prepared by regenerating cellulose from viscose in a cylindrical mould with a core; the core is removed, and the cellulose cylinder washed free from salts; the cellulose is mounted on the spindle or other article to be covered and allowed to shrink thereon, it is then rolled on or by a rigid surface under pressure during the drying process, which may be assisted by heat or hot air. At a certain stage, preferably when the mass contains about 60% of cellulose and 40% of water, glycerin may be applied to the cellulose and worked in by the rolling.

—J. F. B.

*Hanks or warps of yarn, woven or felted fabrics, leather, and fibrous materials; Machinery employed for drying —.* T. Ogle, Leeds. Eng. Pat. 116,214, Oct. 22, 1917. (Appl. No. 15,281 of 1917.)

THE material, suspended from carriers travelling on chains, passes through a central longitudinal chamber separated by partitions from two lateral compartments. Fans and heating arrangements are situated in the lateral compartments and slots are pierced in the partitions through which heated air passes into the central chamber, the slots being arranged in groups alternately so that those which are at the top on one side are directly opposite to slots at the bottom on the other side. The air is thus caused to circulate across the chamber diagonally and through the material from one side to the other alternately and in an upward or downward direction, or both.—J. F. B.

*Acetyl cellulose, and process of making same.* W. G. Lindsay, Newark, N.J., Assignor to The Celluloid Co. U.S. Pat. 1,265,216, May 7, 1918. Date of appl., July 11, 1914.

CELLULOSE is treated in a preliminary bath with alcohol and acetic acid or other diluent, which without itself altering the cellulose induces acetylation in the subsequent process; the treated cellulose is then treated with a mixture of acetic anhydride

and a diluent, such as benzene. The product consists of fibrous acetylcellulose which has its original structure unaltered and is soluble in acetylene tetrachloride.—J. F. B.

*Acetylcellulose; Compound of — and process.* W. G. Lindsay, Newark, N.J., Assignor to The Celluloid Co. U.S. Pat. 1,265,217, May 7, 1917. Date of appl., Sept. 1, 1916.

AN acetylcellulose, which becomes plastic in chloroform, is dissolved together with camphor in a common solvent, a liquid monohydroxyaliphatic alcohol having more than two carbon atoms is added, and the mixture dried.—J. F. B.

*Pyroxylin composition.* F. Sparre, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,266,073, May 14, 1918. Date of appl., Sept. 3, 1914. Renewed Sept. 28, 1917.

A PYROXYLIN which is soluble to the extent of at least 50% in a mixture of equal parts of commercial ethyl alcohol and benzene is dissolved in a solvent containing 45–60% of benzene, 30–45% of alcohol, and 3–15% of a more powerful solvent, such as amyl acetate.—J. F. B.

*Lignonesulphonic acid or its salts; Method of preparing nitroso compounds from —.* E. Oman, Stockholm, Sweden. Eng. Pat. 103,654, Jan. 4, 1917. (Appl. No. 205 of 1917.) Under Int. Conv., Jan. 20, 1916.

LIGNONESULPHONIC acid or one of its salts is treated with nitrous anhydride or substances yielding nitrous anhydride. For example, sulphite-cellulose waste lye, after precipitation of the ligninsulphonates by salt and concentration to about one-sixth of its volume, is treated with a solution of about 60 grms. of sodium nitrite per litre of concentrated filtrate and 300–500 grms. of 20–25% sulphuric acid whilst cooling and stirring, and the nitroso compound is precipitated by adding a salt, e.g., sodium sulphate. (Reference is directed in pursuance of Sect. 8, Sub-sect. 2, of the Patents and Designs Act, 1907, to Eng. Pat. 103,653; this J., 1918, 146A.)

—J. F. B.

*Sulphite cellulose waste lye; Method of treating — to make it suitable for further treatment.* E. Oman, Stockholm, Sweden. Eng. Pat. 105,492, May 12, 1917. (Appl. No. 6793 of 1917.) Under Int. Conv., May 13, 1916.

SULPHITE waste lye at a concentration of 10% dry substance contains about 25% of sugars, 25% of calcium ligninsulphonates, and 5% of calcium lignonesulphonates. The ligninsulphonates are precipitated by saturating the waste lye, without any preliminary treatment, with sodium chloride and heating to 50°–70° C., and the lignonesulphonates remain dissolved in the saturated liquid, even after concentration (see also this J., 1918, 121A). (Reference is directed under Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 20,036 of 1893; this J., 1894, 1085.)—J. F. B.

*Paper, textilose, or other similar material; Manufacture of thread, string, and cord from strips of —.* Soc. Alsacienne de Constructions Mécaniques, Belfort, France. Eng. Pat. 113,970, Mar. 6, 1918. (Appl. No. 3914 of 1918.) Under Int. Conv., Apr. 27, 1914.

*Wood; Machine for comminuting — [for making wood pulp].* A. G. Bloxam, London. From Zellstoff-fabrik Waldhof, Mannheim-Waldhof, Germany. Eng. Pat. 116,604, Aug. 10, 1917. (Appl. No. 11,536 of 1917.)



*Paper; Apparatus for regenerating old* — B. A. Navarre, Lyon, France. U.S. Pat. 1,265,809, May 14, 1918. Date of appl., Sept. 14, 1917.

SEE Eng. Pat. 114,257 of 1917; this J., 1918, 264 A.

*Drying apparatus for use in drying fruit, herbs, vegetables, seeds, chemicals, fibre, and the like.* Eng. Pat. 116,006. See I.

*Process of printing paper.* Eng. Pat. 116,109. See VI.

*Purification and deodorisation of industrial sewage.* Ger. Pat. 304,040. See XIX B.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Acer ginnala; Application of leaves of* — [for dyeing, etc.] in Korea. Y. Ueda. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 211—219.

A SPECIES of maple tree, *Acer ginnala* (Korean name "Shinnamu"), is widely distributed in Korea. The leaves contain about 16% of tannin and a small quantity of yellow colouring matter. The extract is prepared by boiling the leaves with water and evaporating; the yield is about 30%. Analyses of Shinnamu extract showed: moisture, 16.29 and 19.50%; tannins, 38.47 and 37.71%. Used as a dyestuff it gives, on cotton, black and grey shades with iron mordant and khaki with chrome-copper mordant; on silk and tussah, black suitable for weighting purposes with iron mordant; on wool, fast khaki with chrome mordant.—J. F. B.

*Aniline Black; Notes on steamed* — E. Bechtel. Färber-Zeit., 1917, 28, 300. Z. angew. Chem., 1918, 31, Ref., 182.

IN place of hydrochloric acid a mixture of hydrochloric and nitric acids may be used with advantage. The vegetable fibre is not injured and the liberated chlorine contributes to the oxidation of the aniline. Potassium ferrocyanide may be replaced by the sodium salt in presence of which the black is oxidised more rapidly. The calcium salt is not so satisfactory, as it tends to form sparingly soluble double salts with potassium and sodium salts. The ferrocyanides also exert a neutralising influence. The mineral acid decomposes principally the chlorates, with the formation of oxygen, chlorine, and chlorous acid, and this mixture of oxidising gases becomes active during the steaming of the goods to develop the colour. A mixture of ferrocyanide with aniline oil and acid does not produce a black but gives a blue on vegetable fibres; this imparts a certain freshness to a black which is not excessively soaped and neutralises in a physical sense the action of the reducing agents which cause "greening." In the strongly oxidising atmosphere which is necessary for the development of Aniline Black, the discharge of *p*-Nitraniline Red by hydrosulphite-formaldehyde is very easily effected. A temperature of 90° C. and a short treatment of one minute is amply sufficient, whereas in the ordinary way in an atmosphere free from air a temperature above 100° C., and a treatment of several minutes are employed.

—J. F. B.

*Vat dyestuffs; Process for printing coloured reserves with certain* — R. Haller. Färber-Zeit., 1917, 28, 247—249. Z. angew. Chem., 1918, 31, Ref., 183. Sealed communication, Soc. Ind. Mulhouse, Jan. 21, 1913.

IT is well known that zinc or manganese salts or a mixture of the two act as an efficient reserve in Indanthrene dye-baths, and there are various methods for thus obtaining white effects. If a vat dyestuff be added to such a reserve, e.g. Indanthrene Yellow, together with stannous oxide and anthraquinone, it is possible by simple dipping of the printed goods in an Indanthrene or other similar dye-bath, to fix the added colour in pure shade. The alkali of the Indanthrene dye-bath penetrates into the reserve and, in conjunction with the stannous oxide, reduces the vat dyestuff present in it, while the manganese and zinc salts also present in the reserve prevent it from taking up the colour from the main dye-bath. Still better results are obtained if "Hydrosulphite conc." and a solution of potassium sulphite are added to the printing colour. By this means the added dyestuff is already reduced in the printing colour, so that the leuco-compound is dissolved by the alkali of the dye-vat and converted into a form in which it is most easily fixed on the fibre. The addition of a little stannous chloride assists the reduction process. It has also been found that Rongalite and ferrous sulphate mixed with the printing colour and added to the dyestuff will also produce coloured reserves in Indanthrene dye-vats. The printed goods are steamed at 105°—110° C., passed through the dye-bath, scoured, washed, and dried. The alkali dissolves the leuco-compound and enables it to be fixed on the fibre. The ferrous sulphate acts in the first place as a reserve but also completes the reduction of the printed dyestuff.

—J. F. B.

*Vat dyestuffs; Explanation of Haller's process for printing coloured reserves on certain* — H. Pomeranz. Färber-Zeit., 1917, 28, 309—310. Z. angew. Chem., 1918, 31, Ref., 183.

IN Haller's process (see preceding abstract) it remains unexplained how the neutralisation of the alkali and its action as a fixing agent can proceed simultaneously. Haller uses for his printed reserve an addition of anthraquinone and zinc chloride. The special case of Indanthrene Yellow printed as a reserve in an Indanthrene Blue vat is explained by the fact that the anthraquinone and the zinc hydroxide precipitated by the alkali from the zinc chloride present a certain resistance to the fixation of the Indanthrene Blue but not to that of the Indanthrene Yellow. This difference in behaviour of the two dyestuffs would be sufficient to account for the fixation of the yellow in preference to the blue, in view of the short time of immersion in the blue dye-vat. A similar explanation holds for the second reserve method described by Haller, only in this case there is the additional influence of the ammonium base in the Rongalite CL, which is also opposed to the fixation of the Indanthrene Blue.—J. F. B.

*Investigations on textile fibres.* Harrison. See V.

## PATENTS.

*Wool, yarn or other fibrous material; Apparatus for scouring, dyeing, or otherwise treating with liquor* — H. Walker, Cleckheaton. Eng. Pat. 116,129. (Appl. Nos. 7603, May 26, and 17,253, Nov. 23, 1917.)

THE liquor is contained in a trough in which are situated a number of perforated cylinders with or without vanes inside them. A travelling apron carries the material under the perforated cylinders

and between groups of rollers situated between each two cylinders throughout the length of the machine. The material may be held in position on the apron by one or more guiding tapes or bands which travel with the apron and return outside the trough above the cylinders. The action of the perforated cylinders on the travelling material causes the liquid to circulate in and out of the material in a vertical direction without disturbing its arrangement.—J. F. B.

*Azo colours; Production of insoluble — on the fibre.* The Imperial Trust for the Encouragement of Scientific and Industrial Research, London, and G. G. Hepburn, Manchester. Eng. Pat. 116,360, June 11, 1917. (Appl. No. 8303 of 1917.)

THE material is printed or impregnated with a solution obtained from a "diaz salt" or "tetrazo salt" prepared by mixing diazonium salts with salts of 2-naphthol-1-sulphonic acid (Eng. Pat. 11,757 of 1895; this J., 1896, 446), and dissolving the compound so obtained in a solution of caustic soda, in the proportion of one molecule of the latter for each diazo group, or in a solution of a salt having an alkaline reaction, and then developing the colour by ageing or steaming or by an acid bath or atmosphere. In printing, the solution may be printed on the material previously impregnated with an acid or acid salt. A solution for producing *p*-Nitraniline Red is prepared by treating the diazo salt from *p*-nitraniline and 2-naphthol-1-sulphonic acid with an alkali or alkaline salt.—J. F. B.

*[Aniline] Black; Production of — on vegetable fibres.* The Calico Printers' Association, Ltd., and E. A. Fourneaux, Manchester. Eng. Pat. 116,562, June 23, 1917. (Appl. No. 9022 of 1917.)

THE ingredients specified in Eng. Pat. 115,278 of 1917 (this J., 1918, 367 A) are employed, with the addition of boric acid, by itself or in conjunction with a phosphoric acid, preferably metaphosphoric acid, which gives a relatively soluble aniline salt. The proportion of boric acid may vary between 0.5 and 1 mol. per mol. of aniline, but when bases of the benzidine series are present it may be considerably less. The use of boric acid, with or without phosphoric acid, obviates the loss of aniline which tends to occur when the goods are dried without due care. Example: aniline, 60; aniline salt, 20; *p*-phenylenediamine dihydrochloride, 3; formic acid (90%), 90; lactic acid (30%), 48; metaphosphoric acid, 12.75; boric acid, 20; sodium chlorate, 48; copper chloride, 11 parts by weight, made up with water to 1000 by volume with the required amount of thickening.—J. F. B.

*Slubbing yarn and the like; Dyeing of — and apparatus therefor.* P. Crayton, Bradford. Eng. Pat. 116,652, Dec. 7, 1917. (Appl. No. 18,123 of 1917.)

THE tops are unwound and the slubbing is passed into an upright dyeing box perforated at the top and bottom and divided by means of perforated loose shelves into a series of superposed chambers, so as to permit the dye liquor to circulate freely in an upward or downward direction through the material, each chamber containing an equal proportion of the slubbing, and the slubbing in the successive chambers being passed through a central opening in each shelf and maintained in one continuous unbroken length suitable for rewinding from the box. The dye box also has upright slots in the sides which can be closed when necessary, and dye liquor may be circulated either in a vertical direction or in a lateral direction without disturbing the arrangement of the slubbing.

—J. F. B.

*Beam dyeing machine.* E. and L. Holt, Burlington, N.C. U.S. Pat. 1,265,332, May 7, 1918. Date of appl., July 13, 1914.

THE yarn treating apparatus comprises a closed receptacle, a mixing tank surrounding the receptacle in the form of a jacket, and a circulating system for circulating liquid from the mixing tank in different directions through the receptacle. The receptacle may be constructed in the form of a vat having ports, and fluid is introduced from the circulating system through either port and is withdrawn through the other port; a perforated yarn-holding member is enclosed within the vat having an open end above one of the ports, guides are fixed converging towards the last-mentioned port to be engaged by the yarn-holding member and to guide it into position, and means are provided for holding it in position.—J. F. B.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,266,108 and (B) 1,266,109, May 14, 1918. Dates of appl., (A) Apr. 6, (B) May 16, 1917. (See also this J., 1918, 412 A.)

(A) A HORIZONTAL dyeing receptacle has pyramidal flanges at both ends and a series of parallel foraminous plates within the dyeing receptacle; means are provided for maintaining the plates at predetermined distances from each other, and a chamber is situated at each end of the receptacle having flanges which abut against those of the receptacle, making a tight joint; means are provided for passing liquid and air through the chambers and foraminous plates, also for removing the liquid from the dyeing chamber after it has passed through a chamber and some of the plates, means for revolving the dyeing receptacle and for removing the receptacle from abutment with the chambers. A smaller and similar testing device is provided externally so that the material may be examined without interrupting the main treatment. (B) In a vertical type of apparatus the receptacle contains a series of fibre chambers with non-perforated sides and foraminous plates, top and bottom, adjustable relatively to each other, a second series of fibre chambers, a liquid chamber below the series of fibre chambers, a second liquid chamber containing a series of nested members diverging upwards, a pump which forces the liquid upwards between the nested members, and through the liquid chambers and fibre chambers to a reservoir, from which it returns to the pump. The foraminous plates are composed of a series of concentric rings with connecting members, the top plate having elements extending downwards and the bottom having elements extending upwards.

—J. F. B.

*Fabric-treating device.* H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,266,110, May 14, 1918. Date of appl., June 20, 1917. (See also this J., 1918, 412 A.)

THE receptacle contains a foraminous cylinder comprising a series of spaced parallel members having projections extending outwards within the receptacle, an inner foraminous cylinder spaced from the outer cylinder, tubes connecting the openings in the inner cylinder with a supply source of liquid, steam, and air, sliding annular members capable of closing all the openings on the outer cylinder which are not covered by fabric and arranged to abut on the edges of the fabric wound on the cylinder, a flexible foraminous cover tightly adjustable around the outside of the fabric, and means for rotating the cylinders and for forcing liquid, steam, and air through the goods. A testing device which revolves with the cylinders is connected with the space between the two foraminous cylinders.—J. F. B.



*Sericin soap for dyeing silk and yarns and fabrics of silk or silk waste; Manufacture of a substitute for —.* Gebr. Schmid, Basle, Switzerland. Ger. Pat. 305,239, Sept. 12, 1917. Addition to Ger. Pat. 291,075 (see Eng. Pat. 100,169 of 1916; this J., 1916, 734).

A SMALL quantity of acid is added to the water used in the original process, to facilitate the dissolving of the chrysalides by acting on their oil and to prepare them for the further treatment.

—J. F. B.

*Silk; Process for dyeing black and weighting —.* F. C. Schmidt, Wolgast. Ger. Pats. (A) 305,275, (B) 305,770, Mar. 9, 1913, and Feb. 19, 1916.

(A) IN the usual process of dyeing Logwood Black on silk and weighting the goods, the number of tin phosphate baths is reduced and sodium or potassium silicate is substituted for the Marseilles soap in the first logwood bath. In order to avoid the usual topping with an aniline dyestuff, a suitable iron bath is given after the first logwood bath and dyeing is completed in the second dye bath without an aniline dyestuff. Treatment with tannin is omitted and the shade thereby becomes richer and the silk retains more lustre; a loading of 75–80% is attained with the same number of tin phosphate treatments as give only 20–25% when tannin and iron are employed. By this process the haematoxylin is oxidised in the fibre to haematein and is absorbed in much greater quantities than when tannin has been used. In dyeing without iron the aniline dyestuff may be added to the logwood bath, whereas the tannin-iron black hinders the absorption of aniline dyestuffs. (B) Sodium silicate is used in the logwood bath instead of soap and one or several dyeing treatments may be given. The substitution of silicate for soap gives a higher loading and a more bulky fibre.—J. F. B.

*Paper; Process of printing —.* C. Jäger G.m.b.H., and R. W. Carl, Düsseldorf, Germany. Eng. Pat. 116,109, Dec. 27, 1916. (Appl. No. 18,508 of 1916.)

DYES soluble in water or insoluble lakes or pigments are mixed with gelatin compounds such as glue, agar-agar, sugar materials, or casein and with a substance capable of forming formaldehyde at a temperature such as is attained by the rollers of the printing machine (up to 80° C.), e.g. hexamethylenetetramine. If a dyestuff be employed which itself gives insoluble compounds with formaldehyde, e.g. the direct cotton dyestuffs, the nitrogenous colloid may be wholly or partially omitted or replaced by substances which do not react with formaldehyde. Example: 100 parts of lampblack is mixed with 400 of water, 1500 of glue dissolved in 1500 of water is added and then 500 of hexamethylenetetramine. Paper printed with these inks can be bleached and used again.

—J. F. B.

*Dyeing or staining and mordanting of leather [with titanium salts].* Eng. Pat. 116,123. See XV.

*Purification and decolorisation of industrial sewage.* Ger. Pat. 304,040. See XIXb.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

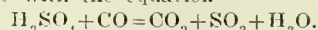
*Sulphuric acid; Intensive system of manufacturing —.* J. Thede. Z. angew. Chem., 1918, 31, 2–3, 7–8.

THE intensive system of manufacturing sulphuric acid, which is characterised by an increased circulation of the nitrogen compounds through the

chambers, requires special arrangements to obtain full working efficiency. Petersen's chamber regulator (this J., 1910, 1250) has given good results in practice, whilst Meyer's process, in which the gases are introduced tangentially into cylindrical chambers, promotes the formation of the acid. The primary requisite, however, is great height of the lead chambers, which should not be less than 10 to 15 m. high. As an example of how a considerably increased output can be attained without special plant, the author describes the results obtained in a plant of the following dimensions, without any special devices: The plant consisted of a low four-chamber system, in which the second and third chambers were 6 m. high and the first and fourth respectively 5.2 and 5.1 m. high. The first chamber was provided with an extension 7 m. in height for the introduction of the gases. The lead mantle of the Glover tower was attached directly to the stonework, the air-cooling being so efficient that little repair was necessary. This form of construction is only advisable in cases where the gases are free from fluorine and will not attack the stoneware. The Glover tower had a diameter of 3 m. and was 9 m. in height, and there were two Gay-Lussac towers. The burner gases containing about 7% by vol. SO<sub>2</sub> were forced by a wrought iron fan into the Glover tower at about 500° C., whilst there was a second fan of hard lead between the first and the central Gay-Lussac tower. As a rule the amount of circulating acid is equal to from 2 to 4 times the amount of acid produced, but in this plant the amount reached 1000% for the Glover tower and 500% for each of the two Gay-Lussac towers. The strong and weak nitrous vitriol were united and delivered together to the Glover tower. The direct consumption of nitric acid (36° B., sp. gr. 1.33) was 240 kilos., equal to 1.3%, referred to 100 kilos. of sulphuric acid (60° B., sp. gr. 1.71). In addition the circulating strong nitrous vitriol contained 1018 kilos. (1.1% 36° B., HNO<sub>3</sub>) and the weak nitrous vitriol 278 kilos. (0.3% 36° B., HNO<sub>3</sub>). The total quantity of nitric acid of 36° B. present was thus 1536 kilos., or about 8% of the sulphuric acid produced (18,500 kilos. of 60° B.). An advantage of the large volume of circulating acid was that an acid relatively free from arsenic (0.04 to 0.09%) was obtained. The gases leaving the Glover tower at 70° C. were heated by the reaction in the first chamber to 130° to 140° C. Drawbacks of this system are that the lead of the fourth chamber is strongly attacked, and that slight irregularities in working tend to cause the reaction to be delayed, to such an extent indeed that it takes place in the Gay-Lussac tower. Petersen's chamber regulator (*loc. cit.*) tends to prevent this, and the system may also be rendered more stable by cooling the acid from the Glover tower to about 60° C. before entering the Gay-Lussac towers, the conversion of the sulphur dioxide into sulphuric acid being effected more rapidly at a lower temperature (see Fr. Pat. 406,641 and Addition thereto; this J., 1910, 487, 627). Under these conditions the consumption of nitric acid of 36° B. is as stated, about 1.3% for the production of 100 kilos. of sulphuric acid of 60° B., or 1.04% for acid of 50° B. (sp. gr. 1.53).—C. A. M.

*Sulphuric acid; Reduction of — by means of carbon monoxide.* J. Milbauer. Chem.-Zeit., 1918, 42, 313–315.

SULPHURIC acid is reduced by carbon monoxide in accordance with the equation—



The reaction, which does not take place to any appreciable extent below 200° C., is not affected by the water which is produced, so long as the strength

of the sulphuric acid is not reduced below 91%. It is promoted by the catalytic action of many substances, notably lead, rubidium, iridium, selenium, ruthenium, silver, gold, and tin, but platinum, although acting catalytically, is also a "poison," and osmium and copper sulphate are inactive. The velocity of the reaction depends upon the concentration of the catalyst. Dilution of the carbon monoxide with nitrogen or with oxygen checks the velocity, and when the admixture reaches 50% the reaction is practically inhibited. This reaction explains the formation of carbon dioxide in Fownes and Rayleigh's method of preparing carbon monoxide by the interaction of potassium ferrocyanide and sulphuric acid (*Proc. Roy. Soc.*, 1899, 62, 204). It also accounts for the fact that in the decomposition of secondary and primary carboxylic acids by hot sulphuric acid the liberated carbon monoxide is contaminated with sulphur dioxide.—C. A. M.

*Ammonia; Effect of acetylene on the oxidation of — to nitric acid.* G. B. Taylor and J. H. Capps. *J. Ind. Eng. Chem.*, 1910, 10, 457–459.

The activity of the platinum catalyst is seriously affected when acetylene is present in the ammonia-air mixture; in the presence of 0.02% of acetylene the yield falls from 95% to 89%, or less, whilst with 0.1% of acetylene it falls to 65%. Acetylene and other non-reacting gases may be removed almost entirely from ammonia by absorbing the latter in water to form a concentrated solution, and then vaporising the ammonia by means of air. —W. P. S.

*Alkalis in silicates; The Lawrence-Smith method for the determination of —.* P. Wenger and E. Brange. *Monit. Scient.*, 1918, 8, 97–99.

In this method the finely divided silicate is heated with nine times its weight of a mixture of calcium carbonate and ammonium chloride (1:8); the alkalis are thus converted into chlorides. Investigation of the method showed that the decomposition of the silicate is complete after the mixture has been heated at 700° C. for 2 hrs.—W. P. S.

*Oceanic salt solutions; Complete review of —.* IV. E. Jänecke. *Z. anorg. Chem.*, 1918, 103, 1–54.

A CONTINUATION of previous work (this J., 1918, 142 A). The equilibrium of the system  $(Na_2-K_2-Mg)(Cl_2-SO_4)$

is further considered in relation to temperature. The equilibrium conditions are illustrated by numerous diagrams. (See also *J. Chem. Soc.*, Sept., 1918.)—E. H. R.

*Magnesium oxalate; Precipitation of —, and the theory of the formation of precipitates.* A. Astruc and J. Camo. *J. Pharm. Chim.*, 1917, 17, 381–386.

In the separation of calcium from large quantities of magnesium, the precipitation of the calcium oxalate should be made from very dilute solution. The tendency of magnesium oxalate to precipitate at the same time increases with the concentration of magnesium acetate, but is decreased by increasing quantities of oxalic acid and by the presence of ammonium chloride.—W. P. S.

*Bismuth subnitrate; Determination of nitric acid in —.* E. Luce. *Bull. Soc. Chim.*, 1918 [iv.], 23, 264–271.

The nitric ion in bismuth subnitrate may be determined by using a modification of Deboureaux's

method for nitrates. A solution is prepared containing 20 grms. of crystallised oxalic acid and 60 c.c. of concentrated sulphuric acid in 500 c.c. To 50 c.c. of this solution are added 1 gm. of manganese sulphate and 0.5 gm. of the bismuth subnitrate, and this mixture is heated on a water-bath for two hours under a reflux condenser, a slow current of carbon dioxide being passed through the flask during the whole time. The liquid is then thoroughly cooled, filtered, and made up to 100 c.c., and of this 50 c.c. is titrated with standard potassium permanganate (approx. 2%). The original oxalic acid solution is also titrated, and from the difference in the readings, the percentage of nitric acid in the bismuth subnitrate is calculated on the basis that one molecule of nitric acid oxidises three molecules of oxalic acid.—W. G.

*Spent oxide; Analysis of —.* *Z. angew. Chem.*, 1918, 31, 45–46.

The following methods are used by the "Wirtschaftl. Vereinigung deutscher Gaswerke" and by the "Deutschen Gold- und Silber-Scheideanstalt" for controlling the sale and purchase of spent oxide. *Moisture, etc.* 25 grms. of the powdered sample is dried at 80° C., for about 4 hrs., or until the loss in weight between two successive weighings does not exceed 0.05 gm. *Sulphur.* 10 grms. of the dry sample is shaken occasionally during a few hours with 75 c.c. of carbon tetrachloride, then diluted with the solvent to 105 c.c., mixed, allowed to settle, and 50 c.c. of the clear solution is filtered into a small weighed flask, the filter washed with carbon tetrachloride, the contents of the flask evaporated, and the residue of sulphur weighed. *Ferrocyanide.* 10 grms. of the dry sample is shaken with 50 c.c. of 10% potassium hydroxide solution; after 16 hrs., the mixture is diluted to 255 c.c., filtered, and 100 c.c. of the filtrate is treated with 25 c.c. of hot ferric chloride solution (ferric chloride, 6 grms., hydrochloric acid, 20 c.c., water, to 100 c.c.). The mixture is heated at 80° C. for some time, filtered, and the filtrate agitated with 20 c.c. of 10% potassium hydroxide solution until the blue colour has disappeared. The whole is then diluted to 250 c.c., 1 gm. of lead carbonate is added, and after filtering, 100 c.c. of the filtrate is acidified with 5 c.c. of sulphuric acid (1:5), and titrated with copper sulphate solution which has been standardised against pure potassium ferrocyanide under the same conditions; ferric chloride is used as an external indicator.—W. P. S.

*Halogens, sulphur, and nitrogen; Determination of — in the presence of mercury.* M. François. *Comptes rend.*, 1918, 166, 1000–1003.

For the estimation of halogens in mercury compounds the mercury is removed by means of zinc turnings by the method previously described (this J., 1918, 445 A), omitting the addition of potassium iodide. The filtrates and decanted liquids are mixed and in this solution the halogens are estimated by means of silver nitrate in the usual manner. Sulphur present as sulphide is first oxidised by bromine in hydrobromic acid as described (*loc. cit.*) and then, after the removal of the mercury by means of zinc, is precipitated as barium sulphate and weighed. For the estimation of nitrogen in mercuriammonium or mercuriamine compounds, the filtrate, after the removal of mercury, is made alkaline and distilled into dilute hydrochloric acid, and the nitrogen weighed as ammonium chloride or amine hydrochloride as the case may be.—W. G.



*Critical elaboration of quantitative precipitation methods, exemplified by a method for the determination of phosphoric acid.* Heidenhain. See XXIII.

*Determination of phosphoric acid as magnesium pyrophosphate. III. Magnesium tetra-ammonium phosphate.* Balareff. See XXIII.

#### PATENTS.

*Nitric acid; Concentration of* —. Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania, Norway. Ger. Pat. 305,915, Apr. 29, 1915. Addition to Ger. Pat. 278,867.

The sulphuric acid employed for drying moist nitric acid vapour (see Fr. Pat. 465,504 of 1913; this J., 1914, 643) is more or less strongly cooled. For example, 80% sulphuric acid at 30° C. may be introduced at the top of the tower, and the quantity of the mixed nitric acid and steam is so regulated that the nitric acid vapour leaves the tower at about 95° C.—L. A. C.

*Oxalic acid; Production of* — by direct oxidation of alkali fusions of substances containing cellulose. A. Mensen, Neustadt. Ger. Pat. 303,166, Feb. 25, 1914.

HEATED air is blown through the melt which is stirred to obtain uniform and efficient oxidation and maintained limpid by the addition of water. Cooling of the mass is avoided, and the melt may have a depth of up to 1 metre, while the oxidation proceeds more vigorously than in the older process. —H. J. H.

*Alkali metals or the like; Process for producing* —. W. L. Morrison, Canonsburg, Pa., Assignor to Electric Reduction Company. U.S. Pat. 1,265,360, May 7, 1918. Date of appl., Aug. 17, 1916.

A COMPOUND of an alkali metal (e.g. basic silicate) is heated with a non-carboniferous reducing agent, under such conditions that the production of reaction gases is avoided as far as possible, and the alkali metal vapour is condensed and collected. The vapour may be burnt to form alkali peroxide. —C. A. K.

*Aluminium compounds; Process for the manufacture of* — poor in iron. Det Norske Aktieselskab for Elektrotenisk Industri Norsk Industri-Hypotekbank, Christiania, Norway. Eng. Pat. 112,948, Jan. 24, 1918. (Appl. No. 1403 of 1918.) Under Int. Conv., Jan. 24, 1917.

PLAGIOCLASE rocks of the labradorite-anorthite series or other raw materials rich in plagioclase are treated with dilute or concentrated mineral acids, e.g., 30–70% sulphuric acid, 25–60% nitric acid, or 10–30% hydrochloric acid, whereby solutions of salts of aluminium, calcium, and the alkali metals are obtained from which alumina is precipitated in the usual manner. Under such conditions the minerals rich in iron (e.g., hypersthene, diallage, almandine, and epidote) are undecomposed, while the alumina of the plagioclase is dissolved. —L. A. C.

*Aluminium compounds [and a fertiliser]; Production of* —. H. Goldschmidt, Christiania, Norway. Eng. Pat. 113,276, Feb. 4, 1918. (Appl. No. 2019 of 1918.) Under Int. Conv., Feb. 2, 1917.

MATERIAL containing other metals (potassium, sodium, calcium) besides aluminium (e.g., plagioclase rocks of the series labradorite-anorthite, etc.) is treated with nitric acid and the mixture of nitrates thus obtained is heated, whereby the

hydrated aluminium nitrate is decomposed into aluminium oxide and nitric acid, the decomposition beginning at 140° C., and being complete at about 300° C., below which temperature other nitrates present undergo no decomposition. The aluminium oxide, produced in a form which is readily filtered, is separated by washing and filtering or decanting. Nitric acid is recovered from the escaping gases, and the solution of nitrates obtained by leaching the decomposition products may be used either for the production of a mixed fertiliser or for separation into different nitrates, or for the manufacture of nitric acid. The decomposition may be carried out under reduced pressure.—L. A. C.

*Ammonium sulphate; Process of manufacturing* —. T. Fujiyama, Tokyo, Japan. Eng. Pat. 116,321, May 31, 1917. (Appl. No. 7838 of 1917.)

AMMONIA gas and carbon dioxide are caused to act on a solution of sodium sulphate, whereby sodium bicarbonate is precipitated and a solution of ammonium sulphate is obtained. To the solution, containing traces of ammonia and dissolved sodium bicarbonate, sulphuric acid is added, drop by drop, until these are converted respectively into ammonium sulphate and sodium sulphate. By evaporating the solution (preferably above 100° C.) and cooling (preferably to below 0° C.), the sodium sulphate can be completely separated, ammonium sulphate alone remaining in solution.—L. A. C.

*Cyanide; Manufacture of [alkali]* —. G. Calvert, Twickenham Park, Middlesex. Eng. Pat. 116,365, June 12, 1917. (Appl. No. 8404 of 1917.)

IN the manufacture of alkali cyanide by the action of nitrogen and carbon on an alkali metal or salt in the presence of a catalyst (iron), effective distribution of the catalyst throughout the reaction mass is ensured by introducing it in the form of iron carbonyl vapour together with the nitrogen. The mixture of iron carbonyl and nitrogen may be prepared by passing air over coke to form nitrogen and carbon monoxide, and then passing the product over finely-divided iron to convert the carbon monoxide into iron carbonyl. A convenient form of apparatus consists of a horizontal iron retort, heated by a gas burner, in which briquettes of carbon and sodium carbonate are piled. Iron carbonyl is supplied into a distributing box at the top of the retort, whence it passes into the retort by numerous perforations. The supply pipe is lagged with insulating material to avoid decomposition of the iron carbonyl. Nitrogen is supplied by a pipe entering the retort at one end, the gaseous by-products escaping by a pipe at the other end. The escaping gases may be led into a flue or may be used again in the cycle of operations.—L. A. C.

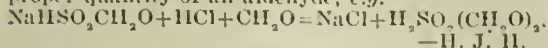
*Potassium chloride; Process for the manufacture of* —. E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 116,438, Oct. 16, 1917. (Appl. No. 14,968 of 1917.)

POWDERED felspar (orthoclase) is heated in a retort or muffle to about 700°–900° C., and arsenic trichloride vapour is introduced into the retort, resulting in the formation of potassium chloride, arsenic trioxide, aluminium silicate, and silica. The arsenic trioxide vapour is condensed and may be used for the preparation of arsenic trichloride. The residue is lixiviated with water and the solution of potassium chloride decanted or filtered and evaporated to dryness. If iron is present, this is separated from the liquor by precipitation with calcium hydroxide; if sodium is present, the mixed chlorides are separated by crystallisation. —L. A. C.

*Dialdehyde sulphylic acids; Production of —.*

A. Bluz, Berlin. Ger. Pat. 303,478, Nov. 3, 1916.

SALTS of monoaldehyde sulphylic acid in presence of acids, or the free acids themselves, are treated with an aldehyde, e.g., formaldehyde, or sodium hydrosulphite in acid solution is treated with the proper quantity of an aldehyde, e.g.



—H. J. H.

*Fullers' earth; Process for increasing the decolorising action of —.* L. Kern, Hamburg.

Ger. Pat. 304,076, Dec. 23, 1916.

THE crude earth is mixed with a dilute acid boiling about 100° C. or below, e.g. acetic, sulphurous, or ultric acid alone or mixed, and dried, whereby the product retains its porosity and low specific gravity. The decolorising action is influenced more by the organic than by the basic inorganic impurities.—H. J. H.

*Hygroscopic substances; Production of crystals of —.*

Elektrochemische Werke G.m.b.H., Berlin, and Dreibrodth, Blitterfeld. Ger. Pat. 304,087, April 27, 1917.

SUBSTANCES such as ammonium nitrate, sodium nitrate, calcium nitrate, and glycollic acid, when crystallised from solutions containing gelatin, egg albumin, casein, or dextrin, yield crystals with a colloidal coating and markedly diminished hygroscopic properties. The utility of ammonium nitrate for manurial and other purposes is increased. Crystals of sodium nitrate obtained in this way, owing to their double refraction, can be used instead of calc spar for optical purposes.—H. J. H.

*Magnesium carbonate and pure carbon dioxide; Production of —.* Chem. Fabrik Buckau,

Magdeburg, and T. Sillermann, Halle. Ger. Pat. 304,681, Apr. 20, 1915.

MAGNESIUM sulphate in solution is treated with quicklime or burnt dolomite to precipitate calcium sulphate, and then with carbon dioxide under pressure to produce a solution of magnesium bicarbonate. After separating the calcium sulphate, the bicarbonate is heated to produce magnesium carbonate and pure carbon dioxide, which can be recovered.—H. J. H.

*Magnesium hydroxide; Preparation of pure — from the magnesium chloride liquors of potash works.* W. Kahn, Nordhausen. Ger. Pat.

304,682, Jan. 27, 1916.

THE combined sulphuric acid is first precipitated by an alkaline-earth sulphide or hydrosulphide and then magnesium hydroxide is precipitated from the resulting clear liquid by the same reagent. The hydrogen sulphide evolved is recovered.—H. J. H.

*Magnesium hypochlorite; Manufacture of basic —.* E. Merck Chem. Fabr., Darmstadt. Ger.

Pat. 305,419, Sept. 1, 1916. Addition to Ger. Pat. 297,874.

MAGNESIUM oxide or hydroxide is treated with a solution of a hypochlorite, or a solution of an alkali or alkaline-earth is added to a solution of a magnesium salt containing hypochlorous acid. The process may be carried out by passing chlorine into a solution containing excess of magnesium oxide or magnesium hydroxide prepared as described above.

—L. A. C.

*Hydrated zinc oxide; Manufacture of technical — free from chlorides.*

Bayerische A.-G. für chem. u. landwirts.-chem. Fabr., H. Hackl, and H. Bunzel, Heufeld. Ger. Pat. 305,738, Dec. 14, 1916.

THE basic salt precipitated by the action of strong bases on a solution of zinc chloride is separated from the liquor, then treated with milk of lime at the boiling point at atmospheric pressure, and washed. The operations are repeated several times, if necessary.—L. A. C.

*Sulphur; Process for the separation of tar from —.*

Norddeutsche Präparaten-Fabr. Heydorn u. Biegel, Harburg. Ger. Pat. 305,418, July 4, 1917.

CRUDE, tarry sulphur is treated with a weak aqueous alkaline solution and then washed with water.—L. A. C.

*Cyanogen compounds; Method for production of —.*

A. R. Lindblad, Ludvika, Sweden. U.S. Pat. 1,264,949, May 7, 1918. Date of appl., June 1, 1916.

SEE Eng. Pat. 107,426 of 1916; this J., 1917, 962.

*Ammonium sulphate; Production of neutral —.*

F. Capron, London. U.S. Pat. 1,266,212, May 14, 1918. Date of appl., Sept. 17, 1917.

SEE Eng. Pat. 108,990 of 1916; this J., 1917, 1093.

*Preparation of readily soluble, soap-like washing tablets from water-glass.* Ger. Pat. 305,461. See XII.*Process for manufacturing calcium arsenate for insecticidal use.* U.S. Pat. 1,265,258. See XIXb.

## VIII.—GLASS; CERAMICS.

*Tempering of muffles for zinc extraction.* Mühlhaeuser. See X.

## PATENTS.

*Glass plates or sheets; Manufacture of —.*

H. K. Hitchcock, Assignor to Hitchcock Experiment Co., Pittsburgh, Pa. Re-issue, 14,468, May 7, 1918, of U.S. Pat. 1,178,448, Feb. 16, 1915. Date of appl., Apr. 3, 1916.

AN apparatus for the manufacture of glass consists of a receptacle for the molten glass, and a drawing orifice with a continuous wall extending from within the glass to a point above the glass level, the area of the orifice gradually increasing from the bottom to the level of the glass so as to permit the surface from which the glass is withdrawn to lose heat by radiation. Means for drawing the glass upwards through the orifice and for regulating the temperature of the walls surrounding the orifice are provided. The restricted portion of the orifice may have the same shape as the finished article.—A. B. S.

*Oven or furnace for baking earthenware.* J.

Litinsky, Elkins, W.Va. U.S. Pat. 1,265,097, May 7, 1918. Date of appl., July 1, 1916.

IN a three-storey, round pottery oven, the primary chamber occupies the middle storey and is separated from the one below it by a thin floor and ceiling through which pass short gas-flues. Fireboxes of the usual pattern are arranged on the circumference



of the middle chamber, and supplementary fire-boxes in the lowest chamber deliver hot gases through perforations in the floor of this chamber, and by means of vertical flues through perforations in the floor of the highest chamber. The two lower chambers may be worked with either up- or down-draught; the highest chamber with up-draught only.—A. B. S.

*Silicon carbide articles; Method of producing* —. O. Hutchins, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,266,478, May 14, 1918. Date of appl., Mar. 3, 1917.

A MIXTURE of silicon carbide with an organic binder, such as pitch, is moulded into a desired form and baked slowly, in the absence of air, to remove volatile matter, leaving a mass composed of silicon carbide in a matrix of carbon. This is heated to the temperature of formation of silicon carbide while exposed to vapours of silicon and silica, when the body is converted into a homogeneous mass of silicon carbide.—H. J. H.

*Tunnel kiln for drying, firing, and cooling.* J. Janka-Vales, Radotin, Bohemia. Ger. Pat. 304,016, Aug. 9, 1916. Under Int. Conv., Feb. 12, 1914.

THE kiln is two storeys high, the ware being dried on the wagon while passing along the upper storey and fired and then cooled in returning along the lower. A fan placed between the drying and the cooling chambers circulates air which passes under the firing chamber through a flue, the roof of which is formed by the floor of the waggon and thence back to the upper storey. Heat radiated by the waggon while in the firing chamber is thereby conducted to the drying room.—H. J. H.

*Refractory material and process for making same.* C. B. Stowe, Lakewood, Ohio. U.S. Pat. 1,265,545, May 7, 1918. Date of appl., Aug. 29, 1917.

ARTICLES formed of a mixture of magnesium carbonate and water, are coated with pulverised iron oxide and burned at such a temperature as to expel the carbon dioxide and water and to cause a partial combination of the iron and magnesium oxides. The iron oxide may be replaced by other metallic oxides.—A. B. S.

*Burning bricks and the like; Method of* —. S. G. S. Dicker, London. From Lambert Process Co., Chicago, U.S.A. Eng. Pat. 116,745, June 15, 1917. (Appl. No. 8602 of 1917.)

SEE U.S. Pat. 1,239,189 of 1917; this J., 1917, 1130.

## IX.—BUILDING MATERIALS.

*Cement clinker; Microscopical examination of* —. Part II. T. Hattori. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 306—314.

TWO samples of cement clinker from a Dietzsch kiln, of similar composition, made from the same materials, A well burnt and B under-burnt, were examined. The alite grains in well burnt clinker from a Dietzsch kiln are intermediate in size between those of well burnt clinker from a stationary kiln and a rotary kiln respectively. The alite grains were large, with square or hexagonal section, in sample A, but were small and round in B. Eutectic structure was found to a much larger extent in the celite of A than in that of B. Celite, etched with alcoholic dilute hydrochloric acid, is seen under the microscope by reflected light to be composed of

white and grey areas, sharply divided and bounded by straight lines. The white area resists the etching reagent but the grey is readily attacked. The value of the hydraulic modulus, as calculated by well-known formulae, only holds good if there is an absence of free lime in the cement, but it is difficult to obtain commercial Portland cement which does not contain free lime. The hydraulic modulus of sample A was lower than that of B, nevertheless according to the microstructure the quality of A was better than that of B. Hence it is misleading to judge the quality of Portland cement by its hydraulic modulus, unless the effect of the presence of free lime be taken into account. (See also this J., 1918, 59 A.)—J. F. B.

*Portland cement; Absorption of carbon dioxide by* — on aeration. T. Hattori. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 297—305.

ABOUT 30 kilos. of Portland cement was placed in a wooden box, 60 × 60 × 20 cm., lined with sheet zinc, stirred every day, and analysed every two weeks for carbon dioxide and loss on ignition. The following results are recorded: Sample A, before exposure, carbon dioxide 0.40, moisture and combined water 1.10%; after 22 weeks' exposure, carbon dioxide 4.57, moisture, etc. 2.61. The results for sample B before exposure were 0.48 and 0.72% respectively, and after 22 weeks' exposure, 4.88 and 2.34%. Under different climatic conditions the same cements gave very much lower results. The graph showing the rate of absorption of carbon dioxide was a straight line.—J. F. B.

*Reinforced concrete v. salt, brine, and sea-water.*

H. J. M. Creighton. Faraday Soc., July 23, 1918. [Advanced proof.] 8 pages.

THE deterioration of reinforced concrete when exposed to sea-water or saline solutions has been attributed to a number of causes, including electrolysis due to the action of a salt solution on the reinforcement. The author has found that when a bar of iron is immersed in a dilute solution of common salt, the salt hydrolyses, the hydroxyl ions uniting with ferrous ions which have passed into solution from the bar and forming undissociated ferrous hydroxide, which is gradually precipitated as a hydrated oxide; small quantities of hydrogen are evolved simultaneously. In this way the bar gradually disappears and hydrated iron oxide accumulates. As both iron oxide and the hydrated oxide occupy a larger volume than the iron from which they were formed, an expansive force is developed which is sufficient to crack the strongest concrete. The rate of deterioration is proportional to the porosity of the concrete. The author examined a number of reinforced concrete floors on which salt and brine solutions had frequently been spilled, and found, on the under sides, iron-stained cracks, which ran parallel to the reinforcing rods; from the larger cracks it was possible to remove iron oxide adhering to the corroded rods. In some cases the deterioration was very serious. In a number of tanks used for storing brine it was found that, after less than one year's use, the strength of the concrete was much below normal. In the prevention of this deterioration of concrete, the customary methods of waterproofing are far from satisfactory; the Schoop process, in which the concrete is coated with a film of metal by spraying, is more promising. Another protective measure, suggested by Gardner (this J., 1915, 434), consists in coating the reinforcement with a paint containing (a) boiled or bodied oils, oils which dry by semi-polymerisation, or oils which dry with a flat surface, (b) coarse pigments which are inert and non-conductors of electricity, preferably those of the

basic or chromate type. In addition, the cement used (especially in the construction of concrete ships) should be finely ground, low in alumina, high in silica, as free as possible from gypsum, absolutely devoid of free lime, slow in setting, and quick in hardening.—A. B. S.

#### PATENTS.

*Fibrous [paring] composition and process for making the same.* G. A. Henderson, St. Albans, W. Va. U.S. Pat. 1,265,655, May 7, 1918. Date of appl., Feb. 12, 1916. (See also U.S. Pat. 1,187,314 of 1916; this J., 1916, 843.)

A MONOLITHIC pavement material is made by mixing 40% of wood, shredded into bevelled, ragged-edged particles of substantially uniform thickness not exceeding  $\frac{1}{8}$  in. and of irregular lengths and breadths between 1 in.  $\times$   $\frac{1}{4}$  in. and  $\frac{1}{2}$  in.  $\times$   $\frac{1}{4}$  in., with 50% of wood shredded into sizes between  $\frac{1}{8}$  in.  $\times$   $\frac{1}{4}$  in. and a cube of diameter equal to the thickness of the particles, and with 10% of wood in shreds varying between that of the largest cube to those of the average thickness.

The material is mixed *in vacuo* with a preheated bitumen of about 70 standard penetration, using about 50% in excess of that required in the finished product, and is then agitated whilst strongly heated to volatilise part of the bitumen and reduce its penetration to about 40, to cause the wood to swell and undergo partial distillation, whereby all moisture, sap-essences, cellular tissue, and pulpy matter may be expelled or destroyed and the pores of the wood impregnated with the molten bitumen. Then, on vibrating the container, the superfluous bitumen drains away and the particles of wood settle horizontally, after which the material is compressed so as to remove the excess of bitumen, leaving only sufficient to fill the pores, when subsequently contracted, to coat the shreds of wood, and to occupy the spaces between them and bind them together. The mass is then cooled and compressed gradually into the desired shapes.—A. B. S.

*Wood; Process for fireproofing* —. C. H. Teedale, Madison, Wis., and R. E. Prince, Elgin, Ill., Assignors to the Govt. and people of the United States. U.S. Pat. 1,265,549, May 7, 1918. Date of appl., Jan. 23, 1917. (Dedicated to the public.)

A PAINT consisting of zinc borate mixed with any other pigment and ground in a drying oil.—A. B. S.

*Wood; Composition for preserving* —. F. Peters, Berlin, Germany. U.S. Pat. 1,265,370, May 7, 1918. Date of appl., Nov. 13, 1914.

A COMPOSITION containing a dinitrophenol or a salt of a dinitrophenol with a monovalent metal, together with a soluble salt of an inorganic acid (such as chromic acid) incapable of attacking iron, and a fireproofing soluble salt.—A. B. S.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Oxygen in iron.* F. Schmitz. Stahl u. Eisen, 1918, 38, 541—542.

The determination of oxygen in iron by heating drillings to redness in a current of pure, dry hydrogen gives only the amount which is present as iron oxide; occluded oxygen and oxides of other elements (manganese, silicon) are not affected. The apparatus recommended for the determination comprises a hydrogen cylinder from which the gas

passes in succession through two wash-bottles containing alkaline pyrogallol and one containing concentrated sulphuric acid, an unglazed porcelain tube containing fine iron drillings heated in an electric furnace, and two U-tubes containing phosphorus pentoxide. The purified gas then passes over the drillings of the sample to be analysed heated in a Mars electric furnace, and through a U-tube with phosphorus pentoxide and a sulphuric acid guard tube. Hydrogen is passed through the apparatus for an hour, after which the temperature of the furnace is raised to 850° to 900° C. in half an hour. The following results of oxygen estimations are given: grey cast iron, 0.001—0.005%; Swedish white iron, up to 0.002%; spiegel, 0.008—0.110%; silicospiegel, 0.037%; ferro-tungsten, 0.058%; high-speed steel, 0.024%; nickel-steel (0.45% C, 25% Ni), 0.020%; ingot iron, 0.031%; crucible steel (1% C), 0.24%; wrought iron, 0.217—0.244%. The oxygen content of basic steel just before the addition of the "killing" elements varied from 0.066 to 0.112%, while after these additions the oxygen present had fallen to 0.014—0.040%. The highest oxygen content ever found by the author in badly overblown basic Bessemer steel was 0.113%, corresponding to 0.51% FeO.—F. C. Th.

*Basic Bessemer process; Non-removal of sulphur in* —. L. Blum. Stahl u. Eisen, 1918, 38, 625—629.

CONTRARY to the experience of Hilgenstock (Stahl u. Eisen, 1893, 455), who found that on the average 58% of the sulphur present in the charge could be removed in the basic converter ( $\frac{1}{10}$ th during the decarburisation,  $\frac{1}{3}$ rd during the after blow and  $\frac{1}{10}$ th by the manganese additions), the author only succeeded in removing one-third. With a mixer iron containing 0.4% Si 28% of the sulphur passed out of the metal, but when 1% Si was present this percentage was much reduced, and at times even an increase of sulphur occurred. This was traced to the lime added, which contained from 0.33 to 0.69% SO<sub>2</sub>. During the first minute or two of the blow, while the lime additions are being dissolved and the silicon is being oxidised, a silicious slag is formed and the following reaction occurs:  $\text{CaSO}_4 + 4\text{Fe} = \text{FeS} + 3\text{FeO} + \text{CaO}$ . As the lime additions become incorporated in the slag this sulphurisation stage passes into one of desulphurisation, which, however, can never be complete; it is most effective during the last few minutes of the blow. A high silicon content in the original charge results in a longer period during which sulphurisation can occur, with a corresponding reduction in the desulphurisation stage. A lime low in sulphur should be used. The percentage desulphurisation is higher as the sulphur content of the charge rises. It is not good practice from the point of view of desulphurisation to produce pig iron high in silicon merely to obtain more complete reduction of the manganese in the ore. The harmful effect of the silicon more than counterbalances any benefit derived from the manganese.—F. C. Th.

*Steel; Influence of forging on the mechanical properties of* —. G. Charpy. Comptes rend., 1918, 167, 12—14.

In many French specifications a "coefficient of working" is embodied which is the ratio of the original section of the ingot to that of the finished article. In general a value of 3 to 4 is prescribed. To determine how far such a practice is sound, experiments were carried out on a pure medium-hard gun steel, on samples taken both longitudinally and transversely. Increasing the "coefficient of (hot) working" from 1.7 to 6.1 produced comparatively little effect on the tenacity or elongation.



in both transverse and longitudinal test-pieces, but the reduction of area, bending angle, and impact value were considerably influenced. In tests made upon the steel in a longitudinal direction, these properties were improved as the degree of hot rolling increased, but in the case of transverse test-pieces exactly the opposite effect was observed. With an increase from 1.7 to 6.1 in the coefficient, the value of the reduction of area fell from 76 to 31% and the impact value from 5.3 to 3.5 kilogram-metres. The influence of the hot working upon the properties of the steel in a transverse direction becomes accentuated in impure, segregated, or porous steels to such an extent that with a certain reduction of section the elongation and impact value fall almost to zero.—F. C. Th.

*Electric steel; Triplex process of making* —. T. W. Robinson. Amer. Iron and Steel Inst., May 31, 1918. Chem. and Met. Eng., 1918, 49, 15—20.

THE development of electric steel is based upon the fundamental conception that the electric furnace is pre-eminently adapted to the manufacture of the highest grade of carbon and alloy steels. Large-scale experiments in making steel by a duplex process, in which Bessemer blown metal was refined in a 15-ton basic-lined Heroult furnace, indicated that electric and open-hearth rails of like chemical composition had practically the same resistance to wear, and that the electric steel was rather more ductile. The duplex process necessitates removal of phosphorus in the electric furnace by the use of an oxidising slag, with subsequent deoxidation by a reducing slag, and while excellent steel can be produced by this practice, there is greater liability to irregularity than when the phosphorus has been eliminated previously. The triplex process is based on producing Bessemer-blown metal, with dephosphorisation in an open-hearth furnace, and final refining in an electric furnace. Close assembly of the various units is essential for rapid and economical practice. The Bessemer-blown metal is poured into a 65-ton ladle and the slag eliminated by nozzle pouring. Dephosphorisation is performed in three tilting open-hearth furnaces, each of which has a hearth area of 892 sq. ft. and is heated by producer gas or tar. The electrical plant consists of three 25-ton Heroult furnaces, with a combined output of about 12,000 tons per month, and experience has justified the erection of furnaces of large capacity. As soon as the steel is dead-melted the current is reduced until a proper pouring temperature is obtained. Stress is laid on careful manipulation of electric steels during and after pouring. Steel produced by the duplex or triplex process is strictly only electrically refined rather than electrically made, and the superiority of electrically produced steel lies in the pure heat of the electric furnace and the accurate control of the furnace atmosphere.—C. A. K.

*Manganese steels as substitute for special steels for machine parts.* T. János. Stahl u. Eisen, 1918, 38, 567—570.

The strength of steels containing 0.7—2.0% Mn increases for the forged state 2.3 kilos. per sq. mm. for each 0.1% Mn. The yield point of such steels melted in the crucible or electric furnace may reach 80—90% of the maximum stress compared with 50—60% in metal melted in the open-hearth furnace. The critical point on heating was determined by quenching a series of specimens of each steel at increasing temperatures and determining the Shore hardness numbers. For purposes of annealing the temperature was chosen which had given the

greatest hardness. The author concludes that as a war-time substitute for special steels in motor-car construction manganese steels containing from 0.7% to 1.6% Mn are the most satisfactory. They do not, however, reach the same high standard as the special steels. The steels need appropriate heat treatment.—F. C. Th.

*Gold recovery; The sodium sulphide process of* —. F. Wartenweiler. Bull. 166, Inst. Min. Met., July 25, 1918. [Advance copy.] 15 pages.

At the Prestea mine (Gold Coast), where the gold occurs together with arsenical and antimonial sulphides in quartz seamed with carbonaceous schist, the table concentrate is roasted, amalgamated, and cyanided; the tailing is classified into sand and slime, the latter being at present run to waste. The former is cyanided and then leached with 0.2% sodium sulphide solution. The gold thus dissolved is not precipitated by the carbonaceous schist; it is recovered by passing the solution through boxes filled with copper shavings. For cleaning up, the contents of the boxes are filter-pressed, dried, and screened through a 14-mesh sieve. The oversize is returned to the precipitation boxes; the undersize is at present roasted and sold for its gold and copper content. The increase in extraction due to sulphide leaching averages 2s. per ton of sand, with a sulphide consumption of 1.2 lb. An aluminium-zinc alloy (50:50), which is far superior to copper as a precipitant for gold, will be used as soon as market conditions permit.—W. R. S.

*[Gold.] Pure zinc dust for precipitation.* R. Wheeler. Eng. and Min. J., 1918, 106, 22.

CONTRARY to the views generally held, pure zinc dust (98—99.8%), as finely ground as possible, is as efficient a precipitant as the 90% product containing lead. The consumption of the pure dust is 30—40% less, while the gold bullion is purer and more easily refined.—W. R. S.

*Copper cathodes; Oxygen and sulphur in the melting of* —. S. Skowronski. Amer. Inst. Min. Eng. Eng. and Min. J., 1918, 106, 15.

WELL-WASHED cathode copper always contains 0.002—0.004% of sulphur as copper sulphate; the other source of contamination of the re-melted product is the fuel. When cathodes are melted under a coke cover, the sulphur content is higher and the conductivity lower than when a charcoal cover is used. It is pointed out that sulphur raises the "set," hence a certain amount of oxygen is required to depress it and so counterbalance the influence of sulphur. When the percentage of cuprous oxide in the bath is reduced by poling, the molten metal absorbs sulphur dioxide more easily with the formation of cuprous sulphide and oxide.—W. R. S.

*Copper; Effect of boiling sulphur on* —. C. R. Hayward. Chem. and Met. Eng., 1918, 48, 650—651.

SMALL cylinders of copper were immersed in boiling sulphur for periods varying from 5 mins. to 5 hours and the shell separated from the core of copper. The loss of copper increased from 15.4% in 5 mins. to 44% in 30 mins., 70% in 1 hr., and 100% in 4 hrs. The copper content of the shells varied from 77.12 to 78.02% (average 77.38%). It is thought probable that cupric sulphide acts as a carrier of sulphur from the bath to the metal. The formula most nearly corresponding with the material produced is  $5\text{Cu}_2\text{S}_2\text{CuS}$ , which contains 77.31% Cu.

—C. A. K.

*Zinc industry; Research preparedness in the —.*  
P. C. Choate. Chem. and Met. Eng., 1918, 19, 20—22.

THE recent over-production of spelter, with consequent fall in price, emphasises the need for the study of efficient and economical treatment of complex zinc ores. Present processes of retort distillation with acid production and leaching are regarded as unsound, but leaching with electrolytic refining will survive owing to the demand for almost pure zinc. Electric furnace distillation has not extended rapidly because of the inefficiency of the process, but a radically different mode of electric smelting, in which the raw ore is treated directly at the mine, without previous roasting, will probably be successful. A process termed "volatile concentration" is advocated in which briquetted ore would be distilled in vertical retorts, heated by gas produced at the plant (preferably by the low temperature distillation of coal), the coke itself being used in the retort charge. Recuperation of waste heat from the retorts is effected by a flash boiler and hot blast stoves. Briquetting of the ore makes for economy in both carbon and zinc; 6% of pitch, 25% of carbon, and up to 10% of caustic lime are added, the lime reducing the zinc content of the residue to 1%. Irrespective of the complex character of the ore. Reversals of usual practice are suggested in that lead and silver are leached electrolytically from the raw zinc blende concentrates, and the sulphur is recovered in the elemental form from the calcium sulphide in the residue instead of acid being made from sulphur oxides in the roaster gases. Conservation of fuel and labour are claimed for each stage, but practical details are not furnished.—C. A. K.

*Muffles for zinc extraction; Tempering of —.*  
O. Mühlhauser. Metall u. Erz, 1918, 15, 202—208.

BEFORE being built into the zinc furnace the muffles are tempered by gentle heat, to drive off the water of constitution of the clay without fusing the binding material. This is done in furnaces heated from below, the hot gases passing up side flues and then into the furnace at the top and away through holes in the floor. The air-dried muffles are packed in this furnace about 2 hours after the last charge has been removed, the door is luted up and the furnace left for about 15 hours, the temperature being distinctly higher than 100° C. The temperature is then gradually raised during 24 hours to 800° C., after which the muffles are removed to the zinc furnace. When tempered the muffles consist essentially of alumina and silica, resulting from the reaction,  $Al_2Si_2O_5H_4 = Al_2O_3 + 2SiO_2 + 2H_2O$ , together with small quantities of iron oxides, burnt gypsum, felspar, etc., which subsequently exert a beneficial influence as fluxes in closing the pores in the tempered muffle. The product should possess some considerable strength, withstand a temperature of 1300° C., and for a short period even 1600° C., and possess a fairly high conductivity for heat. In course of time, as the porous tempered muffle glazes over, the refractory nature of the material decreases, but the mechanical properties improve. The usual life of a muffle is 63 days.

—F. C. Th.

*Zinc dust; Analysis of —.* L. A. Wilson. Chem. and Met. Eng., 1918, 19, 32—34.

THE hydrogen evolution method is found to give the most consistent results for the determination of the metallic zinc content of zinc dust. A small conical flask is fitted with a separating funnel and an outlet tube connected through a two-way tap with a graduated measuring tube. 1 gm. of zinc dust is transferred to the flask, in which is also placed a

piece of sheet platinum and about 5 grms. of ferrous sulphate. The flask and connecting tube are filled with distilled water saturated with hydrogen, and the measuring tube completely filled with a 10% solution of sulphuric acid, also saturated with hydrogen. The system is now completely filled with liquid and ready for the generation of hydrogen. 30 c.c. of 1:1 sulphuric acid is slowly run into the flask from the separating funnel and the gas evolved, together with some solution and a small amount of zinc dust, passes over into the measuring tube, in which the zinc dust, coming in contact with the 10% sulphuric acid solution, dissolves completely. When the reaction is finished water is run from the separating funnel to drive all the gas from the flask into the measuring tube, and the volume of hydrogen is obtained at atmospheric pressure after levelling by means of the levelling bottle. Corrections for barometric pressure and temperature conditions are applied and the percentage of metallic zinc calculated. For a large number of routine determinations a correction tube similar to the measuring tube may be usefully employed.—C. A. K.

*Zinc [in alloys]; Gravimetric and volumetric determination of — as zinc mercury thiocyanate.*  
G. S. Jamieson. J. Amer. Chem. Soc., 1918, 40, 1036—1039.

LUNDELL and Bee's method of estimating zinc in alloys as zinc mercury thiocyanate (Trans. Amer. Inst. Met., 1914, 146) is criticised. The factor for obtaining the weight of zinc should be 0.13115, not 0.1266, since the precipitate has the composition  $ZnHg(SCN)_4$  after being dried at 102°—108° C. Excellent results are obtained by using ammonium thiocyanate instead of the potassium salt. Cadmium, cobalt, copper, bismuth, manganese, and mercurous compounds should be absent. Nickel in small amounts does not interfere appreciably. Ferric compounds in appreciable quantities should be reduced by sulphur dioxide. Arsenious compounds need not be removed. Instead of weighing the zinc mercury thiocyanate the following volumetric method, based on the equation:  $ZnHg(SCN)_4 + 6KIO_3 + 12HCl = ZnSO_4 + HgSO_4 + 2H_2SO_4 + 4HCN + 6ICl + 6KCl + 2H_2O$ , gives trustworthy results. The zinc mercury thiocyanate corresponding with 0.03—0.10 gm. of zinc is precipitated and washed, the filter containing the precipitate is transferred to an 8-oz. titration bottle, a thoroughly cooled mixture of 35 c.c. of concentrated hydrochloric acid and 10 c.c. of water, followed by 7—8 c.c. of chloroform, is added, and a solution of potassium iodate (19.644 grms. in 1000 c.c.; 1 c.c. = 0.00100 gm. of zinc) is at once added rapidly while the bottle is well shaken. When the iodine liberated in the first stage of the reaction has disappeared, the titration is continued slowly, the closed bottle being thoroughly shaken after each addition until the iodine colour has disappeared from the chloroform indicator, which marks the end-point. If more than 50 c.c. of the iodate solution is required, a further 10—15 c.c. of concentrated hydrochloric acid must be added in order to prevent hydrolysis of the iodine monochloride. (See also J. Chem. Soc., Sept., 1918.)—C. S.

*Tin; Determination of — in wolfram ores and concentrates.* A. M. Smoot. Eng. and Min. J., 1918, 106, 25—26.

TWO grms. of the finely powdered ore is digested with 100 c.c. of strong hydrochloric acid at about 50° C. for an hour, with frequent stirring. The liquid is then evaporated to about 50 c.c. and the evaporation continued, after addition of 5 c.c. of nitric acid, to about 15 c.c. The liquid is diluted with 100 c.c. of water, boiled a few minutes,



filtered, and the residue well washed with dilute hydrochloric acid, digested with 15 c.c. of ammonia, and the solution filtered through the same paper; residue and filter are ignited in an iron crucible, and fused with sodium peroxide. The product is leached, and the liquor acidified with hydrochloric acid and 5 grms. of tartaric acid and added to the original acid filtrate. The latter is treated with hydrogen sulphide, the precipitate separated and washed with very dilute sulphuric acid, ignited in an iron crucible, and again fused with peroxide. The melt is leached with water, acidified with hydrochloric acid, and the liquid treated with 5–6 grms. of pure granulated zinc. Before the latter is all dissolved the solution is rapidly filtered through asbestos, the sponge washed with water, returned to the beaker, and the metal dissolved by warming with 100 c.c. of hydrochloric acid (1:1). The liquid is transferred to a conical flask, reduced with nickel coils or iron wire in an atmosphere of carbon dioxide, cooled, and titrated with N/20 iodine solution.—W. R. S.

*Metals; Testing of — by means of X-rays.*  
F. Janus and M. Reppschien. *Stahl u. Eisen*, 1918, 38, 508–514, 533–541, 558–564.

THE authors describe the physical nature, properties, and production of X-rays so far as these are required for the testing of metals. Hard rays produced from, for instance, a Coolidge tube of spark gap 27–42 cm. (equivalent to a potential difference of 180,000 to 240,000 volts) are best suited for the work. Blowholes have been detected in masses of steel 10–12 cm. thick. Visual examination with the fluorescent screen is not possible for sheets of metal thicker than 2 or 3 mm. Larger masses must be investigated photographically, with the aid of a calcium tungstate intensifying screen placed in close contact with the film. To minimise the disturbing effect of secondary radiations the sample should be surrounded by a layer of lead cast around it. This protection must be thicker as the sample increases in size; for very thick specimens a layer 15 to 20 mm. thick is required. For investigating special portions of metal secondary radiations are best excluded by placing a thick lead tube of sufficient diameter above the region with another shorter length between the specimen and the plate. This plate is made with a thick and very rich emulsion, which requires a much longer time of development than an ordinary plate, a glycine developer being recommended. A high tension step-up transformer giving 120,000 to 150,000 volts is far better than an induction coil as a source of current for the Coolidge tube. To minimise risk in such work the tube should be placed in a box with a thick lead lining and operated from another room, a small lead-glass window being provided for observation. The depth of flaws may be measured by making two exposures on the same plate with the tube moved horizontally a known distance. The distance apart of the two shadows then allows the depth to be calculated if the distance of the plate from the anti-cathode of the tube is known.—F. C. Th.

*Gases in metals; Precision method for the determination of —.* H. M. Ryder. *Amer. Electrochem. Soc.*, May, 1918. [Advance copy.] 6 pages.

THE metal in the form of a thin ribbon is mounted in a water-cooled electric-lamp bulb, which is then evacuated. The temperature of the metal is then raised electrically in 50° C. steps to the fusion point, the gases evolved being immediately transferred by means of a diffusion pump to a second bulb. One or two cub. mm. of gas can be analysed in the special apparatus described, which is constructed entirely of hard glass, and has no stop-

cocks or rubber or cement connections. Water and carbon dioxide are first removed by cooling with liquid air, and are then determined by fractional vaporisation and measuring the vapour pressure with a McLeod pressure gauge in the case of carbon dioxide and with a mercury U-tube manometer with an optical lever attachment (Schrader, *Amer. Phys. Soc.*, Dec., 1917) in the case of water vapour. Carbon monoxide, hydrogen, and methane are determined in the known manner by combustion, and nitrogen by difference. A silicon steel commenced to evolve considerable quantities of carbon monoxide at 500° C., and also of hydrogen and nitrogen at 730° C., which is the A2 point of this steel.—F. C. Th.

*Metal spraying process; The Schoop —.* H. Arnold. *Z. angew. Chem.*, 1918, 31, 15–16.

THE specific gravity of metals sprayed with the Schoop apparatus falls as the distance of the object to be coated from the spraying device increases. Thus, taking the specific gravity of molten copper as 8.933, that of copper sprayed on a surface 3 cm. away is 7.823, at 10 cm. 7.51, and at 32 cm. 7.415. For zinc of specific gravity 6.922 in the molten state, the specific gravity of the metal sprayed at a distance of 8 cm. is 6.325, and at 42 cm. 5.903. Much better results are obtained with the Mauser "pistol" using already molten metal. The decrease of specific gravity is probably due to surface oxidation of the fine particles of metal; such oxidation occurs even if inert gases, such as nitrogen, are used for spraying, and is probably caused by oxygen produced by dissociation of water vapour.—F. C. Th.

*[Ore] concentration; Dry sizing as a means of preparing feed for —.* G. V. Bland. *Eng. and Min. J.*, 1918, 105, 908–911.

A PROCESS of ore treatment is described involving dry sizing, followed by wet concentration. An increase of 20–25% in recovery at a relatively small cost has resulted. The main benefits derived from the dry sizing are:—control of pulp dilution; independence of all important concentrating units; greater efficiency of sizing machinery as compared to hydraulic; and greater efficiency of concentrating units on dry-sized feed.—F. C. Th.

*Determination of molybdenum in presence of copper.* Hoepfner and Binder. *See XXIII.*

#### PATENTS.

*Open-hearth steel furnaces; Process for removing molten slag from — by means of air or steam blast.* C. H. F. Bagley, Stockton-on-Tees. *Eng. Pat.* 116,663, Jan. 12, 1918. (Appl. No. 747 of 1918.)

MOLTEN slag is removed from the surface of a bath of metal by means of a blast of air or steam directed on to the surface of the metal in such a way as to blow the slag to one end of the furnace, whence it flows away through suitable outlets.—C. A. K.

*Iron; Process for rendering — resistant to the action of organic acids, especially picric acid.* C. Bunge, Witten. *Ger. Pat.* 303,126, Oct. 18, 1916.

IRON is treated with nitric, chloric, or chromic acid and washed with water, or oxygen is liberated electrolytically in contact with the iron. Iron rendered passive towards nitric acid is also passive towards picric acid, cresylates, and nitro-compounds. The process is applicable when the formation of picrates and picramates is undesirable, as in the components of shells and in shell-filling plant.

—H. J. H.

*Iron ores containing manganese; Treatment of —.*  
F. Wüst and R. Ruer, Aachen. Ger. Pat. 303,507,  
Apr. 24, 1917.

WHEN iron and manganese are present in an ore as homogeneous mixtures—solid solution—they cannot be separated magnetically. The separation can be effected by treating the ore with a gaseous reducing agent under such conditions that only the iron is reduced and then smelting with fluxes so that pig iron is produced, while the manganese passes into the slag, which is worked up separately in a blast furnace to ferromanganese.—H. J. II.

*Ferrophosphorus from iron phosphatic material; Process of making —.* Process of producing ferro-phosphorus in electric furnaces. Process of producing phosphor-copper compounds. J. J. Gray, jun., Rockdale, Tenn. U.S. Pats. (A) 1,265,076, (B) 1,265,149, (C) 1,265,460, May 7, 1918. Dates of appl., Aug. 2, Oct. 13, and Dec. 28, 1916.

(A) CRUDE iron phosphate is mixed with a suitable fluxing material and excess of carbon, and heated in a blast furnace to reduce the phosphate to iron phosphide, with separation of the impurities as slag. The resulting ferrophosphorus is said to contain more than 15% P. (B) A mixture of subdivided phosphate rock with silica in excess and carbon is heated between 1000° C. and 1750° C. in a rotary furnace in contact with air, and the resulting ferrophosphorus is separated from the slag. (C) The method described in (B) is applied to the manufacture of phosphor-copper compounds from compounds containing copper and phosphorus, using air heated above 620° C.—C. A. K.

*Alloys containing nickel; Manufacture of —.*  
Stabbliment "Blak" Ing. A. Pouchain, Assignee of P. Peynetti, Turin, Italy. Eng. Pat. 111,290, Nov. 5, 1917. (Appl. No. 16,131 of 1917.) Under Int. Conv., Oct. 23, 1916.

INSTEAD of making a direct addition of nickel in the preparation of an alloy, a nickel-zinc alloy of any suitable composition is used for admixture with the other constituents of the alloy. Homogeneous ingots free from blisters and oxides are said to be produced by this method.—C. A. K.

*Metals; Electrically fusing and depositing —.*  
E. H. Jones, London. Eng. Pat. 116,308, Apr. 12, 1917. (Appl. No. 5117 of 1917.)

AN electric arc is struck between the object on which the metal is to be deposited and an electrode comprising a mild steel containing all the materials necessary for the deposition of high-speed steel, covered by slag-forming material, such as asbestos yarn. For example, the electrode may consist of W 22, Cr 9, C 1.3, V 0.25, Fe 67.45%. By the use of these electrodes the cutting edges of high-speed or other tools may be renewed or the entire tool may be cast in a mould of the desired shape.—F. C. TH.

*Copper tubes; [Electrolytic] production of —.*  
S. O. Cowper-Coles, Sunbury-on-Thames. Eng. Pat. 116,509, Mar. 6, 1918. (Appl. No. 936 of 1917.)

COPPER is deposited electrolytically on a vertical rotating mandrel, the lower end of which is supported in a recess in a block of wood or other insulating material, the anode being of impure copper cast in the form of rough tubes or rings, or a cylindrical grid, supported concentrically with the mandrel.—B. N.

*Annealing furnaces; Continuous —.* Gibbons Bros., Ltd., and M. van Marle, Lower Gornal, Staffs. Eng. Pat. 116,605, Aug. 15, 1917. (Appl. No. 11,718 of 1917.)

FOR the annealing of brass or other metal tubes and bars, and of small articles which can be enclosed in tubes, externally heated, a retort is provided with cooled mouthpieces furnished with stuffing-boxes through which the tubes are pushed. Air is excluded from the retort by means of steam under pressure. A series of tubes of the same diameter may be connected together by double-ended plugs so that continuous movement may be effected, and steam may be admitted to or formed in the interior of the tubes so as to preserve a bright surface.—C. A. K.

*Crucible furnaces.* J. and G. Keith, London. Eng. Pat. 116,635, Oct. 20, 1917. (Appl. No. 15,254 of 1917.)

A SEPARATE receptacle to catch spilt metal from the crucible is provided beneath the furnace. It has a removable cover, and communicates with the lower part of the combustion chamber by a vertical pipe, the lower end of which is sealed by a lute on the cover of the catch pot.—C. A. K.

*Hot blast stove.* J. I. Lorimer, Joliet, Ill. U.S. Pat. 1,265,923, May 14, 1918. Date of appl., Mar. 6, 1917.

A NUMBER of passes in a hot blast stove is obtained by providing a dome at the top of the stove and a wall or combination of walls intersecting the space within the dome, and arranged at an oblique angle to the flow of gases so as to deflect them from one pass into the next pass.—C. A. K.

*Heat treatment of metal objects; Process and apparatus for —.* C. Napier, Assignor to Napier Saw-works, Inc., Springfield, Mass. U.S. Pat. 1,265,944, May 14, 1918. Date of appl., Jan. 21, 1918.

METAL objects are connected by temporary links of relatively considerable mass so as to form a chain which is drawn through a heated chamber and then subjected to suitable cooling conditions. The portions of the objects adjacent to the links are prevented from cooling rapidly and are hence left in the annealed condition.—C. A. K.

*Ore crushing and amalgamating mill.* D. W. Church, San Diego, Cal., Assignor to R. A. Dal-lugge, Los Angeles, Cal. U.S. Pat. 1,265,496, May 14, 1918. Date of appl., Nov. 22, 1916.

THE pan of a grinding mill of the edge-runner type is in the form of an annular trough having an inclined inner wall, and adapted to contain mercury. The edge-runners are supported above the bottom of the pan and away from the outer wall, so as to run only on the inclined inner wall, with freedom of movement in a vertical direction.—C. A. K.

*Smelter smoke; Process for rendering — innocuous.* W. Küstermann, Burgörner. Ger. Pat. 302,996, May 1, 1914.

THE smoke is pumped through a bed of hot coke and then up a tower scrubber down which water, acid, or tar is sprayed. A curtain dipping into the liquid at the base of the scrubber forms a seal through which the smoke must bubble. The pump consists of a U-tube containing water which is caused to rise and fall by the motion of a piston in one limb.—H. J. II.



*Metal electrode used in electric-arc deposition of metals.* E. H. Jones, London. U.S. Pat. 1,265,453, May 7, 1918. Date of appl., Sept. 29, 1917.

SEE Eng. Pat. 109,652 of 1916; this J., 1917, 1135.

*Lead; Extraction of — from its ores or lead-containing materials.* H. L. Sulman and H. F. K. Picard, Assignors to The Metals Extraction Corporation, Ltd., London. U.S. Pat. 1,265,547, May 7, 1918. Date of appl., June 28, 1917.

SEE Eng. Pat. 109,817 of 1916; this J., 1917, 1181.

*Machines for grinding ores, minerals, stones, and the like.* Eng. Pat. 116,583. See I.

*Regenerative gas furnaces.* Eng. Pat. 116,870. See IIA.

*Process for producing alkali metals or the like.* U.S. Pat. 1,265,360. See VII.

## XI.—ELECTRO-CHEMISTRY.

*Electrolytic preparation of p-aminophenol.* Shōji. See III.

*Oxygen and sulphur in the melting of copper cathodes.* Skowronski. See X.

### PATENTS.

*Electrodes; Manufacture of carbon —.* C. H. Thompson, Stourbridge. Eng. Pat. 116,661, Dec. 24, 1917. (Appl. No. 19,065 of 1917.)

ANTHRACITE, which has been previously heated to a high temperature to drive off volatile matter, is mixed with a binding material, moulded, and then heated in a muffle for some time, surrounded by anthracite to be used later in the manufacture of the electrode material.—B. N.

*Insulating materials [from pitch]; Electric —.* A. R. Müller, Walthamstow. Eng. Pat. 116,532, June 11, 1917. (Appl. No. S343 of 1917.)

A MIXTURE of hard pitch and asbestos is heated to from 400° to 800° C., and then submitted to pressure in the heated condition.—B. N.

*Electrolytic apparatus.* C. H. Thomson, Maryville, Tenn. U.S. Pat. 1,265,551, May 7, 1918. Date of appl., April 7, 1917.

THE apparatus consists of a series of pots each provided with several electrodes supported from above, each pot being connected with the electrode support of the next by means of conductors. The conductors from the several pots are separated by and clamped to insulators.—D. F. T.

*Electric furnace.* H. Nathusius, Friedenshütte, Germany. U.S. Pat. 1,265,945, May 14, 1918. Date of appl., Jan. 15, 1914.

SEE Eng. Pat. 13,951 of 1914; this J., 1916, 547.

## XII.—FATS; OILS; WAXES.

*Olive oil; Stability of —.* E. B. Holland, J. C. Reed, and J. P. Buckley, jun. J. Agric. Res., 1918, 13, 353—366.

SAMPLES of a pure Californian olive oil were placed in glass bottles, some of which contained 5 c.c. of water, whilst from others moisture was excluded. In some of the experiments the oil was heated at 70° C. on successive days to destroy enzymes, if present. From an examination of the samples at intervals up to six years the following conclusions were drawn:—Air by itself causes slow destruction of the colour of olive oil, and slow production of rancidity and decomposition of unsaturated fatty acids, whilst light alone destroys the colour more rapidly than air, but has less effect in developing rancidity. The combined action of light and air has the greatest effect in destroying the colour, rapidly decomposing the unsaturated fatty acids, and producing rancidity, whilst it causes a slow but pronounced liberation of free fatty acids. Moisture has no effect on the colour and no appreciable influence on the development of rancidity, but it causes an oil to become turbid and yield a precipitate, and effects slow hydrolysis, with the liberation of a considerable amount of free fatty acids. The combined action of air and moisture, and of light and moisture is essentially that of the individual factors, whilst the combined action of the three factors is practically the same as that of the separate individuals, except that a greater proportion of free fatty acids is produced. Hence, to preserve olive oils in their natural condition it is essential to exclude air, light, and moisture as completely as possible.—C. A. M.

*Castor oil; Determination of the purity of —.* C. Frabot. Ann. Chim. Analyt., 1918, 23, 120—125.

THE author agrees with Chercheffsky (this J., 1918, 273 A) that the solvent power of petroleum spirit varies, but maintains that the determination of a foreign oil in castor oil by extraction with petroleum spirit (this J., 1918, 98 A) is sufficiently accurate provided that a control experiment with an oil mixture of known composition is made at the same time. Extraction with petroleum spirit has also the advantage that it enables the nature of the foreign oil to be determined.—W. P. S.

*Pumpkin seed oil; Manufacture of —.* E. W. Albrecht. Z. angew. Chem., 1918, 31, 132.

AT an oil factory in Roumania, sunflower seeds and pumpkin seeds have been employed with much success when the usual raw materials were scarce. A special kind of pumpkin is grown locally, which contains very many seeds and very little pulp, and which is used for feeding cattle and pigs. The seeds are always obtained in a very moist condition, and must first be dried thoroughly, then crushed between a pair of rollers and extracted with petroleum spirit. A yield of 40—42% of a green oil is obtained from the dry seeds, which is very suitable for the manufacture of soaps and solid engine greases; when refined it affords a good edible oil of pleasant flavour. In Hungary the pumpkin-seed oil is obtained by pressing. Sunflower seeds treated in the same manner yield 32—33% of oil; this oil is used for all industrial purposes, including the manufacture of varnish; in Russia it is largely employed as an edible oil. If the above seeds are to be treated by the pressing method, it is advisable to dry them very thoroughly and to remove the shells. The pressed cakes containing about 8% of oil are broken up and extracted by solvents.

—J. F. B.

*Rancidity; Chemical tests for the detection of* — R. H. Kerr. *J. Ind. Eng. Chem.*, 1918, 10, 471–475.

KREIS' test, which depends on the red coloration obtained when the fat is shaken with resorcinol dissolved in hydrochloric acid (see this J., 1904, 1001), is extremely sensitive, and its chief value is in detecting incipient rancidity which is not obvious to smell or taste. Issoglio's "oxidisability" method (this J., 1916, 898) yields less definite information, but it is useful as a confirmatory test. A low "oxidisability" value is not evidence of the absence of rancidity.—W. P. S.

*Fats; Action of symbiotic bacteria on the constituents of* — H. Pierry and P. Portier. *Comptes rend.*, 1918, 166, 1055–1057.

The symbiotic bacteria isolated from the testicle of a pigeon oxidise glycerol giving a C<sub>2</sub>-sugar, and also they cause the  $\beta$ -oxidation of butyric or  $\beta$ -hydroxybutyric acid. (See also *J. Chem. Soc.*, 1918, I., 358.)—W. G.

*Tussah chrysalis; Oil of the* — S. Kobayashi. *Kōgyō-Kwagaku-Zasshi* (*J. Chem. Ind.*, Tokyo), 1918, 21, 321–326.

TUSSAH silk is largely cultivated in Shantung, and the yearly production of chrysalis is estimated at about 48,000 tons. The author has examined the oil extracted by ether from the chrysalides, of an average weight of 5.8 grms. and the following composition: water, 80.41; crude fatty oil, 1.41 (7.2% of the dry substance); nitrogen, 2.66; ash, 1.23%. The oil had a clear, light brownish-red colour and the following characteristics: sp. gr. at 10°/4° C., 0.9263; saponification value, 185.6; acid value, 53.91; iodine value (Wijs), 124.21; refractive index (20° C.), 1.4763; butyro-refractometer reading (20° C.), 76.3. The m.p. of the fatty acids was 30.5°–32.5° C., and the amount of bromide insoluble in ether, 28%, m. pt. 178.5°–180° C. The unsaponifiable matter contained a sterol (possibly phytosterol) and a waxy hydrocarbon.—J. F. B.

*Oil; Reaction of the aqueous medium in the enzymic hydrolysis of* — Y. Tanaka. *Kōgyō-Kwagaku-Zasshi* (*J. Chem. Ind.*, Tokyo), 1918, 21, 112–117.

IN 1910, the author showed that extracted castor beans, treated with acid and washed, showed a maximum lipolytic activity in neutral aqueous media, and that the function of the acid was to develop the enzyme from its zymogen (this J., 1910, 1259; 1912, 884). These results were further confirmed by Armstrong and Gosney (this J., 1913, 917). Recently Kita (this J., 1918, 312A) found that an activated lipase exerted its maximum effect in an acidified medium. It would appear, however, that Kita employed a lipase containing zymogen which had not been fully activated by the previous acid treatment, and that the acidity of the medium had a further action on the zymogen during hydrolysis. The author has repeated his experiments by triturating 100 grms. of pressed castor beans with 800 c.c. of N/10 acetic acid, washing and drying the residue. Hydrolysis of soya bean oil with this powder at 38°–39° C. in presence of increasing quantities of N/10 sulphuric acid confirmed the fact that the most energetic action took place in presence of pure water, and that the addition of small amounts of acid had a retarding influence proportional to the amount of acid present.—J. F. B.

*Beeswax from Ogasawara (Bonin) Islands*. S. Ueno. *Kōgyō-Kwagaku-Zasshi* (*J. Chem. Ind.*, Tokyo), 1918, 21, 319–320.

THE author has examined the wax of *Apis mellifica* L., var. *ligustica*, from Ogasawara Islands: The

refined wax was light yellow in colour and more viscous than the Japanese and Korean varieties; the chemical characteristics were also different. The following data are recorded: Sp. gr. at 100°/4° C., 0.8096; m.p. (capillary tube method), 65.5° C.; refractive index (measured at 65° C. and reduced to 40° C.), 1.4518; reading in butyro-refractometer (ditto), 44.4°; acid value, 18.71; ester value, 70.03; saponification value, 88.74; iodine value (Wijs), 6.93.—J. F. B.

*Determination of water [in soap, etc.] by distillation*. Besson. *See* XXIII.

#### PATENTS.

*Oils, fats, and the like; Apparatus for distilling* — E. C. R. Marks, London. From Soc. Franco-Belge de Fours à Coke, Brussels. *Eng. Pat.* 116,309, Apr. 14, 1917. (Appl. No. 5290 of 1917.)

DISTILLATION of fatty or mineral or tar oils, etc., is effected in an apparatus containing one or more chambers through which circulate heating gases, each chamber containing a bundle of tubes provided with means for the admission of steam, and connected at one end with a box for the introduction of the liquid to be distilled, and at the other with a box for the collection of the distillate. By admitting gas from a furnace separately into each of a number of the chambers, the distillation may be carried out as a continuous fractionating process.—C. A. M.

*Fats; Apparatus for and method of recovering* — F. B. Dehn, London. From National Sanitary Service Co., Chicago, U.S.A. *Eng. Pat.* 116,384, July 3, 1917. (Appl. No. 9591 of 1917.)

FAT-CONTAINING liquids, and, in particular, slaughter-house liquid refuse, is forced as a progressively accelerating transverse current beneath a larger quiescent volume of liquid, whereby the fat rises to the surface, whilst the solid impurities subside and are drawn off as a sediment. Apparatus for this process comprises a tank divided into two sections, each having an inlet at one end and an outlet, at higher level, at the opposite end. The first section, in which most of the sediment collects, contains an inclined deflecting plate between the inlet and outlet, and its lower portion is relatively larger than the upper. In the second section the entering liquid is forced by a baffle to flow along the sloping bottom, and then passes under another baffle at the opposite end of the tank and rises to the outlet. Both sections are provided with drain pipes controlled by valves.—C. A. M.

*Catalyst [for hydrogenating oils]; Production of* — from metal salts. Bremen-Besigheimer Oelfabriken, Bremen. *Ger. Pat.* 304,043, Aug. 18, 1912.

KIESELGUHR or asbestos is impregnated with a solution of a salt of nickel, e.g., the acetate, and a limited quantity of oil is incorporated with the mass, which is then heated in a vacuum at 150°–200° C. Hydrogen is passed through towards the end, and the mass is not only dried, but a portion of the volatile acid which would otherwise be set free during the reaction is removed. The resulting catalyst retains its activity for a long time.

—H. J. H.

*Washing tablets from water-glass; Preparation of readily soluble soap-like* — A. Kaufmann und Co., Asperg. *Ger. Pat.* 305,461, Oct. 15, 1916.

WATER-GLASS is heated with a small percentage of a salt which readily takes up water, e.g., anhydrous sodium sulphate, and the product formed into tablets.—L. A. C.



*Soap; Process for deodorising* —. C. Stiepel, Berlin-Steglitz. Ger. Pat. 305,702, Oct. 11, 1916.

A CURRENT of steam is passed under pressure into a solution of the soap at 200° C. or over until the issuing vapour is free from odour.—L. A. C.

*Process for treating {deodorising} naphthenic acids.* Ger. Pat. 305,771. See IIa.

*Process for increasing the decolorising action of fullers' earth.* Ger. Pat. 304,076. See VII.

*Process and apparatus for treating soya beans.* Eng. Pat. 116,158. See XIXa.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Oil varnishes; Colour designation of* —. F. A. Wertz. J. Ind. Eng. Chem., 1918, 10, 475—476.

THE colour of oil varnishes may be satisfactorily matched by solutions of potassium bichromate in strong sulphuric acid (sp. gr. 1.84). For example, standards Nos. 2 to 10 of the 10-point scale used by varnish manufacturers may be represented by solutions of bichromate containing respectively 0.1, 0.25, 0.35, 0.5, 1, 1.5, 2, 4, and 8 grms. or more in 100 c.c. of sulphuric acid. The liquids are compared by transmitted light, in tubes of 1 to 2 cm. diameter, in quantities of not less than 2.5 c.c. Heating the solution to prevent deposition of chromic anhydride does not have any appreciable effect on the colour comparison.—C. A. M.

#### PATENTS.

*Coating by means of emulsions; Process of* —. B. Smith, Glens Falls, N.Y., Assignor to General Electric Co. U.S. Pat. 1,266,335, May 14, 1918. Date of appl., Nov. 6, 1917.

THE oil phase is separated from the water phase of an emulsion by bringing the emulsion into contact with a solid object heated to a temperature above 100° C.—J. H. P.

*Varnish-gums; Process of melting* —. O. C. Griem, Assignor to Berry Brothers, Detroit, Mich. U.S. Pat. 1,265,326, May 7, 1918. Date of appl., Nov. 17, 1917.

VARNISH gums are covered with water and heated until the particles are softened and flow together; the water is then poured off and the heating is continued until the gums are completely melted. The process ensures the protection of the particles of gum from the air during the former operation.

—D. F. T.

*Process [paint] for fireproofing wood.* U.S. Pat. 1,265,549. See IX.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Depolymerisation of raw* —. K. Kawakami. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 226—231.

THE author criticises and points out inaccuracies in the experiments by Takeuchi (this J., 1918, 313 A), but he agrees with the general proposition that viscosity measurements may be of value in controlling the processes of manufacture. The viscosity is not an absolute function of the depolymerisation,

since it is affected also by the non-caoutchouc matters, e.g., proteins and rubber resins, which will not change in the same proportion as the caoutchouc. The degree of variation of the acetone extract during mastication falls within the limits of experimental error. The viscosity of raw rubber cannot be used as a sole measure of valuation, because there are several factors of value which have nothing to do with the viscosity of the solution; for instance, the amount of impurities and loss in washing, also the physical and chemical properties of the vulcanised products. The proper times of mastication obtained from Takeuchi's curves, on the basis of an absolute viscosity of 0.1 in the rubber solution, work out at 40 mins. for plantation and 80—90 mins. for Brazilian Hevea, but these times appear far too long from a technical point of view.

—J. F. B.

*Vulcanisation process; Investigations on the* —. A. van Rossem. Communications of the Netherlands Govt. Inst. for advising the Rubber Trade and Rubber Industry. Internat. Assoc. for Rubber Cultivation in the Netherlands Indies, Part VI., 179—222.

RUBBER can be submitted to very considerable mastication without its rate of vulcanisation being appreciably affected, and in a mixing containing 92½% of rubber and 7½% of sulphur after various degrees of mastication, combination of sulphur and rubber occurred at the same rate, although the relative viscosity of the 5% solutions in benzene ranged from 177 to 63. The reduction of the breaking load and elongation observable after prolonged mastication of the mixing before vulcanisation is not due to alteration of the position of the stretch-strain curve, but only to a recession of the end-point of the curve. With the same mixing the quantity of sulphur which enters into combination is approximately proportional to the period of vulcanisation at any one temperature, whilst with mixings containing 2—10% of sulphur the percentage combined under a constant period of vulcanisation is approximately proportional to the original sulphur content. Between 130° and 150° C. the temperature coefficient for the vulcanisation process has a value between 2 and 3. Rubber showing a high viscosity number tends to give a stronger product than one of lower viscosity, not only because it suffers less depolymerisation under the same degree of working, but also because it vulcanises more rapidly. The resinous constituents of rubber are not essential to vulcanisation, although they appear to exert a slight acceleration on the process; their presence also causes a slight reduction in the breaking strength, which, however, may not be as great as the reduction due to the effect of the treatment necessary for their extraction. There is no proportionality between the percentage of nitrogen and the rate of vulcanisation of Hevea rubber. The introduction of inert fillers, such as powdered flint or pumice, does not alter the rate of combination with sulphur, but many basic compounds have a marked accelerating effect, whilst other substances, notably such as are acidic, have a retarding influence. Of *p*-nitrosodimethylaniline, piperidine, and a proprietary article consisting of a condensation product of acetaldehyde and ammonia, the first is the most effective accelerator with the standard mixing containing 7½% of sulphur, and the effect appears to be greater with rubbers of low viscosity. Raw rubber which has been heated for four hours in an atmosphere of carbon dioxide, when subsequently mixed with sulphur vulcanises more rapidly than a mixing made with rubber which has not been so heated; but no such difference is observable if the rubber is heated in air. A weak Hevea rubber of low viscosity by prolonged vulcanisation can be made to yield as strong a product as a strong

rubber, without danger of over-vulcanisation due to the longer duration of the process. Although the introduction of inert solid fillers does not appreciably affect the rate of combination of sulphur with rubber, it does affect the mechanical properties of the product, which is rendered somewhat stiffer, but the chief result is a decline in the elongation and strength at break. On the other hand soft fillers, such as paraffin wax, have an unfavourable influence on the mechanical properties of vulcanised rubber. When an accelerator, such as *p*-nitrosodimethylaniline, is used, not only is the rate of combination of the rubber with sulphur increased, but the product is stiffer and yields a stress-strain curve showing a decidedly reduced extensibility for the degree of vulcanisation attained, although with sufficient load it is capable of being stretched to an even greater extent than rubber vulcanised under ordinary conditions; with the use of additional catalysts, therefore, the coefficient of vulcanisation is no longer an indication of the probable course of the elongation curve. When rubber mixed with sulphur is heated for only a short time by steam under 50 lb. pressure, the product may be completely dissolved in the usual solvents, but the relative viscosity is greatly increased, a similar although less marked effect, greater than could be attributed to the mere "recovery" of the mixing, being observable on storing the mixing for several months at the ordinary temperature. On further vulcanisation the rubber becomes insoluble, the elongation at break attaining a maximum at a vulcanisation coefficient of approximately 2, rubber vulcanised to a less extent than this often being porous; when the coefficient exceeds 5 the effect of over-vulcanisation is observable, the rubber exhibiting brittleness when stretched, and for practical purposes with rubber-sulphur mixings a maximum coefficient of 2-4 is desirable, which is below that of maximum breaking load. When vulcanised rubber is stored at the ordinary temperature, the mechanical properties undergo considerable alteration without any appreciable concomitant increase in the amount of combined sulphur.—D. F. T.

#### PATENTS.

*Latex; Processes and apparatus for drying coagulated and uncoagulated — and products obtained thereby.* W. J. Mellersh-Jackson, London. From General Rubber Co., New York. Eng. Pat. 116,324, June 1, 1917. (Appl. No. 7899 of 1917.)

THE excessive foaming which occurs on attempting to dry latex under reduced pressure can be prevented by rendering the nitrogenous constituents of the latex insoluble prior to drying. This may be effected in a number of ways, such as heating the latex before or after coagulation to 80°-85° C.; treating the latex with suitable enzymes or ferments, such as rennet, or with chemicals which precipitate nitrogenous material. Antiseptics, *e.g.*,  $\beta$ -naphthol (0.2%), which aids in rendering the nitrogenous material insoluble, or sodium naphtholate, or pine-tar creosote dissolved in sodium cresylate (0.5%), or sodium fluoride (0.1%), may be added before or after insolubilisation. The latex or coagulum is then dried under reduced pressure on a series of trays in a current of air de-oxygenated by passage over burning charcoal.—E. W. L.

*Rubber, rubber-like and similar vulcanisable material; Treatment of unvulcanised —.* W. J. Mellersh-Jackson, London. From General Rubber Co., New York. Eng. Pat. 116,322, June 1, 1917. (Appl. No. 7885 of 1917.)

UNVULCANISED rubber, rubber-like or similar vulcanisable material is subjected to a vacuum, with or without heating, in order to remove moisture,

*etc.*, and the vacuum is subsequently broken by the introduction of an inert substance, liquid or gaseous (*e.g.*, carbon dioxide, ammonia, hydrogen, or nitrogen), which fills the cells or pores of the substance, thereby preventing oxidation.—E. W. L.

*Hard-rubber substitute; Method of making a — and the product thereof.* P. Pickl, Assignor to E. Pickl, Chicago. U.S. Pat. 1,265,108, May 7, 1918. Date of appl., Apr. 23, 1917.

A SUBSTITUTE for hard rubber is prepared by heating elaterite with 3-5% of resinous material and 2-3% of sulphur.—D. F. T.

*Plastic composition [rubber substitute].* C. B. Wood, Charlestown, W.Va. U.S. Pat. 1,265,855, May 14, 1918. Date of appl., July 1, 1914.

SHELLAC is dissolved in strong alcohol to form a thick paste; this paste is poured on to fibrous material arranged in layers; the ingredients are thoroughly mixed together and subjected to pressure, and the pressed material is saturated with formaldehyde.—J. F. B.

*Manufacture of plastic masses resembling horn or ebonite [from yeast].* Ger. Pats. 302,931, 302,930, and 303,123. See XV.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Application of leaves of Acer glinnala in Korea.* Ueda. See VI.

#### PATENTS.

*Leather; Dyeing or staining and mordanting of — [with titanium salts].* H. Wrigley, H. Spence, and P. Spence and Sons, Ltd., Manchester. Eng. Pat. 116,123, May 25, 1917. (Appl. No. 7522 of 1917.)

LEATHER is treated with a solution of titanous acid in sulphuric acid or hydrochloric acid in which the ratio of  $\text{SO}_3$  is greater than 2 mols., preferably 4-9 mols. (or the equivalent of hydrochloric acid) to one mol. of  $\text{TiO}_2$ . The operation is carried out at a dilution equal to about 1 part of  $\text{SO}_3$  per 1000 of water, and the bath is heated to about 48° C. Example: Titanium sulphate containing 121 grms. of  $\text{TiO}_2$  and about the same weight of  $\text{SO}_3$  is dissolved in a mixture of 800 c.c. of strong sulphuric acid with 1 gall. of water, and the liquid is added to a bath of 200 galls. of water at 48° C. in which the skivers are agitated for about 30 mins., the completion of the action being judged by the colour.

—J. F. B.

*Tanning animal hides; Process of —.* Badische Anilin und Soda Fabrik. Ger. Pats. (A) 304,859, Mar. 14, 1915 (Addition to Ger. Pat. 281,484), and (A) 305,777, Feb. 9, 1915 (Addition to Ger. Pat. 290,965).

(A) PRODUCTS soluble in water and useful for tanning purposes are obtained by the introduction of sulphonic acid groups into naphthol pitch. (B) According to the main patent sulphonic acids of substances of high molecular weight are employed, which contain several benzene and naphthalene rings in their structure, also those containing such ring nuclei joined together with intermediate carbon atoms, provided they fulfil the following conditions: that they contain no phenolic hydroxyl and that they possess the property of precipitating glue and gelatin solutions. The addition patent provides for the use of products of this class which contain two or more aromatic nuclei, all or some of which are different from the others. Example: a condensation product of  $\beta$ -naphthalenesulphonic acid with benzyl alcohol.—J. F. B.



*Leather substitute; Manufacture of a —.* O. Steiner, Frankenberg. Ger. Pat. 304,497, May 12, 1917.

Nets made of spiral springs filled with cellulose are boiled in tar, dried, then coated with cellulose mixed with dissolved resin and finally dried under pressure. Belts and soles so prepared possess the pliability and tensile strength of leather.—J. F. B.

*Plastic masses resembling horn or ebonite; Manufacture of — [from yeast].* H. Blücher, Leipzig, and E. Krause, Steglitz. Ger. Pats. (A) 302,931, Nov. 4, 1914 (Addition to Ger. Pat. 275,857), (B) 302,930, May 20, 1915, and (C) 303,133, May 12, 1916 (Additions to Ger. Pat. 289,597). (See also this J., 1915, 223, 972; 1916, 322.)

(A) YEAST or yeast residues are mixed with tar or tar oils, with or without the addition of aldehydes. Example: 1 kilo. of pasty yeast containing 15% dry substance, in which the yeast has been killed by heating, is stirred with 150 grms. of 40% formaldehyde and 15 grms. of tar. The mixture is dried, powdered, and pressed into heated moulds at 80° C. under a pressure exceeding 180 atmos. The tar may be replaced by anthracene oil. If desired 0.5% of washed and powdered sulphur may be mixed with the dried powder before pressing. (B) The yeast is previously dried and then treated with formaldehyde in the form of vapour, solution, or polymerised product. Substances are added which either take part in the reaction with the formaldehyde or serve merely as fillers; in the former case the substances may be previously treated by themselves with formaldehyde. The process of drying the yeast by heat induces certain changes in the proteins, so that the products have different properties from those formed by the action of formaldehyde on moist yeast. (C) Similar masses are prepared from beer sediments, the materials being ground, dried, and pressed under a pressure of 300 atmos. at 90° C.

—J. F. B.

*Tanning hides; Method of —.* G. Bosio and A. Peradotto, Turin, Italy. U.S. Pat. 1,266,203, May 14, 1918. Date of appl. Apr. 19, 1916.

SEE Eng. Pat. 16,764 of 1915; this J., 1916, 748.

## XVI.—SOILS; FERTILISERS.

*Organic matter in soils; Determination of — by the loss-on-ignition method.* J. B. Rather. Bull. 140, Arkansas Expt. Stat. J. Ind. Eng. Chem., 1918, 10, 439–442.

ORGANIC matter in soils may be accurately determined from the loss on ignition of the sample after a preliminary treatment with a mixture of dilute hydrochloric and hydrofluoric acids to remove hydrated mineral constituents, carbonates, etc.:—One grm. of the soil, prepared in accordance with the official (American) method, is digested for 5 mins. with 50 c.c. of water in a platinum basin at 85° C., the aqueous extract decanted through an asbestos filter, with the aid of suction, and the residue digested with a second portion of 50 c.c. of water at 85° C. The united extracts are evaporated to a small volume, whilst the residue is digested with a mixture of 10 c.c. of 2.5% hydrochloric acid, 10 c.c. of 2.5% hydrofluoric acid, and 30 c.c. of water, the extract decanted through the asbestos filter, which is then transferred to the platinum basin containing the residue, and the digestion and decantation of the whole repeated five times. Finally the residue is washed with water, the concentrated aqueous extract added, the liquid evaporated to dryness, and the residue weighed, ignited, and again weighed. There is no appreciable loss of organic matter during the digestions.—C. A. M.

*Phosphate rock; Agricultural availability of raw ground —.* W. H. Waggaman and C. R. Wagner. J. Ind. Eng. Chem., 1918, 10, 442–444.

FIELD experiments under varying conditions over periods up to 5 years showed that the application of medium or large quantities of crude mineral phosphate (tricalcium phosphate) to most soils causes an increase in the yields of many crops during the first year of cultivation. The effectiveness of this form of phosphate as a fertiliser depends upon its being thoroughly distributed in a finely divided state, whilst the presence of decaying organic matter in the soil increases the effectiveness, probably owing to the greater bacterial activity and higher proportion of carbon dioxide under such conditions. For these reasons the effectiveness is usually increased after the rock phosphate has remained for a year or more in the soil. Acid phosphate (superphosphate) acts more rapidly than bone, basic slag, or crude rock phosphate, and is therefore probably the best form of phosphoric acid to use when rapid growth of the crop is the main consideration. To obtain the best results from natural phosphates, it is necessary to use a much greater proportion than is effective in the case of superphosphate, and the question whether an increased yield can be obtained more economically by the use of insoluble than of soluble phosphate depends upon the soil, method of cultivation, and other local conditions.—C. A. M.

*Peat; Inorganic composition of a — and of the plant from which it was formed.* C. F. Miller. J. Agric. Res., 1918, 13, 605–609.

THE peat examined was obtained from Florida from a very extensive deposit, ten feet deep, formed by the remains of saw grass (*Cladium effusum*) growing on the surface. The dried peat contained 7–10% of ash. On the assumption that the silica remained constant during the conversion of the grass into peat, it was calculated that seven parts of grass had yielded one part of peat. The percentage losses of the other constituents of the grass were: 12 of iron and aluminium oxides; 24 of lime; 41 of magnesia; 96 of potash; 84 of soda; 70 of phosphoric acid; and 33 of nitrogen.—J. H. J.

*Dicyanodiamide; Determination of — in old nitrolim (crude calcium cyanamide).* H. Kappen. Z. angew. Chem., 1918, 31, 31–32.

AN old sample of nitrolim examined by the author was free from cyanamide nitrogen. Determinations of the dicyanodiamide by Caro's and von Hager's methods (see this J., 1916, 974) yielded different results, the former giving 72.7% and the latter 82.2%. This difference was apparently due, at least in part, to the presence of urea in the substance. A trustworthy method for the determination of dicyanodiamide in the presence of urea is not, at present, known. (See following abstract.)

—W. P. S.

*Calcium cyanamide with large percentage of dicyanodiamide or urea; Analysis of —.* E. Hien and A. van Haaren. Z. angew. Chem., 1918, 31, 129–131.

WITH normal samples of cyanamide, Caro's method of analysis (this J., 1911, 23) gives satisfactory results, but in the presence of large proportions of dicyanodiamide and urea the results are liable to be erratic unless certain precautions are taken. The authors have obtained satisfactory results by the following procedure: A solution containing cyanamide, dicyanodiamide, and urea equivalent to about 0.2 grm. of nitrogen is diluted to about 200 c.c. and precipitated with 20 c.c. of 10% silver nitrate and 30 c.c. of 10% potassium hydroxide. The precipitate, which contains all the cyanamide

and dicyanodiamide and some of the urea, is washed, the filter is transferred to 150 c.c. of water, and the precipitate dissolved in the minimum quantity of nitric acid. The whole is diluted to 200 c.c.; 100 c.c. is treated with 2 c.c. of 10% silver nitrate with the addition of 15 c.c. of 10% potassium hydroxide. The precipitate, which contains the cyanamide and dicyanodiamide, is treated by the Kjeldahl process for the determination of nitrogen ( $N_1$ ). The nitrogen of the urea may be estimated in the combined filtrates. In another sample the cyanamide nitrogen ( $N_2$ ) is determined by precipitation with silver in a similar manner, using ammonia instead of potassium hydroxide. The urea may be determined more simply by a determination of the total nitrogen ( $N_3$ ). Then  $\text{urea} = N_3 - N_1$ ;  $\text{dicyanodiamide} = N_1 - N_2$ ;  $\text{cyanamide} = N_2$ , all in terms of nitrogen. If the quantity of cyanamide is small compared with that of the dicyanodiamide the precipitation in presence of ammonia may be repeated.—J. F. B.

*Sodium compounds; Action of — on wheat.* J. A. Voelcker. J. Roy. Agric. Soc., 1916, 77, 262–264. Bull. Agric. Intell., 1918, 9, 552.

The sodium compounds used in the pot-cultures were the hydroxide, carbonate, chloride, and sulphate, the hydroxide being applied in solution and the others in the dry state. The different sodium compounds have very different effects both on the germination and the growth of wheat. The hydroxide and carbonate at first retard germination, but eventually have a beneficial effect, even when applied at rates up to  $2\frac{1}{2}$  tons per acre. They cause an increase in yield of grain and in nitrogen content. Sodium chloride has a beneficial effect if used in quantities not exceeding 1 ton per acre, but is harmful to germination and production when used in larger quantities, and at the rate of  $2\frac{1}{2}$  tons per acre may destroy the plant entirely. Sodium sulphate affects neither germination nor production, and may safely be used in quantities up to  $2\frac{1}{2}$  tons per acre. Both the hydroxide and the carbonate cause "caking" and darkening of the soil, but the chloride and sulphate do not.—W. G.

*Magnesium salts; Action of — on wheat.* J. A. Voelcker. J. Roy. Agric. Soc., 1916, 77, 260–262. Bull. Agric. Intell., 1918, 9, 551.

The results of pot-cultures with spring wheat, the magnesium being supplied in the form of sulphate or chloride, indicate that the action of magnesium compounds on wheat varies very greatly with the salt used. The chloride may be used at rates up to 1 ton per acre, but in larger quantities it injures or totally destroys the crop. Magnesium sulphate may be used safely and advantageously up to 5 tons per acre. Increased nitrogen content, such as is obtained in cereals by the use of magnesium oxide, is not produced by magnesium sulphate.—W. G.

*Magnesium carbonate; Injurious action of — on plants.* H. Coupin. Comptes rend., 1918, 166, 1006–1008.

Water culture experiments with a number of different species of plants indicate that, apart from certain exceptional cases, magnesium carbonate is markedly injurious to plants. The injury, which varies slightly in intensity and character with the plant species, is indicated by diminution in the length of the principal root, considerable reduction in the number and size of the rootlets, the brown or blackish colour of the roots and rootlets, reduction of the number of absorbent hairs where these are formed, and by the lesser length of the aerial portion of the plant. In no case had magnesium carbonate any useful effect under the experimental conditions.—W. G.

*Gas liquor; Use of crude — as fertiliser.* Kayser. J. Gasbeleucht., 1918, 61, 121–123.

The growing shortage of supplies of sulphur in Germany necessitates the consideration of methods of making use of gas liquor other than in the manufacture of ammonium sulphate. With regard to direct application of the liquor as a fertiliser, the presence of plant poisons—thiocyanates, tar acids and bases, etc.—precludes its use as a top dressing. If the liquor is applied to the soil in the autumn, so as to allow a long interval before the time of sowing, it is possible that the harmful constituents are destroyed, but then the loss of nitrogen will be excessive. In the absence of definite experimental results caution is advisable.—H. J. H.

*Spent oxide; Use of — as a fertiliser.* J. Gasbeleucht., 1918, 61, 152.

The presence of sulphur and cyanogen compounds, which are plant poisons, seems to stand in the way of the use of spent oxide as a fertiliser. Still, in France it is applied to the land in considerable quantities, especially in connection with vine culture—probably on account of a destructive action on pests. Transport restrictions render its disposal in Germany difficult, and an outlet in agricultural operations is sought, especially in view of its small ammonia content.—H. J. H.

*Phosphoric acid; Determination of citric-soluble — in superphosphates.* P. Wagner. Z. angew. Chem., 1918, 31, 136.

HARTLEY'S method of replacing 2% citric acid by dilute nitric acid for the determination of citric-soluble phosphoric acid in superphosphates (this J., 1918, 384 A) gives, as a rule, considerably lower results. In 31 test experiments the amounts in the majority of cases ranged from 1 to 2% less than those obtained by the older method.—C. A. M.

#### PATENTS.

*Soil-testing apparatus.* M. O. Reiche, Milwaukee, Wis. U.S. Pat. 1,259,669, Mar. 19, 1918. Date of appl., Nov. 3, 1915.

A PORTABLE apparatus which can be used for estimating carbonates and humus in the soil and the acidity of the soil. In the case of alkali soils it can be used for estimating sodium carbonate in the soil.—W. G.

*Fertiliser; Process of making a —.* H. Blumenberg, jun., Los Angeles, Cal. Assignor to F. Blumenberg, New York, and J. J. Seeman, Los Angeles, Cal. U.S. Pat. 1,266,198, May 14, 1918. Date of appl., Jan. 22, 1917.

GROUND felspar, sodium nitrate, and an acid sludge containing sulphuric acid are heated together to liberate the nitric acid, and then further heated to  $800^{\circ}$ – $1000^{\circ}$  C. The fused mass, consisting of aluminium, potassium, and sodium sulphates, is mixed with calcium nitrate and soluble calcium phosphate. The calcium nitrate and soluble calcium phosphate are obtained by treating rock phosphate with the nitric acid collected from the first process.—W. G.

*Fertiliser and process of making the same.* H. Blumenberg, jun., Los Angeles, Assignor to F. Blumenberg, New York. U.S. Pat. 1,266,199, May 14, 1918. Date of appl., July 16, 1917.

The claim is for a fertiliser consisting of a fused mass containing sodium nitrate and tricalcium phosphate.—W. G.



*Production of aluminium compounds [and a fertilizer].* Eng. Pat. 113,276. See VII.

*Preparation of crystals of hygroscopic substances.* Ger. Pat. 304,087. See VII.

## XVII.—SUGARS; STARCHES; GUMS.

[Sugar] juice; Direct filtration of clarified [cane] — using kieselguhr. S. S. Peck. Rept. to Hawaiian Chem. Assoc., Oct., 1917. Intern. Sugar J., 1918, 20, 332—333.

DIRECT filtration of the entire limed juice should not only lessen the duration of the process of clarification, but should at the same time give a better quality of sugar and diminish the losses of manufacture. In a series of experiments carried out in Hawaii, using a Sweetland filter-press with a total filtering area of 860 sq. ft., the leaves of which were dressed with cotton drill, kieselguhr was added at the rate of 5 lb. per ton of cane to the first lot of juice so as to form a preliminary coating on the cloth, after which filtration proceeded continuously and satisfactorily. Allowing half an hour for "dumping" the press, and taking the average rate of filtration under these conditions as 2.98 gallons per sq. ft. per hour, the area necessary for direct juice filtration is 150 sq. ft. per ton of cane per hour, with 20 sq. ft. for re-pressing the cake, giving a total of 170 sq. ft. as compared with 190 sq. ft., the total area necessary when clarified juice and mud are handled separately. Similar trials carried out in several factories in Cuba are reported to have proved successful.—J. P. O.

*Decolorising carbons; Vegetable —, and their use in the cane sugar industry.* F. W. Zerbán. Louisiana Bull., No. 161, Feb., 1918.

CONTINUING the experiments recently described by Schneller (this J., 1918, 316 A), the author has found that highly active decolorising carbons can be prepared with the use of stannous chloride, which has about the same effect in this respect as zinc chloride; if magnesium chloride be used, a carbon is obtained that is 15 times as active as Norit (the decolorising power of which is 10—30 times that of animal charcoal). Investigations carried out at the Louisiana Experiment Station with the object of establishing the factory conditions most suitable for the utilisation of these decolorising preparations, showed that by far the greatest decolorisation results when clarification is effected with lime and phosphoric acid, as, *e.g.*, when the juice is limed to neutrality, boiled, and filtered, treated with phosphoric acid and then with Norit, and again boiled and filtered; as little as 0.5% of the carbon produces a strong decolorisation, and 1% makes the juice practically colourless. Clarification with lime alone, with lime and sulphurous acid, or even with lime and sulphurous and phosphoric acids, gave less satisfactory results, the effect depending also to some extent upon the variety of cane from which the juice had been crushed. In addition to removing colour from the juice, the carbon at the same time increases the purity, improves the viscosity and rate of filtration, and slightly reduces the ash. Moreover, the decolorising effect, while considerable, is also permanent, and from the tests it is concluded that a greater yield of high-grade sugar is obtainable by the use of decolorising carbon than without it, or, in other words, that more molasses can be boiled back without impairing the colour and quality of the final product.—J. P. O.

*Glucosazone reaction.* I. D. Garard and H. C. Sherman. J. Amer. Chem. Soc., 1918, 40, 955—969.

THIS paper contains the results of a study of the glucosazone reaction. The authors find that phenylglucosazone melts at 208.0° C. (corr.) whether recrystallised from 60 per cent. alcohol or from pyridine. The yield of osazone from 0.2 gm. of dextrose obtained by heating together for one and a half hours 2.0 grms. of phenylhydrazine, 2.5 grms. of glacial acetic acid, and 1.367 grms. of sodium acetate in a volume of 20 c.c. was 76.95% and 76.7% of the theoretical in two experiments. With these quantities four 10 c.c. portions of water at room temperature will wash the precipitate sufficiently for analytical purposes. The maximum yield of pure product is obtained by heating for two hours. Longer heating increases the yield but causes decomposition. Within fairly wide limits a variation of the concentration of the sugar used has very little effect on the percentage yield of osazone beyond that due to the constant loss which results from the solubility of the osazone. An increase in the concentration of phenylhydrazine above about 2 grms. in 20 c.c. results in a rapid decrease in the yield of osazone. The acidity of the solution also has a marked influence on the yield, the range in which the reaction takes place being between  $P_H = +4.0$  and 6.0; the maximum is about 4.7 and is best secured with acetic acid and sodium acetate. For the detection of minute amounts of dextrose, for instance 0.005 gm. in a total volume of 10 c.c., the best results are obtained with 0.4 gm. of phenylhydrazine, 0.5 gm. of acetic acid, and 0.5 gm. of sodium acetate. Maltose, dextrin, and lactose retard the formation of the precipitate but starch has very little effect. The reaction continues at least three hours.—W. G.

*Manufacture of spirit from molasses.* Galle. See XVIII.

## PATENTS.

*Sugar cane; Process and apparatus for macerating crushed —.* C. McNeil, Glasgow. Eng. Pat. 116,521, May 14, 1917. (Appl. No. 6824 of 1917.)

THE sugar cane, after crushing, is again subjected to pressure and at the same time sprayed with water or weak juice, applied to it under such a head that it absorbs the full amount of liquid before it can expand to its full bulk and draw in air. This pressure liquid may be applied to the cane at the point in its travel where the maximum compressive pressure applied to it is just released. The operation may be performed by means of a fluted or serrated roller with loaded bearings and a box situated beneath the roller and perforated on the side adjacent to the roller, means being provided to force the liquid into the box and through the perforations on to the crushed cane passing under the roller; alternatively a trough is placed so that the box and part of the roller are so submerged that the crushed cane is soaked before it is subjected to the additional compressive pressure. —J. F. B.

*Adhesive [from starch]; Vegetable glue or —.* R. W. Tunnell, Philadelphia, U.S.A. Eng. Pat. 116,620, Sept. 6, 1917. (Appl. No. 12,500 of 1917.)

SEE U.S. Pat. 1,248,039 of 1917; this J., 1918, 67 A.

## XVIII.—FERMENTATION INDUSTRIES.

*Alcoholic fermentation; Formation of esters of phosphoric acid during* —. A. Lebedev. *Biochem. J.*, 1918, 12, 87—92.

A MIXTURE of esters of phosphoric acid can be separated from a fermenting sugar solution, indicating that hexose-phosphate is not the only ester of phosphoric acid produced during fermentation. (See further *J. Chem. Soc.*, 1918, 1, 361.)—H. W. B.

*Yeast; Nature of the proteolytic enzyme of* —. N. Ivanov. *Biochem. J.*, 1918, 12, 106—119.

THE proteolytic enzyme of yeast consists of a mixture of pepsin and protease. It is possible, by means of heat or the addition of a suitable activating substance, to inhibit the action of one of the component enzymes without affecting the activity of the other. (See further *J. Chem. Soc.*, 1918, 1, 365.)—H. W. B.

*Wines; Composition of the fixed acidity of* —. J. Laborde. *Ann. Chim. Analyt.*, 1918, 23, 127—132.

THE following quantities of non-volatile acids were found in normal red and white wines:—lactic acid, 1.06 to 1.45; succinic acid, 0.61 to 0.75; malic acid, 0.45 to 2.86; tartaric acid, 1.66 to 2.88%. Bad wines contained lactic acid, 2.24 to 2.40; succinic acid, 0.69 to 0.78; malic acid, 0.36 to 0.67%; tartaric acid, traces. Succinic acid is least attacked by bacteria. (See also this *J.*, 1918, 68 A.)—W. P. S.

*Wine; Detection of cider in [white]* —. F. Mach and M. Fischler. *Chem.-Zeit.*, 1918, 42, 326.

THE method proposed by Medinger and Michel (this *J.*, 1918, 286 A) for the detection of cider or perry in grape wines has been tested with various genuine wines from the Baden district, but was found not to be sufficiently sensitive or reliable for practical use. A definite result could not be obtained with less than 40% of cider, and in certain cases the genuine grape wine itself gave the reaction even more strongly than cider.—J. F. B.

*Alcoholic beverages; Alterations in* — after long storage. O. Reinke. *Chem.-Zeit.*, 1918, 42, 315.

A WELL-CORKED bottle originally containing either beer or wine and several centuries old was found in a wine-cellar. The contents were turbid and dark brown and contained yeast cells, cocci, and spores and mycelia of mould fungi. The liquid, which was acid and free from alcohol, contained 0.48% residue, 0.14% ash, 0.049% phosphoric acid, and 0.067% nitrogen. In the case of a partially filled bottle of champagne which had lain open on its side for 5 to 8 years, the contents were dark brown and flavourless, and contained spores, mycelia, etc., of *Aspergillus niger*. The filtrate was strongly alkaline and slowly produced hydrogen peroxide. It contained 0.27% total solids, 0.148% ash, 0.006% nitrogen, but no alcohol or sugar.—C. A. M.

*Molasses; Manufacture of spirit from* —. E. Galle. *Z. angew. Chem.*, 1918, 31, 3—4, 6—7.

IN the case of molasses containing raffinose, fermentation by beer yeast is the only applicable method of obtaining a satisfactory yield of alcohol; under certain conditions, mucilaginous gum-like substances may be formed during fermentation. Too large an amount of salts, or the presence of certain salts is another cause of bad fermentations. In practice it has been found that nitrites and fatty acids have a strong restrictive

influence on the fermentation. In several molasses distilleries ammonium phosphate is added to the yeast cultures to obtain a more vigorous growth. A fermentation test should be applied and the molasses stored and used separately according to the results. A quantity of molasses corresponding with 42 grms. of sugar as indicated by polarisation is diluted with 300 c.c. of water, neutralised with N/1 sulphuric acid, an additional 3 c.c. being added, and then treated with 30 c.c. of yeast culture which has been fermented down to 7 saccharimeter degrees. The flask is connected with Halden's apparatus, which has been charged with sulphuric acid and weighed, and the whole is immersed in water at 24° R. (30° C.), and weighed at intervals of 6 hours. A good sample of molasses should yield 0.5 gm. of carbon dioxide after 6 hours, 1.5 gm. after 12 hours, and at least 4 grms. after 18 hours. In accordance with the results of this test and the alkalinity, commercial molasses may be classified into the following groups:—(1) Alkalinity requiring up to 4 c.c. of N/1 acid per 100 c.c.; carbon dioxide in 18 hours, 4 to 6 grms. (2) Alkalinity, 5 to 8 c.c.; carbon dioxide, 3 to 4 grms. (3) Alkalinity, over 8 c.c.; carbon dioxide, less than 3 grms. The regulations of the Austrian Sugar Exchange prohibit the sale of acid molasses, but products which have undergone acetic or butyric fermentation are sometimes neutralised with soda or lime. Such products ferment badly, and yield volatile fatty acids when distilled with dilute sulphuric acid. For the fermentation of molasses a yeast acclimatised to hydrofluoric acid is the most suitable, but attempts to acclimatise beer yeast to hydrofluoric acid so as to ferment the raffinose have not proved successful. A suitable culture of yeast is prepared as follows:—About 10 grms. of good molasses is neutralised with sulphuric acid, diluted with water to 38° B. (sp. gr. 1.357), boiled, cooled, and after 48 hours boiled again. 170 c.c. of this liquid is treated with 70 c.c. of N/1 phosphoric acid, 10 c.c. of hydrofluoric acid (=2 c.c. of N/1 phosphoric acid), and 25 c.c. of a rosin soap solution prepared by boiling 100 kilos. of rosin with 20 grms. of potassium hydroxide in 500 litres of water, and diluting the solution to 4° B. (sp. gr. 1.029). The liquid is made up to a litre in a fermentation flask, and treated with 60 c.c. of pure yeast culture from a previous cultivation, which has fermented down to 7 saccharimeter degrees, the flask being immersed in water at 15° R. (19° C.), while a slow current of sterilised air is drawn through the liquid. After 24 hours the liquid will have fermented down to 7 saccharimeter degrees, and further cultivations are made from it in the same way, until eventually 500 litres is obtained, two-thirds of which is used for pitching the molasses. By carrying out the fermentation in stages, the yeast cultivation from one tun being introduced into a second tun, and so on, it is possible to ferment good molasses down to 6° in 12 to 14 hours, but it is essential to prevent the temperature rising above 24° to 26° R. (30° to 32.5° C.), or the yeast will degenerate. For the distillation of the wash Barbet's or Guillaume's combined distillation and rectifying apparatus is suitable. The residue from the still is evaporated to 42° B. (sp. gr. 1.41); and in Rueb's process for the recovery of nitrogenous compounds is distilled in an apparatus resembling a coke oven, or it may be consumed in a Parion or Gauer furnace, without recovery of the nitrogen.—C. A. M.

*Action of symbiotic bacteria on the constituents of fats.* Bierry and Portier. See XII.

*Alimentary preparations from blood and meat by means of yeast.* Gauducheau. See XIXa.



*By-products of the fermentation of cabbage.* Nelson and Beck. See XIXA.

#### PATENTS.

*Boiling-pans [brewing coppers] and the like.* A Hill, Burton-on-Trent. Eng. Pat. 116,486, Mar. 23, 1918. (Appl. No. 5136 of 1918.)

A BOILING-PAN, set-pan, or the like is provided with inclined baffle-plates to facilitate circulation of the liquid. A supporting hinge is fixed above the bottom of the pan, at an angle of about 30° to the horizontal, and two plates, attached to it, extend in opposite directions across the pan. The outer ends of the plates are attached by chains to a central vertical chain, which may be raised or lowered to adjust the inclination of the plates to the horizontal and to one another and thus vary the circulation of liquid.—W. F. F.

*Distilling in multiple effect alcoholic liquors and the like; Process and apparatus for —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 107,599, June 30, 1917. (Appl. No. 9465 of 1917.) Under Int. Conv., June 9, 1914.

SEE Fr. Pat. 478,885 of 1914; this J., 1916, 1172.

*Manufacture of plastic masses resembling horn or ebonite [from yeast].* Ger. Pats. 302,931, 302,930, and 303,133. See XV.

*Mcal or flour [from brewers' and distillers' grains] and method of obtaining and preparing same.* Eng. Pat. 116,357. See XIXA.

### XIXA.—FOODS.

*Butter or oleomargarine; Detection of added colour in —.* H. A. Lubs. J. Ind. Eng. Chem., 1918, 10, 436—439.

TWENTY grms. of the fat is dissolved in ether and the mixture is shaken with dilute sodium hydroxide solution; if the latter is coloured yellow, a vegetable colour is indicated. The presence of annatto is detected by filtering the alkaline solution, washing the paper free from alkali, and testing it with acid stannous chloride solution; a red coloration indicates annatto. Another quantity of 20 grms. of the fat is dissolved in 50 c.c. of petroleum spirit and the solution is shaken with 10 c.c. of a mixture of 1 vol. of sulphuric acid and 10 vols. of glacial acetic acid. Azo dyes impart a yellow, brown, red, or blue colour to the acid layer; annatto gives a green to brown colour, and turmeric a violet-red colour, but these two dyes are detected by the alkaline test. One or two pounds of the melted fat is then diluted with an equal volume of petroleum spirit, and the solution is shaken with about one-half its volume of a mixture of sulphuric acid 10, glacial acetic acid 90, and water 10 c.c. If Yellow AB or Yellow OB is present, the acid layer is coloured wine-red. The acid layer is then mixed with dilute hydrochloric acid and shaken with ether, which extracts Sudan I, II, and III, and Carminaph Garnet; Yellow AB and OB remain in the acid solution. The latter is then separated, diluted, neutralised, and extracted with ether; the ethereal layer is drawn off, washed with water, then with alkali solution, and evaporated to dryness. The residue obtained consists of Yellow AB or OB, or a mixture of both. Confirmatory tests may be applied to the residue.

—W. P. S.

*Gluten; Hydration capacity of —.* R. A. Gortner and E. H. Doherty. J. Agric. Res., 1918, 13, 389—418.

WEIGHED discs of gluten from different flours were immersed in solutions of lactic, acetic, boric, phosphoric, hydrochloric, and oxalic acids of various concentrations, with and without the addition of certain salts in 0.005 molar concentration. It was found that, notwithstanding the difference in their physical properties, the moist glutes from these flours absorbed almost the same amount of water. The results also showed that gluten from a "weak" flour has a much lower rate of hydration than that from a "strong" flour, and changes from a gel to a sol at a much lower degree of hydration. The hydration curves observed were of two types. Dilute solutions of hydrochloric and oxalic acids cause rapid absorption of water by the gluten, whereas somewhat stronger solutions of these acids extract water from moist glutes. On the other hand, in the case of lactic, acetic, and phosphoric acid, the use of stronger solutions causes only a slight reduction in the absorption of water by gluten. The relative hydration capacity of gluten immersed in acid solutions is reduced by the presence of inorganic salts, the glutes from different flours being differently affected, but the difference in baking properties between the glutes from "strong" and "weak" flours is not attributable to the acid and salt contents of the flours. The physico-chemical properties of the glutes from the different types of flour would not be identical, even if the flours had originally contained the same proportion of acid and salts. Neither can the different physical conditions of gluten be attributed solely to the presence or absence of an electric double layer surrounding the colloidal particles. It is suggested that the differences between "strong" and "weak" glutes may be due to the size of the particles, and that in the case of "weak" glutes a portion of the particles may approach more closely to the boundary line between the colloidal and crystalloidal conditions. A "strong" gluten has the emulsoid properties of a nearly perfect colloidal gel to a much greater extent than a weak gluten. (Compare T. B. Wood, this J., 1907, 1290; 1908, 175.)—C. A. M.

*Alimentary preparations from blood and meat by means of yeast.* A. Ganduchau. Comptes rend., 1918, 166, 1058—1059.

THE blood of pigs, bullocks, or horses is heated to coagulate the albumins and to disinfect it, then powdered, and fermented in slightly acid medium, in the presence of a small quantity of sugar, with brewer's yeast. A paste is obtained, which is porous and has a delicate aroma and may suitably be made into pasties or biscuits. Such a biscuit of fermented blood and flour constitutes a complete food.—W. G.

*Canned foods; Influence of cold shock in the sterilisation of —.* L. D. Bushnell. J. Ind. Eng. Chem., 1918, 10, 432—436.

RAPID cooling (blanching) immediately after heating is of no value in reducing the time required properly to sterilise tinned foods. The growth of bacteria is but little affected by the addition of small amounts of common salt, but a small quantity of acetic acid has a distinctly retarding action on the growth of bacteria in tinned vegetables. In many cases an unsterile product will keep indefinitely if sealed properly; sealing should not, however, be expected to take the place of a proper sterilising process.—W. P. S.

*Proteins; Comparative study of the toxicity and nutritive power of alimentary — used in the pure state.* F. Maignon. *Comptes rend.*, 1918, **166**, 1008—1010.

Feeding experiments with white rats indicate that fibrin, casein, and washed extracted meat powder, like egg-albumin, are incapable by themselves of supporting life, or of assuring no loss in weight even over a short period. Casein is the least toxic, then comes fibrin, and lastly egg-albumin, the meat powder being comparable with fibrin. In the case of casein and fibrin there is no seasonal effect as there is with egg-albumin. Further, when the two former proteins are fed, there is a deposition of fat in the liver, more marked with casein than with fibrin, whilst there is no fat deposition when egg-albumin or meat powder is fed. There is, therefore, an apparent correlation between the readiness of conversion into fat and the length of time over which the protein will support life.—W. G.

*Antineuritic and antiscorbutic factors; Differential behaviour of the — towards adsorbents.* A. Harden and S. S. Zilva. *Biochem. J.*, 1918, **12**, 93—105.

The antineuritic vitamin can be quantitatively removed from a solution containing antineuritic and antiscorbutic substances, e.g., a mixture of autolysed yeast and orange juice, by adsorption with fullers' earth, without altering the antiscorbutic power. Attempts to recover subsequently the adsorbed antineuritic vitamin in an active form from the fullers' earth have not yet been successful.

—H. W. B.

*Cabbage; By-products of the fermentation of —.* V. E. Nelson and A. J. Beck. *J. Amer. Chem. Soc.*, 1918, **40**, 1001—1005.

In ordinary commercial fermented cabbage (sauerkraut) the volatile acidity represents from 25 to 50% of the total acidity. The volatile acidity (equivalent to from about 86 to 122 c.c. N/1 H<sub>2</sub>SO<sub>4</sub> per 200 grms. of sample) is due to acetic and propionic acids, the former preponderating, though in two cases formic acid was also found, and the fixed acidity to an inactive form of lactic acid. Alcohols, consisting entirely of ethyl and propyl alcohols, are present to about the same extent as volatile acids. Esters were only present in small amount, and 2.0—2.5% of mannitol was found.

—W. G.

*Lupine seeds as a coffee substitute.* M. Gonnermann. *Chem.-Zeit.*, 1917, **42**, 296.

LUPINE seeds which have been freed from bitter substances do not contain any alkaloid or saponin; the roasted seeds form a coffee substitute, and the author has used the latter for some months without suffering any ill effect.—W. P. S.

*Action of symbiotic bacteria on the constituents of fats.* Bierry and Portier. *See XII.*

*Determination of water [in cheese, etc.] by distillation.* Besson. *See XXIII.*

#### PATENTS.

*Soya beans; Process of, and apparatus for treating —.* T. W. Rees, and The Eastern Flour Co., Ltd., Staines. *Eng. Pat.* 116,158, June 8, 1917. (*Appl.* No. 8179 of 1917.)

Soya beans are cleaned by spraying them with an alkali solution, and are then roasted, cooled, crushed, and freed from the husks by sifting or otherwise. The separated kernels are pressed to

remove all but 5 to 6% of the oil, and the press-cake ground to a flour, which may be mixed with wheat flour in suitable proportions. Automatic apparatus for carrying out this process includes a cleaning and brushing machine, a roasting chamber in which the beans are made to pass through a flame, a storage hopper and spraying device between the cleaning machine and the roaster, and means for cooling and crushing the beans, for separating the husks, and conveying the kernels to a press.

—C. A. M.

*Meal or flour [from brewers' and distillers' grains], and method of obtaining and preparing same.* J. Sleeman, Stonehouse. *Eng. Pat.* 116,357, June 8, 1917. (*Appl.* No. 8218 of 1917.)

BREWERS' or distillers' grains are dried in a malt-drying kiln by means of a forced draught, being turned while drying, and are then winnowed or otherwise treated to separate the husks, which will be less broken up than when the grains are dried in a drying machine. The grain is then ground and the meal dressed to separate the residual husks, and, if desired, separated into two grades, one of which may be mixed with ordinary flour for human food, whilst the coarser grade may be made into dog biscuits.—C. A. M.

*Milk-like food for the feeding of agricultural stock.* W. J. Melhuish, Poole. *U.S. Pat.* 1,265,227, May 7, 1918. *Date of appl.*, Apr. 23, 1917.

*See Eng. Pat.* 101,264 of 1916; this J., 1916, 1078.

*Drying apparatus for use in drying fruit, herbs, vegetables, seeds, chemicals, fibre, and the like.* *Eng. Pat.* 116,606. *See I.*

#### XIXB.—WATER PURIFICATION; SANITATION.

*Water; Softening of — by the lime-sodium carbonate and lime-sodium hydroxide processes.* H. Noll. *Z. angew. Chem.*, 1918, **31**, 5—6, 9—11.

THE amounts of reagents required for softening water by the lime-sodium carbonate and lime-sodium hydroxide methods may be calculated from the total hardness and the carbonate-hardness, non-carbonate-hardness, and magnesia-hardness respectively. In determining the total hardness by Blacher's method with potassium palmitate solution (this J., 1912, 555), zinc and iron do not interfere with the process, as stated by Zink and Hollandt (this J., 1914, 803), provided they are first precipitated by means of sodium hydroxide. The carbonate-hardness is determined by titrating the water with standard acid, with methyl orange as indicator, and the magnesian hardness by the following modification of the method of Froboese (this J., 1915, 99):—100 c.c. of the water is rendered neutral to methyl orange with hydrochloric acid, then boiled for 10 mins., treated with 5 c.c. of a saturated solution of sodium oxalate, and again boiled for 1 to 2 mins. After the precipitate has subsided, the liquid is titrated with N/10 potassium palmitate solution, with phenolphthalein as indicator. The number of c.c. required multiplied by 2.8 gives the magnesian hardness in German degrees (one German degree=40 mgrms. CaO per 100 c.c.). The carbonate-hardness multiplied by 10 gives the amount of lime in mgrms. per litre of water required to convert bicarbonates into carbonates, whilst the magnesian hardness multiplied by 10 gives the amount of lime (mgrms. per litre) required to convert the magnesium carbonate into hydroxide and magnesium chloride and sulphate into the corresponding calcium salts. The non-carbonate-hardness multiplied by 19 gives the amount of



sodium carbonate required (mgrms. per litre), but in practice it is necessary to add an additional 10 to 20%. As a rule water rich in salts requires a smaller excess than a water containing a small amount of salts. The same data are used for calculating the amounts of reagents required for the lime-sodium hydroxide process. In the case of a water in which the carbonate-hardness, excluding magnesian hardness, is greater than the non-carbonate-hardness, the latter multiplied by 1.43 gives the mgrms. of sodium hydroxide required per litre, whilst the sum of the carbonate and magnesian hardness, less the non-carbonate-hardness, multiplied by 10 gives the amount of lime required. To give the necessary excess of sodium carbonate the amount of sodium hydroxide should be increased and that of the lime reduced by quantities corresponding with 1° of hardness, and for this purpose sodium carbonate must be added to waters in which the carbonate-hardness does not exceed the non-carbonate-hardness by at least 1°. Where the combined hardness due to carbonates and magnesium salts is greater than the non-carbonate-hardness, whilst the carbonate-hardness is equal to or less than the non-carbonate-hardness, the amount of sodium hydroxide required is obtained by multiplying the non-carbonate-hardness by 1.43, and the lime by subtracting the non-carbonate-hardness from the sum of the carbonate and magnesian hardness and multiplying by 10. In the presence of magnesium bicarbonate in waters of this type, sodium carbonate should be added in a proportion corresponding with 1° (=19 mgrms. per litre). In the case of waters in which the combined carbonate and magnesian hardness is equal to the non-carbonate-hardness, the latter multiplied by 1.43 gives the required amount of sodium hydroxide. In this case no lime is required, and an excess of sodium carbonate must be added to the water. In the case of waters in which the combined carbonate and magnesian hardness is less than the non-carbonate-hardness, the sum of the carbonate and magnesian hardness multiplied by 1.43 gives the sodium hydroxide; the carbonate plus the magnesian hardness, less the non-carbonate-hardness, multiplied by 10, gives the amount of lime required; and the non-carbonate less the carbonate hardness, plus the magnesian hardness, multiplied by 19 gives the required amount of sodium carbonate. For softening alkaline water lime only is required.—C. A. M.

*Nitrates in water; Detection of* —. Escaïch. J. Pharm. Chim., 1917, 47, 395.

THE nitrate is reduced to nitrite by means of amalgamated aluminium, the reduction requiring about 1 hour for completion. The nitrite is then detected by treating 15 c.c. of the water with 2 c.c. of 10% aqueous antipyrine solution, 4 drops of acid mercuric sulphate solution, and 1 drop of 5% potassium ferrieyanide solution; a red coloration develops with as little as 0.1 mgrm. of nitrite per litre of water.—W. P. S.

*Insecticides; Physical properties governing the efficacy of contact* —. W. Moore and S. A. Graham. J. Agric. Res., 1918, 13, 523–538.

CONTACT insecticides may be divided into two groups: (1) those which “wet” the insect, owing to adhesion between the insecticide and the chitin which forms the outer covering of the insect, “spread” over the surface of the body, owing to the cohesion of the liquid being less than the adhesion between the liquid and the chitin, and pass into the trachea by capillarity; (2) those which “wet” the insect, but do not “spread” over the body owing to the cohesion of the liquid being greater than the adhesion between liquid and insect,

nor pass into the trachea by capillarity. Cockroaches and other insects were immersed in the liquid, previously coloured with Sudan III. or with trypan-blue, for periods of  $\frac{1}{2}$ –2 hours, depending on the viscosity of the liquid. The insect was then removed and cut open, and the tracheæ which had been penetrated were observed to be coloured either red or blue. Among the substances which penetrated farthest were fat solvents and the substances soluble in them, whilst aqueous solutions, except soap solutions, did not penetrate at all. The rate of penetration was governed by the viscosity. In the case of emulsions, spreading depended upon the character of the emulsifier and not on the emulsified substance. It was observed that fumigants gained entrance to the insect by way of the trachea, and that volatile oils could enter either as liquid or vapour.—J. H. J.

#### PATENTS.

*Sewage sludge; Air-lifts for [activated]* —. T. Caink, Worcester. Eng. Pat. 116,580, July 7, 1917. (Appl. No. 837 of 1917.)

A VERTICAL pipe is arranged with its lower opening near the floor of the settling chamber, and its upper end is surrounded by an annular chamber having a weir at one side over which the sewage sludge flows to another chamber and thence to the aeration tank. Air is supplied through a small concentric vertical pipe having its lower end terminating in a porous diffuser, through which the air rises into the lift pipe, carrying the sludge with it. A gauge-tube is provided on the annular chamber, so that the height of liquid over the weir and consequently the rate of flow is indicated. The proportion of sludge is indicated by the amount of solid matter settling in the gauge-tube.—W. F. F.

*Sewage; Purification and decolorisation of industrial* —. C. Gunkel, Halle. Ger. Pat. 304,040, May 1, 1915.

THE sewage is first treated with chlorides or sulphates of alkaline-earths, e.g., with the waste liquors of potash works, and then with a mixture of highly plastic clay and the weathered detritus of silicate rocks as described in Ger. Pat. 248,980 (this J., 1912, 837). The process is specially suited to the sewage of textile and dye works.—H. J. H.

*Carbon; Preparation of active — from sewage sludge*. J. v. Kruszewski, Berlin-Pankow. Ger. Pat. 305,895, Oct. 21, 1913.

SEWAGE sludge is dried, mixed with a suitable alkali (e.g., lime), and the mixture subjected to dry distillation.—L. A. C.

*Calcium arsenate for insecticidal use; Process for manufacturing* —. J. K. Haywood, Washington, D.C. U.S. Pat. 1,266,258, May 14, 1918. Date of appl., Mar. 30, 1918. (Dedicated to the public.)

DICALCIUM orthoarsenate is prepared by the action of finely powdered limestone on arsenic acid. It may be used alone for certain insecticidal purposes, or it may be subsequently treated with a sufficient quantity of lime to produce tricalcium orthoarsenate, when it is suitable for use as an insecticide on foliage.—W. G.

*Process for rendering smelter smoke innocuous*. Ger. Pat. 302,996. See X.

*Apparatus for and method of recovering fats*. Eng. Pat. 116,384. See XII.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Antitoxin; Destruction of tetanus — by chemical agents.* W. N. Berg and R. A. Kelsner. *J. Agric. Res.*, 1918, 13, 471—495.

EXPERIMENTS were undertaken to determine if possible whether tetanus antitoxin was of a protein or a non-protein nature. The antitoxin was submitted to the action of digestion preparations of trypsin and pepsin for periods up to 119 days. The progress of the digestion was controlled by determinations of the coagulable protein and amino-nitrogen. Any action on the antitoxin was detected by injecting the preparation together with tetanus toxin into a guinea-pig. It was found that the antitoxin was slowly destroyed by 0.5% sodium carbonate solution alone, without the protein being affected. Trypsin in neutral solution and in sodium carbonate solution digested both antitoxin and protein at the same rate. In 0.2% hydrochloric acid solution, antitoxin was destroyed in a few days, without any destruction of protein. Pepsin alone had no effect on the antitoxin, but pepsin-hydrochloric acid digested protein and antitoxin simultaneously. It is concluded that the antitoxin was of a non-protein nature, but that its stability was so closely related to that of the associated protein, that when the latter was broken down the antitoxin broke down with it.—J. H. J.

*Broom; Presence of a fixed alkaloid in the common —.* A. Valeur. *Comptes rend.*, 1918, 167, 26—28.

FROM the mother liquors obtained in the preparation of sparteine sulphate from *Sarothamnus scoparius*, the author has obtained a small quantity of a new alkaloid, *sarothamnine*, which readily forms crystalline compounds with various solvents, of which *sarothamnine-chloroform*, m.pt. 127° C.,  $[\alpha]_D = -38.7^\circ$ , and *sarothamnine-alcohol*, m.pt. 90° C.,  $[\alpha]_D = -25.6^\circ$  are described. The alkaloid is an unsaturated compound, and to it the formula  $C_{15}H_{24}N_2$  is provisionally assigned.—W. G.

*Cotton plant; Chemistry of the — with special reference to Upland cotton.* A. Vichover, L. Chernoff, and C. O. Johns. *J. Agric. Res.*, 1918, 13, 345—352.

UPLAND cotton, *Gossypium hirsutum*, contains quercimeritrin and isoquercitrin, which have been previously isolated from other types of cotton plant, but does not contain gossypitrin and gossypetin. The ethereal oil isolated from Upland cotton differs from that found in the root bark of *G. herbaceum*. The greater part distils between 200° and 300° C., the lower fractions of the distillate being yellow to greenish-yellow, and the higher fractions light bluish-green to dark blue. The oil attracts the boll weevil.—C. A. M.

*Phosphotungstates of certain bases and amino-acids.* J. C. Drummond. *Biochem. J.*, 1918, 12, 5—24.

THE phosphotungstates of a large number of bases and amino-acids have been prepared in a pure condition and the solubilities in various solvents studied. The results indicate that the separation of the components of a complex mixture of bases and amino-acids is not likely to be readily effected by means of a process depending upon the relative solubilities of their phosphotungstates in various solvents. (See further *J. Chem. Soc.*, 1918, i., 336.)

—H. W. B.

*Valerian root; Analysis of —.* Ruyden. *Svensk Farm. Tidskr.*, 1917, 481. *J. Pharm. Chim.*, 1918, 17, 396—397.

TEN grms. of the powdered root is boiled for 1 hour with 100 c.c. of N/2 alcoholic potassium hydroxide solution, then cooled, alcohol is added, if necessary, to make up the original weight, and the solution is filtered; 25 c.c. of the filtrate is distilled with the addition of 20 c.c. of 10% phosphoric acid and 75 c.c. of water, and 110 c.c. of distillate is collected. The distillation residue is then mixed with 25 c.c. of the alkaline alcoholic extract and 95 c.c. of water and the distillation repeated. The distillates are titrated with N/10 barium hydroxide solution; 1 c.c. of the latter is equivalent to 0.0102 gm. of valeric acid. Valerian root yields from 2.91 to 4.77% of volatile acids (calculated as valeric acid), 13.1 to 26.9% of extractives, and 12.7 to 47.2% of ash. The high ash content of some samples is due to insufficient cleansing of the roots.—W. P. S.

*Carbamides; Constitution of —. VI. Mechanism of the synthesis of urea from urethane.* E. A. Werner. *Chem. Soc. Trans.*, 1918, 113, 622—627.

URETHANE undergoes gradual dissociation into ethyl alcohol and cyanic acid in the neighbourhood of its boiling point, and the hydrolysis of urethane by aqueous alkali is not a direct process but involves the intermediate formation of sodium cyanate, which undergoes subsequent hydrolysis to sodium carbonate. Similarly the formation of urea when urethane or ethyl carbonate is heated in solution with ammonia, is dependent on the intermediate formation of cyanic acid, which by combining with the ammonia yields urea; indeed, the ammonia resulting from the partial hydrolysis of the cyanic acid is sufficient for the reaction, and there is no need to add ammonia independently in order to obtain urea. The suggestion is made that two amino-groups, like two hydroxyl groups, cannot be stably attached to one carbon atom and that the  $:C(NH_2)_2$  grouping is probably absent from urea, thiourea, and guanidine. —D. F. T.

*Guaiacol carbonate; Identification of —. A test for ether.* G. Maue. *Pharm. Zeit.*, 1918, 63, 255—256.

ABOUT 0.02 gm. of the sample is dissolved in 1 c.c. of alcohol, 2 drops of ferric chloride solution and 1 drop of formaldehyde solution are added, and the mixture is poured on the surface of 2 c.c. of sulphuric acid; a cherry-red coloration, due to the presence of guaiacol, develops at the junction of the two liquids. Ordinary ether usually contains aldehydes and the latter invariably include formaldehyde, which is essential to the above reaction; consequently, when ordinary ether is substituted for the alcohol in the above test a positive reaction is obtained. Pure ether, such as is used for anaesthetic purposes and which must be free from aldehydes, does not give a coloration with the test. The sensitiveness of the reaction is about 1:300,000. —W. P. S.

*Glycerol; Decomposition of — in the presence of different catalysts: formation of ethyl and allyl alcohols.* P. Sabatier and G. Gaudion. *Comptes rend.*, 1918, 166, 1033—1039.

GLYCEROL is dehydrated when passed over alumina at 360° C., the gaseous products being carbon monoxide, methane, and a little carbon dioxide, and the liquid products acrolein and higher aldehydes resulting from its crotonisation. Passed over finely divided copper at 230° C. the glycerol vapour is decomposed giving hydrogen and carbon dioxide mixed with some methane and carbon monoxide, together with ethyl and allyl alcohols and some of



the higher alcohols. Uranous oxide at 350° C. yields, from the glycerol, more ethyl alcohol and acrolein and less allyl alcohol, and the proportion of carbon dioxide in the gaseous products is increased. By hydrogenation with reduced nickel at 300° C. glycerol yields water and the following gases: hydrogen, methane, ethane, carbon monoxide, and carbon dioxide.—W. G.

*Balsam of tolu; Evaluation of* —. T. T. Cocking and J. D. Kettle. *Pharm. J.*, 1918, 101, 40—41.

THE most satisfactory method for the analysis of tolu balsam is to separate the aromatic acids by boiling with magnesium oxide and water, in the presence of a little xylene to soften the resinous matter. The magnesium salts of the aromatic acids are readily soluble in cold water, those of the resin acids being insoluble. For the determination of the free balsamic acids, 5 grms. of the balsam is dissolved in 25 c.c. of hot alcohol; 5 grms. of light magnesium oxide and 20 c.c. of xylene are added, and the contents of the flask well mixed; 100 c.c. of water is added, and the mixture boiled under a reflux condenser for one hour. After cooling, the whole is poured on a Buchner filter, the aqueous portion of the filtrate is separated, and the xylene, together with the magnesia and the filter paper, is returned to the flask. Boiling with 100 c.c. of water each time is repeated twice more; the mixed aqueous filtrate is washed once with ether, then acidified with hydrochloric acid, and the aromatic acids are extracted with ether and dried over sulphuric acid *in vacuo*. For the total balsamic acids, 2.5 grms. of the balsam is saponified by boiling with alcoholic potash, the greater part of the alcohol is evaporated off, the residue is dissolved in 100 c.c. of hot water and acidified with hydrochloric acid; 5 grms. of magnesium oxide and 20 c.c. of xylene are added, and the mixture is treated as before. The proportion of cinnamic acid in the balsamic acids is determined by the gain in weight on bromination. For the determination of the acid value of tolu balsam, 5 grms. of the sample is dissolved in about 50 c.c. of boiling alcohol, a large quantity of phenolphthalein solution (3—4 c.c.) is added, and the hot solution is titrated with N/1 alcoholic potassium hydroxide until the colour becomes dark brown but not red; it is then boiled under a reflux condenser for a few minutes to break up the precipitate, and the titration is completed. The variations found with a large number of samples indicated a range of 92.2—132.4 for acid value, 59.3—90.9 for ester value, 154.8—208.7 for saponification value, and 32.66—47.56 for total balsamic acids.—J. F. B.

*Nitriles; Direct transformation of secondary and tertiary amines into* —. A. Mailhe. *Comptes rend.*, 1918, 166, 996—997.

THE method used to transform di- and tri-isoamylamine into isoamylonitrile by passing their vapours over finely-divided nickel at 350°—380° C. (this *J.*, 1917, 1193) is shown to be generally applicable to secondary and tertiary aliphatic amines.—W. G.

*Pennyroyal; Estimation of pulegone and other ketones in oil of* —. C. T. Bennett. *Perf. and Essent. Oil Rec.*, 1918, 9, 208.

THE neutral sulphite process is the only practical method at present known for the estimation of the ketones in oil of pennyroyal, and the results include both the pulegone and the menthone. Experiments have shown, however, that the absorption in the case of pennyroyal is much slower than in the case of other oils, such as caraway and dill oils, and instead of one hour's shaking, at least three hours' must be given to obtain good results. For

instance, an oil showed 44% ketones after shaking continuously for one hour, 58% after two hours, and 82% after three hours. Another oil gave 50% after one hour and 88% after three hours. Parry's statement that oil of pennyroyal contains at least 80% of pulegone and other ketones is confirmed.

—J. F. B.

*p-Cymene. I. Nitration. Mononitrocymene.* C. E. Andrews. *J. Ind. Eng. Chem.*, 1918, 10, 453—456.

A YIELD of 85% of mononitrocymene was obtained when cymene was nitrated by mixing it with an equal volume of sulphuric acid, cooling to 0° C., and adding a mixture of nitric and sulphuric acids, the temperature being maintained at 0° C. Small quantities of *p*-toluic acid and *p*-tolylmethylketone were also formed during the nitration. On reduction, the mononitrocymene yielded the corresponding amine which, like the nitro compound, consisted of the 1.2.4-isomeride, i.e., 1-methyl-2-amino-4-isopropylbenzene.—W. P. S.

*Electrolytic preparation of p-aminophenol.* Shōji. See III.

*Chemistry and histology of the glands of the cotton plant.* Stanford and Viehoever. See V.

*Determination of halogens, sulphur, and nitrogen in the presence of mercury.* François. See VII.

*Determination of nitric acid in bismuth subnitrate.* Luce. See VII.

*Differential behaviour of the antineuritic and anti-scorbutic substances towards adsorbents.* Harden and Zilva. See XIXa.

*Halogen derivatives of aromatic amines and their analysis.* Dains and others. See XXIII.

#### PATENTS.

*Benzoic acid; Process for the manufacture of* —. Weston Chemical Co., Ltd., London, and J. Savage, Runcorn, Chester. Eng. Pat. 116,348, June 7, 1917. (Appl. No. S152 of 1917.)

TOLUENE (*c.g.*, 92 parts by weight) is chlorinated at boiling temperature until the liquid is of sp. gr. 1.11, the bulk of the product being benzyl chloride, with traces of benzal chloride and a free toluene. The crude product is boiled under a reflux condenser, for about 2 hours, with a quantity of caustic soda or milk of lime equivalent to the chlorine used (*c.g.*, 400 parts of 20% caustic soda). A solution of sodium or calcium hypochlorite (*c.g.*, 2030 parts of calcium hypochlorite containing 7% of available chlorine) is then added in the course of about 3 hours, and stirring is continued for another 3 hours, when the mixture is distilled until free from toluene. The residual alkali benzoate solution is filtered and benzoic acid precipitated by addition of hydrochloric acid.—L. A. C.

*Diaryl sulphites; Production of* —. Badische Anilin und Soda Fabrik. Ger. Pat. 303,033, Aug. 12, 1916.

AROMATIC compounds with hydroxyl groups in the ring—hydroxy-carboxylic acids excepted—are treated with thionyl chloride in presence of bases, *c.g.*, pyridine, which combine with hydrogen chloride without reacting with the thionyl chloride. The diaryl sulphites thus obtained—excepting diphenyl and ditolyl sulphites—decompose readily with the evolution of sulphur dioxide and are very reactive; hence their value as synthetic reagents, *c.g.*, for preparation of aromatic esters.—H. J. H.

*p*-Cymenesulphonic acid; Preparation of — from 2-bromo-*p*-cymene-3-sulphonic acid. Rheinische Campher-Fabrik G.m.b.H., Düsseldorf. Ger. Pat. 303,095, Aug. 18, 1916.

2-BROMO-*p*-CYMENESULPHONIC acid is treated with an aqueous alkali or alkaline-earth and zinc dust. This method is cheaper than the previous process, in which ammonium is used, and the necessity for working under pressure is avoided.—H. J. H.

Guaiacol; Process for the manufacture of —. E. H. Zollinger and H. Röhling, Berlin. Ger. Pat. 305,281, Jan. 23, 1916.

FREE catechol and alkali or alkaline-earth salts of methylsulphuric acid are heated in presence of veratrol as a diluent at 160°–180° C., with the gradual addition of a weak base, such as sodium carbonate or bicarbonate; a yield of over 85% of guaiacol is said to be obtained.—J. F. B.

Hydrogenised compounds; Manufacture of —. Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 305,347, Oct. 5, 1915.

ISOCYCLIC or heterocyclic aromatic bases are hydrogenised by treatment with alkali metals or alkaline-earth metals and alcohols in presence of an indifferent solvent.—J. F. B.

Production of crystals of hygroscopic substances. Ger. Pat. 304,087. See VII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photomechanical printing plates; A development process without glycerin for the preparation of —. Schulz. Chem.-Zeit., 1918, 42, 277–278.

A CONSIDERABLE reduction of the cost of the developer for photomechanical printing plates can be obtained by the use of certain alcohols or, better still, of a saturated solution of a salt, such as magnesium chloride, instead of the glycerin normally used for reducing the effect of the developer on the exposed parts of the image; and by the use of a dilute mineral acid, preferably hydrochloric acid, instead of an organic acid, such as acetic acid. The developer containing mineral acid has also the advantage that the unexposed part of the colloid-chromate film is completely removed, leaving the metal, zinc or aluminium, quite bare, whereas the organic acid developer leaves a very fine film, apparently of metallic chromate, on the surface of the metal, which interferes with the subsequent inking processes.—B. V. S.

Testing of metals by means of X-rays. Janus and Reppchen. See X.

### PATENTS.

Photographic paper. A. C. McCloskey, Philadelphia, Pa., Assignor of part to A. E. Davis and F. J. Geiger. U.S. Pat. 1,265,464, May 7, 1918. Date of appl., May 25, 1916.

PAPER is provided on both sides with a "ferro-undercoating" and with further coatings containing ferric salts on one side and ferrous salts on the other; or paper may be coated on one side only with either of these combinations.—B. V. S.

Printing surfaces; Method of producing —. O. Foerster, New York, Assignor of part to A. Jaeger. U.S. Pat. 1,265,641, May 7, 1918. Date of appl., Mar. 26, 1917.

A PLATE of glass or like substance is coated with a bichromated gelatin film containing a grain-producing substance, exposed to light, and the soluble parts removed. The hollows in the resulting film are filled in with a non-actinic powder by a dusting process. A preliminary coating of bichromated albumin may be given, which is exposed from the back and the soluble parts removed before applying the bichromated gelatin.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

High explosives; Method of calculating comparative strength and efficiency of — from their composition and apparent densities. C. E. Waller. J. Ind. Eng. Chem., 1918, 10, 448–453.

THE "strength" and "efficiency" of high explosives are defined as proportional to the volume which the gaseous products of explosion of a unit weight (1 gm.) and a unit volume (1 c.c.) respectively of the explosives to be compared would occupy at the temperature of explosion,  $t^\circ$ , and at atmospheric pressure. The temperature of explosion,  $t^\circ$ , is calculated from the heats of formation of the ingredients of the explosive and of the products of explosion and the specific heats of the latter, and the volume of the gaseous products of explosion at  $t^\circ$  is then calculated per gm. and per c.c. of explosive respectively. In analysing the products of an explosion, it is necessary to take into account the presence of substances, such as carbonates and bicarbonates of alkali metals, methane, etc., which are due to secondary reactions taking place on cooling. Only such reactions as have taken place up to the moment that  $t^\circ$  has been reached should enter into the calculations of the strength and efficiency of explosives. When sulphur is present in explosives containing a deficiency of oxygen, part combines to form a sulphide ( $H_2S$ ,  $Na_2S$ , etc.), whilst the remainder is oxidised and combines with the metal to form  $Na_2SO_4$ , etc. The following table gives the calculated temperature of explosion,  $t^\circ$ , and gas volume at  $t^\circ$  per gm. of some of the more common explosives:—

Explosive	$t^\circ$	Gas volume at $t^\circ$ per gm.
	$^\circ C.$	litres.
Nitroglycerin .. .. .	3158	8.8328
Trinitrotoluene .. .. .	2217	6.764
Picric acid .. .. .	2599	8.727
Tetranitromethylaniline .. .. .	3126	9.81
Tetranitroaniline .. .. .	3238	10.55
Nitrostarch, 12.75% N .. .. .	2205.7	8.168
Nitrostarch, 13% N .. .. .	2277	8.28
Nitrostarch, 13.47% N .. .. .	2415.4	8.4937
Mixture of—		
21.27% TNT and 78.73% ammonium nitrate .. .. .	2310	8.2622
34% TNT .. .. .	2195	8.3493
66% ammonium nitrate .. .. .		
44.77% TNT .. .. .	2122	8.3315
55.23% ammonium nitrate .. .. .		
58.6% TNT .. .. .	2146	7.974
41.4% ammonium nitrate .. .. .		
92% nitroglycerin .. .. .	3208	9.0424
8% nitrocellulose (12% N) .. .. .		

An approximate idea of the expansion in c.c. resulting from the explosion of 10 grms. of an explosive may be obtained by multiplying the calculated gas volume at  $t^\circ$  by 37.58. The results agree fairly closely with those found by the Trauzl test.—C. A. M.



## PATENTS.

*Blasting-powder.* W. Treese, Silverton, Oreg. U.S. Pat. 1,265,975, May 14, 1918. Date of appl., Apr. 16, 1917.

A MIXTURE for use as a blasting explosive has the approximate composition:—yellow wax 13, soap stock 7, sulphur 21, nitre 11, potassium chlorate 22, and nitro-starch 26%.—T. St.

*Process for rendering iron resistant to the action of organic acids, especially picric acid.* Ger. Pat. 303,126. See X.

## XXIII.—ANALYSIS.

*Water; Determination of — by distillation.* A. A. Besson. Collegium, 1918, 150—152.

THE substance to be examined (5—15 grms.) is heated with 200 c.c. of petroleum spirit, b.pt. below 170° C., in a wide-mouthed flask fitted with a Soxhlet-like apparatus having a side tube, but instead of the syphon the lower end of the body of the apparatus is constricted to a narrow tube graduated in tenths of a c.c. and sealed off at the lower end. The water and spirit distil off and condense into the wide body of the apparatus, and the water collects in the lower graduated part, where the quantity can be read off directly. The results agree with those obtained by direct distillation or drying *in vacuo*. The author gives data obtained with samples of cheese and soap.—D. W.

*Cations of the so-called third and fourth groups; Petersen's method for the qualitative separation of the —.* I. Bolin and G. Starck. Z. anorg. Chem., 1918, 103, 69—72.

THE Petersen process of qualitative analysis (Z. anorg. Chem., 1910, 67, 253) is found to be very satisfactory with the following modifications. After precipitation of the metals of the second group with hydrogen sulphide, sodium sulphate is used instead of sulphuric acid for precipitating barium and strontium. The precipitate is washed with hot water and the washings tested for calcium with ammonium oxalate. The residue, instead of being fused with sodium carbonate, is reduced by ignition with a little pure carbon. It is then dissolved in 4N acetic acid and examined for strontium and barium in the usual way.

For separating chromium from a mixture of hydroxides of iron, manganese, chromium, calcium, and magnesium, it is better to oxidise with sodium hypochlorite. When sodium peroxide is used, it cannot be completely washed out of the residue, and on acidification the hydrogen peroxide formed reduces manganese dioxide and the manganese, passing into solution with calcium and magnesium, is precipitated with the latter. For detecting zinc in the sodium sulphide precipitate, the latter is boiled with alkaline sodium hypochlorite, zinc and chromium passing into solution. The solution is acidified with acetic acid, the chromium precipitated as barium chromate, and the presence of zinc confirmed in the usual way. In the presence of oxalic acid the test for aluminium fails. The oxalic acid must be destroyed by boiling with 3% hydrogen peroxide and sulphuric acid. (See also J. Chem. Soc., Sept., 1918.)—E. H. R.

*Sodium and potassium [in organic substances]; Determination of —.* S. N. Rhue. J. Ind. Eng. Chem., 1918, 10, 429—431.

To determine sodium and potassium in organic substances, the sulphated ash of the sample is freed from phosphoric acid, iron, calcium, magnesium,

etc., and the residual sodium and potassium sulphates are weighed together. Potassium is then determined in a second portion of the ash by precipitation with platinum chloride, the iron, calcium, etc., having been removed previously. The potassium platinichloride precipitate is washed with 80% alcohol, then with 20% ammonium chloride solution to remove calcium and magnesium chlorides, and again with alcohol. (See also J. Chem. Soc., Aug., 1918.)—W. P. S.

*Phosphoric acid; Critical elaboration of quantitative precipitation methods, exemplified by a method for the determination of —.* H. Heidenhain. J. Ind. Eng. Chem., 1918, 10, 426—429.

SOLUBILITY of the precipitate, its purity, and the influence of concentration, temperature, and presence of other substances are data which must be known before a precipitation method can be termed trustworthy. The author has investigated the determination of phosphoric acid, as phosphomolybdate, on these lines and recommends that the phosphoric acid should be precipitated as potassium phosphomolybdate in order to eliminate ammonium salts which would interfere when the precipitate is titrated subsequently. The precipitate is washed with 10% potassium nitrate solution containing nitric acid (the total solution should have an acidity not exceeding N/100); an allowance is made for the small amount of acid which remains in the precipitate and filter after the final washing. (See also J. Chem. Soc., Aug., 1918.)—W. P. S.

*Phosphoric acid; Determination of — as magnesium pyrophosphate. III. Magnesium tetra-ammonium diphosphate.* D. Balareff. Z. anorg. Chem., 1918, 103, 73—78.

WHEN magnesium ammonium phosphate is precipitated from a solution containing a large excess of ammonium chloride, especially if excess of ammonia is also present, it is contaminated apparently with the compound  $Mg[(NH_4)_2PO_4]_2$ . Attempts to isolate this substance have not been successful. Such contaminated precipitates, after calcination, contain magnesium metaphosphate, which is insoluble in hydrochloric acid. The metaphosphate is only slowly decomposed by heating with a blast flame, but more quickly than the pyrophosphate. It is suggested that magnesium tetra-ammonium diphosphate on heating undergoes decomposition, with the formation of ammonium phosphate, which dissociates, causing the result to be low through loss of phosphoric acid, part of which interacts with magnesium pyrophosphate to form metaphosphate. (See also J. Chem. Soc., Sept., 1918.)—E. H. R.

*Molybdenum; Determination of — in presence of copper.* W. Hoepfner and O. Binder. Chem.-Zeit., 1918, 42, 315.

IN Classeu's method of separating molybdenum from metals of the copper group by digesting the ammoniacal solution with ammonium sulphide, a considerable amount of copper (up to 0.015 gm. per 100 c.c. of ammonium sulphide) may remain in solution and will be determined with the molybdic acid. To obviate this the copper should be precipitated electrolytically after acidifying the ammoniacal solution with sulphuric acid and adding nitric acid.—C. A. M.

*Halogen derivatives of aromatic amines and their analysis.* I. F. B. Dains, T. H. Vaughan, and W. M. Janney. J. Amer. Chem. Soc., 1918, 40, 930—936.

An account is given of the preparation of iodine derivatives from a number of halogenated anilines.

(For details, see J. Chem. Soc., Aug., 1918.) For the estimation of halogen in these and similar organic compounds, the following process is recommended. A small quantity (about 0.1 gram, or less) is dissolved or suspended in 30 c.c. of liquid ammonia in a Dewar bulb and small pieces of sodium are added, until a slight excess is present as indicated by the blue colour. The mixture is stirred and allowed to stand until all the ammonia has evaporated. Any excess of sodium is dissolved by addition of a small quantity of alcohol, water is added, the solution acidified with nitric acid, and a measured excess of  $N/10$  or  $N/40$  silver nitrate added. The solution is filtered and the filtrate titrated with standard ammonium thiocyanate using ferric alum as an indicator. Calcium was used with success in the place of sodium in the case of *p*-chloroaniline.—W. G.

*Errors in ultimate analysis of coal due to the presence of carbonates.* Knublauch. See IIa.

*Determination of toluene in crude petroleum.* Bowrey. See IIa.

*Determination of naphthalene in tars and tar oils.* Knublauch. See III.

*Method for measuring the coefficient of expansion of pitch, asphaltum, and similar substances.* Kawakami. See III.

*Determination of anthraquinone.* Lewis. See III.

*The Lawrence-Smith method for the determination of alkalis in silicates.* Wenger and Brange. See VII.

*Precipitation of magnesium oxalate, and theory of the formation of precipitates.* Astruc and Camo. See VII.

*Determination of nitric acid in bismuth subnitrate.* Luce. See VII.

*Analysis of spent oxide.* See VII.

*Determination of halogens, sulphur, and nitrogen in the presence of mercury.* François. See VII.

*Oxygen in iron.* Schmitz. See X.

*Analysis of zinc dust.* Wilson. See X.

*Gravimetric and volumetric determination of zinc as zinc mercury thiocyanate.* Jamieson. See X.

*Precision method for the determination of gases in metals.* Ryder. See X.

*Determination of tin in wolfram ores and concentrates.* Smoot. See X.

*Determination of the purity of castor oil.* Frabot. See XII.

*Chemical tests for the detection of rancidity.* Kerr. See XII.

*Determination of organic matter in soils by the loss-on-ignition method.* Rather. See XVI.

*Analysis of calcium cyanamide containing large percentages of diacyanodiamide and urea.* Hene and van Haaren. See XVI.

*Determination of diacyanodiamide in old nitrolim (crude calcium cyanamide).* Kappen. See XVI.

*Determination of citric-soluble phosphoric acid in superphosphates.* Wagner. See XVI.

*Glucosazone reaction.* Garard and Sherman. See XVII.

*Detection of cider in [white] wine.* Mach and Fischler. See XVIII.

*Detection of added colour in butter or oleomargarine.* Lubs. See XIXa.

*Detection of nitrates in water.* Escaich. See XIXb.

*Phosphotungstates of certain bases and amino-acids.* Drummond. See XX.

*Evaluation of balsam of tolu.* Cocking and Kettle. See XX.

*Analysis of valerian root.* Rüyden. See XX.

*Estimation of pulegone and other ketones in oil of pennyroyal.* Bennett. See XX.

*Identification of guaiacol carbonate. A test for ether.* Maue. See XX.

*Method of calculating the comparative strength and efficiency of high explosives from their composition and apparent densities.* Waller. See XXII.

#### PATENTS.

*Velocity of a fluid and velocity of a body through a fluid; Measurement of —.* I. Williams, Bristol. Eng. Pat. 116,385, July 4, 1917. (Appl. No. 9612 of 1917.)

ELECTRICALLY heated wires connected in series are arranged transversely across a tube through which fluid is passing. Thermocouples are arranged at both ends of the tubes with their junctions outside, and a connecting platinum wire is fused through the wall of the tube. The thermocouples are connected to a sensitive galvanometer so as to measure the temperature difference between the ends of the tube. The galvanometer may be graduated in terms of velocity of the fluid. The apparatus may be similarly used to determine the velocity of a body through a fluid.—W. F. F.



*Gases of varying density; Apparatus for and actuated by* — J. F. Simmance and J. Abady, London. U.S. Pat. 1,264,985, May 7, 1918. Date of appl., Nov. 25, 1912. Renewed Nov. 27, 1916.  
SEE Eng. Pat. 27,484 of 1911; this J., 1912, 846.

*Soil-testing apparatus.* U.S. Pat. 1,259,669. See XVI.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Booth and Roberts. Furnaces, etc. 12,337. July 29.

Calvert. Apparatus for catalytic reactions. 12,323. July 29.

Carter and others. 12,457. See IX.

Drake and Gorham. Acid pumps for emptying carboys, etc. 12,320. July 29.

Dressler Tunnel Ovens, Ltd., and Rushton. 12,896 and 12,897. See VIII.

Hofmann. Apparatus for drying goods in bulk. 13,200. Aug. 14.

Hofmann. Mechanically-stoked furnace. 13,288. Aug. 15.

Ionides. Drying powdered material. 12,760. Aug. 6.

Jones, and Jones and Attwood. Apparatus for diffusing or supplying air in small globules. 13,391. Aug. 17.

Jorgensen. 12,980. See IX.

Kestner Evaporator and Engineering Co., and Reavell. Evaporators. 12,962. Aug. 9.

Lucas. Furnaces. 13,383. Aug. 17.

Pedersen. Separation of solid substances from liquid in which they are contained or suspended. 12,581. Aug. 1. (Norway, Sep. 13, 1917.)

Phillips. Tubular condensers. 12,845. Aug. 8.

Preville. Apparatus for raising acids, etc. 12,894. Aug. 8.

Thompson. Crucibles. 12,383. July 30.

Windram. Separating crystals from mother liquor. 13,137. Aug. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9893 (1917). Ionides. Furnaces. (117,923.) Aug. 21.

11,039 (1917). Wilton, and Chemical Engineering and Wilton's Patent Furnace Co. Apparatus for treating gases with liquids. (117,843.) Aug. 14.

11,512 (1917). Wright. Art of drying materials. (117,704.) Aug. 8.

11,593 (1917). Rowlands. Apparatus for separating dust or powder from gases or air. (117,957.) Aug. 21.

16,423 (1917). Duodo. Evaporating and distilling apparatus. (111,480.) Aug. 8.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Aktiebol. Ingeniörsfirma Egnell. 12,483 and 12,588. See XXIII.

Anderson. Apparatus for producing oil fuel. 12,706. Aug. 3.

Anderson, Brady, and Deacon. Furnaces for distilling coal, etc. 13,221. Aug. 14.

Barrow, Scott, and Cadbury Bros. Suction gas producers. 12,759. Aug. 6.

Barton and Oddy. Purifying and decolorising hydrocarbons, etc. 12,992. Aug. 10.

Beccari. Oven for making charcoal and gas. 13,163. Aug. 13.

Beccari. Continuous action oven or pyre for making charcoal. 13,162. Aug. 13.

Bone. Treatment of fuels to improve their calorific value. 12,399. July 30.

Chapman Engineering Co. Gas-producers. 13,072. Aug. 12. (U.S., Apr. 20.)

Davidson and Liversedge. Gas-washers. 12,334. July 29.

Everett. Apparatus for treating carbonaceous materials. 12,875. Aug. 8.

Galecki. Apparatus for making solid combustible from vegetable earth. 12,829. Aug. 7.

Garrow. Carbonisation of coal, oil, shale, peat, wood, etc. 12,509. July 31.

Grylls. Carbons for electric arc lamps. 12,661 and 12,662. Aug. 2.

Hackford, Smith, and Pearson and Son. Liquid fuel. 13,285. Aug. 15.

Heise. Gas purifiers. 13,017. Aug. 10.

Heise. Gas generators. 13,018, 13,019, and 13,020. Aug. 10.

Helps. Manufacture, utilisation, and combustion of fuel. 13,256. Aug. 15.

Hill-Jones, Ltd., and Hughes. Manufacture of artificial fuel. 13,414. Aug. 17.

Hislop and Simpson. Gas manufacture. 12,955 and 13,404. Aug. 9 and 17.

Leadbeater. Treating peat to obtain carbon for decolorising. 13,111. Aug. 13.

Parsons and West. Oil-gas producer. 13,068. Aug. 12. (U.S., Aug. 16, 1917.)

Pearson. Distillation of shale, etc. 12,590. Aug. 1.

Phillips. Mechanical retorts or heating chambers. 12,846. Aug. 8.

Smith. Operation of water-gas plants. 12,694. Aug. 3.

Soddy. Separating and fractionating constituents of coal gas by means of charcoal, and improving absorptive power of charcoal for gases. 13,117. Aug. 13.

Tweedale. Manufacture of gas. 12,543. Aug. 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

16,709 (1916). Helps. Utilisation and combustion of fuel. (117,637.) Aug. 8.

8139 (1917). Martin. *See* III.

8743 and 17,298 (1917). Turner. Processes of destructive distillation. (117,645.) Aug. 8.

9248 (1917). Ensulko Seito Takushoko Kabushiki Kaisha, Okada, and Miyake. Manufacture of decolorising carbon. (117,828.) Aug. 14.

9701 and 15,495 (1917). Hughes. Fuel from waste materials. (117,921.) Aug. 21.

10,703 (1917). Espenhahn. *See* VII.

10,759 (1917). Shillstone. Decolorising carbons and method of producing same. (116,253.) Aug. 8.

10,974 (1917). Wellington. Apparatus for destructive distillation of carbonaceous materials. (117,929.) Aug. 21.

11,745 (1917). De Dampierre. Manufacture of volatile oils such as petrol. (109,796.) Aug. 21.

12,625 (1917). Solar. Fuel for use in explosion engines. (109,802.) Aug. 8.

13,041 (1917). Turner. Gas-producer plant. (117,721.) Aug. 8.

16,272 (1917). Dyson. Liquid fuel. (118,021.) Aug. 21.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Barton and Oddy. 12,992. *See* II.

Perruche, and Gros et Bouchardy. Production of nitro compounds. 12,454. July 31. (Fr., Sep. 24, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

8139 (1917). Martin. Producing oils from sharp, middle, creosote, or similar oils. (117,914.) Aug. 21.

10,786 (1917). Dutt and Hamer. Purification of commercial benzol and toluol from thiophene and other similar compounds. (117,683.) Aug. 8.

## IV.—COLOURING MATTERS AND DYES.

## COMPLETE SPECIFICATION ACCEPTED.

201 (1917). Oman. Preparation of azo colouring matters from ligninesulphonic acid or its salts. (103,822.) Aug. 8.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Albrecht. 13,251. *See* XXII.

Budde, and Hendon Paper Works Co. Means for separating cellulose from straw, etc. 12,392. July 30.

Courtaulds, Ltd., and Criggal. Squirtling-nozzles for making strips, etc., from viscose, etc. 12,513. July 31.

Craig, and Spence and Sons. Treatment of textile fibres and fabrics, etc., to render them less inflammable. 12,442 and 12,683. July 31 and Aug. 3.

Grant. Waterproofing paper, etc. 13,165. Aug. 13.

Grolea and Wayler. 12,456. *See* XIII.

Harrison. Treatment of cotton yarns and fabrics. 12,436. July 31.

Partington. Pulp for making paper, etc. 12,930. Aug. 9.

Spicer, and Spicer and Sons. Treatment of parchmentised or vulcanised fibre. 12,663. Aug. 2.

## COMPLETE SPECIFICATIONS ACCEPTED.

201 (1917). Oman. *See* IV.

5769 and 14,675 (1917). Gillard and Heslam. Treatment of textile fabrics, wood, leather, etc. (117,640.) Aug. 8.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

## APPLICATIONS.

Harrison. 12,436. *See* V.

Hindle. Printing woven fabrics. 13,381. Aug. 17.

## COMPLETE SPECIFICATIONS ACCEPTED.

5769 and 14,675 (1917). Gillard and Heslam. *See* V.

12,509 (1917). Calico Printers' Assoc., and Fourneaux. Production of aniline black on vegetable textile fibres. (117,987.) Aug. 21.

12,510 (1917) and 3287 (1918). Calico Printers' Assoc., and Ashton. Mordanting vegetable fabrics and fibres. (117,715.) Aug. 8.

15,364 (1917) and 6685 (1918). Rydings. Applying starch or other adhesive to cops. (118,014.) Aug. 21.

1413 (1918). McComb and McComb. *See* XII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

## APPLICATIONS.

Ascroft. Dehydration of metal salts. 12,345. July 29.

Bithell. Apparatus for making hydrochloric, nitric, and sulphuric acids, etc. 12,728. Aug. 6.

Chance and Hunt, and Glidden. Manufacture of arsenic chloride. 12,965. Aug. 9.

Cox and Matheson. Treatment of alunite. 12,575. Aug. 1.

Distillates, Ltd., and Jarman. Separation of potassium and sodium salts. 12,646. Aug. 2.

Dixon and Harvey. 12,711. *See* XI.

Drake and Gorham. 12,320. *See* I.

Electrolytic Zinc Co. Treatment of zinc solutions preparatory to recovering zinc by electrodeposition. 13,368 and 13,369. Aug. 16. (Australia, Aug. 17, 1917.)

Harger, and Lever Bros. Manufacture of hydrogen, nitrogen, and carbon dioxide. 12,565. Aug. 1.

Hodgkinson. 12,881. *See* XXII.

Hodgkinson and others. 13,294. *See* XX.

Maxted and Smith. Removal of ammonia from high-pressure gases. 12,684. Aug. 3.



Maxted and Smith. Burner for oxidation of ammonia. 13,037. Aug. 12.

Preville. 12,894. *See* I.

Silberrad. Manufacture of salts. 12,757. Aug. 6.

Soc. Gros et Bouchardy. Production of nitrogen dioxide. 12,453. July 31. (Fr., Aug. 20, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,946 (1915). Devaucelle. Manufacture of cyanides and ammonia. Aug. 21.

5063 (1917). Howorth (Norsk Hydro-Elektrisk Kvaestofaktieselskab). Treatment of nitrate of ammonia. (117,823.) Aug. 14.

9347 (1917). Freeth. Manufacture of sulphuric acid from nitre cake. (117,649.) Aug. 8.

10,703 (1917). Espenhahn. Recovery of cyanogen-bearing compounds from coal gas, etc. (109,254.) Aug. 21.

11,224 (1917). Levin. Manufacture of metallo-pyrophosphoric acids or their salts associated with boron compounds or containing boron, and application of the products to tanning. (117,693.) Aug. 8.

11,256 (1917). James, Vint, and Alexander. Apparatus for separating nitrogen and oxygen from liquid air. (117,937.) Aug. 21.

12,887 (1917). Stafford Coal and Iron Co., and Richmond. Recovery of potassium salts from blast furnace flue dust or the like. (117,991.) Aug. 21.

16,013 (1917). Tschirner. Manufacture of potassium compounds from glauconite, etc. (117,870.) Aug. 14.

16,157 (1917). Scholes. Manufacture of potassium compounds. (117,755.) Aug. 8.

3528 (1918). Mineral Products Corporation. Purification of alumina. (118,063.) Aug. 21.

9672 (1918). Dehn (Henshaw). Obtaining alkaline earth fluorides and soluble potassium salts. (117,901.) Aug. 14.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Brooke and Twynam. Refraction bricks or blocks. 12,811. Aug. 7.

Dressler Tunnel Ovens, Ltd., and Rushton. Ovens. 12,896 and 12,897. Aug. 8.

Forster. Feeding molten glass from glass tanks or furnaces. 12,438. July 31.

Hailwood. Highly refractory compositions for use in building and repairing acid-lined metallurgical furnaces, etc. 12,744. Aug. 6.

Soc. Anon. des Prod. Refractaires de l'Ouest. Manufacture of silica bricks. 12,788. Aug. 6. (Fr., Aug. 6, 1917.)

#### COMPLETE SPECIFICATION ACCEPTED.

13,780 (1917). Webb. *See* XI.

#### IX.—BUILDING MATERIALS.

##### APPLICATIONS.

Carter, Ratcliffe, and Wright. Impregnation of porous or permeable substances with liquid or plastic materials. 12,457. July 31.

Jorgensen. Preventing or removing crust formation in rotary kilns. 12,986. Aug. 10. (Sweden, Feb. 28, 1917.)

Mulligan. Non-porous cement. 12,478. July 31.

Pedersen. Process for making wood lastingly soft and pliable. 13,022. Aug. 10.

Powell. Treatment of timber. 13,214. Aug. 14.

Rayner and Rayner. Materials for walls, ceilings, etc. 12,805 and 12,807. Aug. 7.

Rigby. Cement and concrete, and manufacture of cement. 13,306. Aug. 16.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Alloy Welding Processes, Ltd., and Jones. Electrodes for soldering and depositing metals by electric arc. 13,025 and 13,026. Aug. 10.

Aramaki. Zinc-melting apparatus. 13,183. Aug. 14.

Bate and Williams. Converting granulated aluminium to a state for quick reduction to powder, etc. 12,350. July 30.

Blackwell. Manufacture and application of ferro-tungsten. 12,363. July 30.

Blackwell. Manufacture of ferro-titanium. 12,364. July 30.

Dixon. Extraction of gold. 12,458. July 31.

Electrolytic Zinc Co. 13,368 and 13,369. *See* VII.

Garred. Smelting ores, etc. 12,969. Aug. 9.

Guess. Electrolytic separation of nickel from copper. 13,231. Aug. 14. (U.S., Aug. 14, 1917.)

Hadfield. Manufacture of alloy steel. 12,332. July 29.

Hailwood. 12,744. *See* VIII.

Hulst. Treatment of lead bullion. 12,781. Aug. 6.

Jones. Treating metal articles to improve their quality. 13,054. Aug. 12.

Marino. Electrolytic cleaning of iron or steel. 12,960. Aug. 9.

Sarraméa. Bath for electrolytic tinning. 13,151. Aug. 13. (Switz., Sep. 27, 1917.)

Scott. Concentration of ores. 13,209 and 13,216. Aug. 14. (U.S., Aug. 17 and 27, 1917.)

Stonier. 12,710. *See* XIII.

Thompson. 12,383. *See* I.

Thompson. Rust-proofing iron and steel articles. 12,384. July 30.

#### COMPLETE SPECIFICATIONS ACCEPTED.

2878 (1917). Alexander, Vint, and Imbery. Annealing metal wire by electricity. (117,822.) Aug. 14.

5632 (1917). Dekker. Electrolytic deposition of metals. (117,638.) Aug. 8.

12,887 (1917). Stafford Coal and Iron Co., and Richmond. *See* VII.

13,450 (1917). Price. Crucible furnaces for melting brass and the like for casting purposes. (118,000.) Aug. 21.

16,339 (1917). Bolton and Walker. Deposition of metals by electrolysis. (117,872.) Aug. 14.

17,078 (1917). York and Taylor. Hardening, tempering, and case-hardening metal articles. (117,761.) Aug. 8.

17,302 (1917). Harvey, and Morgan Crucible Co. Crucible melting-furnaces heated by gas or oil. (118,037.) Aug. 21.

17,931 (1917). Hoyle and Burn. Annealing, tempering, or heat-treatment furnaces for metals. (118,040.) Aug. 21.

2064 (1918). Sundberg. Separation of mineral values from ores by flotation. (117,781.) Aug. 8.

8076 (1918). McDermott. Apparatus for concentrating crushed ores. (118,074.) Aug. 21.

## XI.—ELECTRO-CHEMISTRY.

### APPLICATIONS.

Benjamin. Electrolytic apparatus. 12,567. Aug. 1. (U.S., Aug. 1, 1917.)

Dixon and Harvey. Production of ammonia gas or ammonium hydrate by electro-decomposition of water and air. 12,711. Aug. 3.

Electrolytic Zinc Co. 13,368 and 13,369. *See* VII.

Exley and Leitner. Electric cells. 12,487. July 31.

Grylls. 12,661 and 12,662. *See* II.

Guess. 13,231. *See* X.

Hutchinson. Accumulators, storage batteries, etc. 12,749. Aug. 6.

Marino. 12,960. *See* X.

Rondelli. Electric furnaces. 13,024. Aug. 10.

Sarraméa. 13,151. *See* X.

Spencer, and Tudor Accumulator Co. Secondary electric batteries or accumulators. 12,653. Aug. 2.

### COMPLETE SPECIFICATIONS ACCEPTED.

5632 (1917). Dekker. *See* X.

12,357 (1917). Oldham and Oldham. Galvanic batteries. (117,981.) Aug. 21.

13,780 (1917). Webb. Melting refractory substances by electric arcs. (115,017.) Aug. 21.

16,321 (1917). Watson and Co., and Travis. Electric furnaces. (118,023.) Aug. 21.

16,339 (1917). Bolton and Walker. *See* X.

3414 (1918). Dean, and Chloride Electrical Storage Co. Electric accumulators. (118,062.) Aug. 21.

## XII.—FATS; OILS; WAXES.

### APPLICATIONS.

De Bruyn. Imparting flavour and aroma to edible oils and fats. 13,091. Aug. 12.

Jackson (Twitchell Process Co.). Refining oils. 13,097. Aug. 12.

### COMPLETE SPECIFICATIONS ACCEPTED.

9120 (1917). Soc. de Stearinerie et Savonnerie de Lyon, and Berthon. Saturation of unsaturated fatty acids and their glycerides. (107,969.) Aug. 8.

9248 (1917). Okada and others. *See* II.

10,759 (1917). Shilstone. *See* II.

11,336 (1917). Zwicky. *See* XIX.

1413 (1918). McComb and McComb. Detergents or washing compounds. (117,885.) Aug. 14.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

### APPLICATIONS.

Damard Lacquer Co., and Potter. Formaldehyde condensation product. 12,312. July 29.

Grolea and Wayler. Production of dopes, varnishes, coatings, etc., with a cellulose-acetate basis. 12,456. July 31. (Fr., Feb. 25.)

Prior. Adhesives or lacquer. 13,218. Aug. 14.

Rogers. Manufacture of oil pastes from wet precipitated pigments and separation of free water therefrom. 13,236. Aug. 14. (Australia, July 24, 1917.)

Stonier. Preparation of zinc white from zinc ores. 12,710. Aug. 3.

### COMPLETE SPECIFICATIONS ACCEPTED.

11,789 (1917). Hiltermann. Production of condensation products from phenols and formaldehyde. (117,857.) Aug. 14.

3900 (1918). Illeman and Montgomerie. Paint. (117,785.) Aug. 8.

## XV.—LEATHER; BONE; HORN; GLUE.

### APPLICATIONS.

Mauerhofer. Digesters for steam-treating glue, etc. 12,971. Aug. 9. (Fr., Feb. 5, 1916.)

Mauerhofer. Treatment of hides and leather. 12,972. Aug. 9. (Fr., Feb. 9, 1916.)

Owen. Liming hides or skins. 12,445. July 31.

Prior. 13,218. *See* XIII.

### COMPLETE SPECIFICATIONS ACCEPTED.

5769 (1917). Gillard and Heslam. *See* V.

9812 (1917). Tullis. Tanning. (117,922.) Aug. 21.

11,224 (1917). Levin. *See* VII.

11,347 (1917). Levin. Tanning of hides and skins. (117,941.) Aug. 21.

## XVI.—SOILS; FERTILISERS.

### APPLICATION.

Imray (Soc. Chem. Ind. in Basle). 12,041. *See* XX.

### COMPLETE SPECIFICATION ACCEPTED.

1793 (1918). Simon-Carves, Ltd., and Brown. Apparatus for making superphosphate and other artificial manures. (118,055.) Aug. 21.

## XVII.—SUGARS; STARCHES; GUMS.

### APPLICATION.

Leadbeater. 13,111. *See* II.

### COMPLETE SPECIFICATIONS ACCEPTED.

9248 (1917). Okada and others. *See* II.

10,759 (1917). Shilstone. *See* II.



## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATIONS.

Faulkner. Malt preparations. 13,013. Aug. 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

7514 (1917). Robinson. Aeration and carbonisation of wort and other liquids. (117,911.) Aug. 21.

10,632 (1917). Anderschou, Lambshead, and Ramsay. Treatment of brewers' yeast to render it suitable for baking purposes. (117,666.) Aug. 8.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Coleman and Jones. Treatment of cereals and their products. 13,344. Aug. 16.

De Bruyn. 13,091. *See* XII.

Ferguson and Peyton. Food preparations. 12,779. Aug. 6.

Hoogenhuyze and Terwen. Manufacture of decolorised, odourless, and tasteless albumins from blood. 13,157 and 13,158. Aug. 13.

Jones, and Jones and Attwood. Purification of sewage, etc. 13,379. Aug. 17.

Jones, and Jones and Attwood. 13,391. *See* I. Mapleton, and Mapleton's Nut Food Co. Food preparation. 12,499. July 31.

Nicholson. Treatment of sewage sludge, etc. 12,338. July 29.

## COMPLETE SPECIFICATIONS ACCEPTED.

8252 (1917). Moseley and M'Cardell. Insecticides. (117,916.) Aug. 21.

9274 (1917). Greville. Treating flour. (117,917.) Aug. 21.

10,632 (1917). Anderschou and others. *See* XVIII.

11,336 (1917). Zwicky. Apparatus for producing food and obtaining fat from animal carcasses and refuse. (117,852.) Aug. 14.

12,130 (1917). Butler. Apparatus for producing dry milk or milk powder. (117,713.) Aug. 8.

1069 (1918). Mapleton's Nut Food Co., and Mapleton. Preparation of foodstuffs. (118,052.) Aug. 21.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Boots Pure Drug Co., and Dutt. Manufacture of guaiacol. 12,859. Aug. 8.

Du Pont de Nemours and Co. Purifying camphor. 13,076 and 13,077. Aug. 12. (U.S., Mar. 16 and 23.)

Grolea and Wayler. Production of esters. 12,455. July 31. (Fr., Feb. 23.)

Hodgkinson, Jephcott, and Martin. Extraction of nicotine, potash, etc., from tobacco waste. 13,294. Aug. 15.

Imray (Soc. Chem. Industry in Basle). Manufacture of the organic phosphorus-reserve-compound of green plants and salts thereof. 12,401. July 30.

Orton. Manufacture of tetranitromethane. 12,714. Aug. 3.

Perruche, and Gros et Bouchardy. 12,454. *See* III.

## COMPLETE SPECIFICATIONS ACCEPTED.

5537 and 12,885 (1917). Irvine and Haworth. Preparation of alkylating reagents. (117,824.) Aug. 14.

9120 (1917). Soc. de Stearinerie et Savonnerie de Lyon, and Berthon. *See* XII.

15,756 (1917). Soc. Chim. des Usines du Rhône. Manufacture of ethylidene diacetate. (112,766.) Aug. 8.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATIONS.

Christensen. Producing coloured photographic pictures. 12,591. Aug. 1.

Comstock, and Technicolor Motion Picture Corporation. Production of complementary images. 13,270. Colour printing. 13,271. Aug. 15.

## COMPLETE SPECIFICATIONS ACCEPTED.

15,226 (1917). Douglass. Films for natural colour cinematography. (117,864.) Aug. 14.

2910 (1918). Hess-Ives Corporation. Coloured photographic image and method of producing same. (113,617.) Aug. 14.

## XXII.—EXPLOSIVES; MATCHES.

## APPLICATIONS.

Albrecht. Fabric for manufacture of bags for propellant explosives. 13,251. Aug. 15.

Curtis's and Harvey, Greene, and Tunks. Treatment of explosives for recovery of solvents. 12,916. Aug. 9.

Harrison (Soc. d'Etudes Chimique pour l'Industrie). Preventing explosion in preparation and handling of explosives. 12,368. July 30.

Hodgkinson. Production of azides. 12,881. Aug. 8.

Perruche, and Gros et Bouchardy. 12,454. *See* III.

## COMPLETE SPECIFICATION ACCEPTED.

18,034 (1916). Creischere and Creischer. Machine for making pellets from pulverulent or granular explosive materials. (117,904.) Aug. 21.

## XXIII.—ANALYSIS.

## APPLICATIONS.

Aktiebolaget Ingeniörsfirma Egnell. Gas-analysing apparatus. 12,483 and 12,588. July 31 and Aug. 1. (Ger., July 31 and Aug. 1, 1917.)

Davis and Davis. Quantitative estimation of vapours in gases. 13,323. Aug. 16.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Evaporator for acid liquids.* E. Hart. J. Ind. Eng. Chem., 1918, 10, 555.

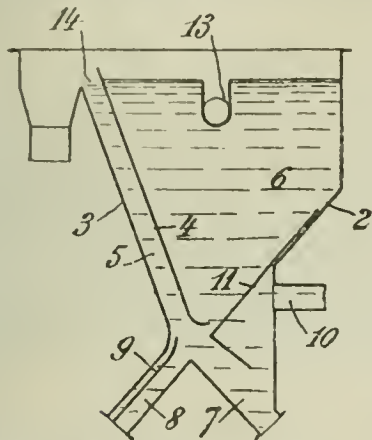
Closed glass tubes, 4 ft. long and 3 in. diam., are arranged horizontally side by side on iron supports covered with asbestos; two or more series of these tubes are placed in steps one higher than the other, and each tube is provided with three 1 in. openings for the introduction of liquid and the escape of steam. The upper series of tubes discharge into the lower and the latter into a collecting channel for the concentrated liquid. The whole system is heated by the gases from a furnace, the liquid being warmed in the first set of tubes and then boiled and evaporated in the second set.

— W. P. S.

**PATENTS.**

*Classifying granular materials; Process and apparatus for —.* R. E. Trottler, Puteaux, France. Eng. Pat. 104,499, Jan. 3, 1917. (Appl. No. 148 of 1917.) Under Int. Conv., Feb. 29, 1916.

In the process claimed, separation is effected not only according to the "equivalence" (i.e., the density and volume) of the particles but also according to their shape. The materials to be separated are introduced into the upper part of the chamber, 6, which is fed with water through the pipe, 10. Any very light bodies remain in suspension and are carried away with the overflow water



through the conduit, 13. The remaining particles fall through the comparatively still water in the upper part of the vessel, 6, and slide down the inclined plates, 2, and 11. The rounded particles of the greatest "equivalence" are able to descend through the upward current of water and pass into the discharge, 7. The rounded particles of less "equivalence" are deviated to the discharge, 8, and the lightest ones to the outlet, 14. All flat particles slide down the plate, 11, and pass into the channel, 5, the inner wall, 4, of which is adjustable. They are turned edgewise by the ascending current of water and slide down the inclined wall, 3, into the discharge, 9.—W. H. C.

*Sorting or separating solid substances; Apparatus for —.* R. E. Trottler, Puteaux, France. Eng. Pat. 117,274, Dec. 22, 1916. (Appl. No. 18,409 of 1916.)

A SIEVE or equivalent device operating in air or any suitable liquid has a jerking motion imparted to it by being drawn backwards by the action of a cam and pulled suddenly forwards against a block by a spring.—W. H. C.

*Separating dust from ores, coal and the like; Apparatus for —.* R. Landien, Ludwigsdorf, Prussia. Eng. Pat. 113,780, Dec. 22, 1917. (Appl. No. 19,005 of 1917.) Under Int. Conv., Feb. 26, 1917.

A CYLINDRICAL separating chamber is surrounded by a receiving chamber, both terminating at the lower end in conical receivers for the dust-free material and the dust respectively. Material to be treated is fed from a hopper on to a "thrower plate" in the inner chamber and meets a current of air produced by a fan situated in a flue connecting the inner and outer chambers. The sectional area at the "thrower plate" where the dust separation takes place, and also that of the return airway from the outer to the inner chamber, is made about equal to that of the fan channel.—C. A. K.

*Volatilised solvents; Recovery by condensation of —.* P. Persch, Cöln-Bransfeld, Germany. Eng. Pat. 116,590, July 28, 1917. (Appl. No. 10,865 of 1917.)

THE mixture of volatilised solvent and air is sucked at a relatively low temperature by a fan into a heater, where it is heated to the boiling point of the solvent to be condensed. The mixture of vapour and air is immediately forced into a cooling chamber where the solvent is condensed and withdrawn through a separator to remove any water. The process is specially applicable to the manufacture of seamless rubber goods, and the whole apparatus may be enclosed and the air replaced by a non-oxidising gas such as nitrogen.—J. H. P.

*Grinding mills, disintegrators, mixers, or the like.* British Dyes, Ltd., J. Turner, and T. P. K. Crossland, Huddersfield. Eng. Pat. 116,958, Aug. 1, 1917. (Appl. No. 11,059 of 1917.)

A CLOSED, slightly conical, grinding cylinder with balls or rollers resting loosely on its bottom, and rotating on a horizontal axis, is provided with a controlled side outlet near the larger end. One end of a curved pipe is attached to the outlet, the other end terminating in a cone which is coaxial with the grinding cylinder and enters dust-tight into a closed receiver. The pipe is rotated with the cylinder. On opening the slide controlling the outlet, the ground material is discharged automatically through the curved pipe as the cylinder rotates, without emission of dust.—W. H. C.

*Grinding or crushing machines.* D. J. G. Miller, Bootle, and R. A. Lloyd, Liverpool. Eng. Pat. 117,281, May 26, 1917. (Appl. No. 7589 of 1917.)

A STATIONARY grinding ring forms the peripheral wall of a chamber the bottom of which consists of a revolving plate. Grinding rollers are supported above and revolve with the bottom plate; the rollers are mounted on arms which pass through the bottom plate and are pivoted below so that the rollers are free to move a limited distance towards and away from the grinding ring. Scrapers and brushes are provided to stir and lift the material.—W. H. C.

*Shaft-furnaces; Method of and apparatus for charging —.* P. Würth, Luxembourg-Hollerich, Luxembourg. Eng. Pat. 117,024, Dec. 8, 1917. (Appl. No. 18,239 of 1917.)

TO economise motive power in charging a shaft furnace, the cars for transporting the charging buckets horizontally are provided with automatic couplings, so that when a car from which a full bucket is suspended is coupled with a car carrying an empty bucket, the two hoisting shafts are also



coupled. The full bucket is then lifted and the empty bucket lowered so that the weights are partly counterbalanced. The cars are then uncoupled and the full bucket transported to the charging point and the empty bucket refilled.

—W. F. F.

*Chemical reactions between gases and/or vapours; Method of and means for bringing about — by means of catalysts.* C. S. Roy, and T. Morson and Son, Ltd., London. Eng. Pat. 117,103, June 2, 1917. (Appl. No. 7941 of 1917.)

AIR or other gas is injected through a nozzle provided with a needle valve into the constricted neck of a mixing chamber. A second gas, or a liquid to be vaporised, fed through a sight tube in a valved pipe, is drawn into the mixing chamber by the injector action of the air jet. If the pressure rises above a predetermined amount, a spring-controlled piston is actuated and operates interconnected valves in the air and vapour supply pipes to control the flow. The mixture is passed, through a preheater if desired, to a casing containing the catalytic substance. The latter is arranged in horizontal tubes and is contained partly in shallow trays in each tube and partly resting in the tube itself. At the beginning of the reaction the catalyst is arranged in two portions, one at each end of the tube, and as the reaction proceeds, the two portions are brought together and then spread out by moving and jerking the trays, the latter being connected to a common rod operated from outside the casing. Excessive rise of temperature is thus prevented. If metallic gauze is used as a catalyst, a number of separate coils are inserted in each tube. The temperature of the catalyst may be varied and controlled by means of heating burners in the base of the casing, and by steam circulated through the casing. The gases pass from the reaction chamber through tubes containing spiral baffle-plates to a purifying tower through which liquid trickles down and thence to an absorbing chamber provided with inclined baffles over which the absorbing liquid flows.—W. F. F.

*Air or gases; Centrifugal apparatus for the extraction and collection of dust from —.* J. C. Wood, Garforth, Yorks. Eng. Pat. 117,173, Aug. 30, 1917. (Appl. No. 12,475 of 1917.)

AIR containing dust is passed into the top of an inner concentric tube in a vertical cylindrical casing. The tube has longitudinal radial ribs extending inwards from the walls, and does not extend to the bottom of the casing, the air passing out under the lower end and upwards through the annular space, which is provided with radial partitions. The apparatus is rotated on its vertical axis, and the dust, which is separated centrifugally, falls to the bottom and is collected.—W. F. F.

*Dust collector.* U. Wedge, Ardmore, Pa., Assignor to The Process Engineering Co., Philadelphia. Pa. U.S. Pat. 1,267,023, May 21, 1918. Date of appl., Sept. 14, 1916.

A CHAMBER is provided with a series of superposed horizontal passages for the flow of the dust-laden gas and with a series of vertical passages through which the deposited dust falls. The vertical passages communicate with the horizontal passages only at the top.—W. H. C.

*Separating matter from a fluid containing the same; Device for —.* U. Wedge, Ardmore, Pa., Assignor to The Process Engineering Co., Philadelphia, Pa. U.S. Pat. 1,267,024, May 21, 1918. Date of appl., Oct. 26, 1916.

THE apparatus consists of a series of superposed tiers of inclined shelves between which the dust-

laden fluid is passed. The superposed tiers of shelves are arranged in pairs, the shelves of each pair being inclined in opposite directions towards a common vertical discharge shoot. The dust is deposited on the shelves, and slides by gravity towards the common shoot, into which it passes through constricted openings.—W. H. C.

*Suspended solids in gases; Process of removing —.* S. Peacock, Philadelphia, Pa., Assignor to Haslup and Peacock, Inc., New York. U.S. Pat. 1,268,486, June 4, 1918. Date of appl., Jan. 26, 1916.

THE gas is treated with a spray of a liquid which will dissolve the solid matter. Any undissolved portions of the latter are circulated by, and subjected to a grinding action in, opposing streams of the solvent while fresh streams of gases are submitted to its action.—W. H. C.

*Covering or jointing of pipes and the like; Material for the —.* V. H. Majerius, Wyke, and J. M. Errington and A. Hammond, Bradford. Eng. Pat. 117,323, Aug. 2, 1917. (Appl. No. 11,110 of 1917.)

THE packing consists of a metallic soap used either alone or mixed with a suitable filling material such as asbestos, china clay, or the like. The metallic base of the soap is preferably chosen so that it forms an insoluble body with the acid or other corrosive material which the jointing has to resist.—W. H. C.

*Drying liquid materials; Method of —.* O. Carr, New York. Eng. Pat. 117,469, Apr. 13, 1917. (Appl. No. 5263 of 1917.)

IN a method of drying liquid by spraying it into a large volume of heated air, the air passes through a drying chamber, a settling chamber, and a concentrating chamber in succession. The liquid is sprayed into the concentrating chamber and the concentrated liquid is collected and sprayed into the drying chamber, where the final evaporation takes place. The dried material separates from the air partly in the drying chamber and partly in the intermediate settling chamber and any particles still remaining in suspension are collected and retained by the liquid spray in the concentrating chamber.

*Solidifying or concentrating liquid or semi-solid materials on a revolving cylinder; Apparatus for —.* J. W. Leitch, Huddersfield. Eng. Pat. 117,560, Nov. 17, 1917. (Appl. No. 16,976 of 1917.)

A GROOVED roller revolves in the molten material, e.g., phenylenediamine, and feeds it on to a revolving drum through which a cooling medium passes. An adjustable scraper removes the solidified material and is situated above a receiving hopper at the bottom of which revolves a worm conveyor. The latter works in close fit within a tubular extension of the hopper, which passes out through the wall of the chamber enclosing the apparatus. The chamber may be evacuated if desired.—J. H. P.

*Drying and the like; Apparatus for —.* A. Tegoli, San Francisco, Cal. U.S. Pat. 1,268,797, June 4, 1918. Date of appl., June 20, 1916.

THE apparatus comprises a blower and a treating chamber provided with coils and connected by flow and return ducts with a series of drying chambers which are also connected together. The latter are provided with interchangeable drawers with apertures in the bottoms.—W. H. C.

*Separation and settlement of solids and semi-solids from chemical and like manufacturing liquids.* W. Clifford, Wolverhampton, and Jones and Attwood, Ltd., Ambleside, Staffs. Eng. Pat. 117,472, May 24, 1917. (Appl. No. 7467 of 1917.)

THE liquid is fed into a bucket in the centre of the settling tank at the top and overflows from the bucket into a relatively large body of liquid inside a guard chamber, open at the bottom, which is adapted to direct the relatively slow moving and large volume downwards in the centre of the body of liquid in the tank, thereby preventing disturbance of the settled solids at the bottom of the tank.

—J. H. P.

*Settling tanks for clarifying liquids.* Simon-Carves, Ltd., and S. Hunter, Manchester. Eng. Pat. 117,576, Jan. 30, 1918. (Appl. No. 1734 of 1918.)

A SETTLING tank is provided with a conical outlet on which are arranged three steam inlets. To remove sediment which has become more or less solid on prolonged stoppage and will not flow through the exit, steam is introduced through the steam inlets. A steam ejector or the like may also be provided to draw water from the upper part of the tank and force it into the sediment after it has passed the outlet valve.—J. H. P.

*Settling tanks for clarifying liquids.* Simon-Carves, Ltd., and S. Hunter, Manchester. Eng. Pat. 117,586, Mar. 21, 1918. (Appl. No. 4993 of 1918.) Addition to Eng. Pat. 117,576, Jan. 30, 1918 (see preceding abstract).

WATER under pressure is used instead of steam as described in the former patent.—J. H. P.

*Spraying and atomising liquids; Apparatus for —.* E. Slater, Greenfield, Yorks. Eng. Pat. 117,481, July 13, 1917. (Appl. No. 10,147 of 1917.)

IN an apparatus for forcing liquid to spraying nozzles, an ejector is connected with the liquid container and with the compressed air container. When the air pressure is normal the ejector is closed by a valve and compressed air passes to the liquid container and forces liquid to the spraying nozzles. If the air pressure falls, the valve is opened by a spring and the compressed air actuates the ejector, which draws in air from the space above the liquid in its container and thus stops the supply of liquid to the spraying nozzles.—J. H. P.

*Steam-generator; Slag-heated —.* U. Wedge, Ardmore, Pa. Assignor to Furnace Patent Co., Philadelphia, Pa. U.S. Pat. 1,267,022, May 21, 1918. Date of appl., July 16, 1914. Renewed Apr. 27, 1916.

MOLTEN slag is delivered into the upper part of a chamber partly filled with water and falls on to a baffle-plate whence it passes into the water in the form of spray and generates steam. The first chamber is connected with a second which has the form of a tubular heater or condenser. The steam and water spaces of the first chamber are connected with the steam and water spaces of one section of the heater, the other being supplied with water from a separate source.—W. H. C.

*Mixing machine.* W. F. Morse, Assignor to S. R. Parry, Rochester, N.Y. U.S. Pat. 1,267,578, May 28, 1918. Date of appl., Mar. 19, 1917.

A CONTAINER for the material to be mixed is clamped on to a turntable which rotates on antifriction

rollers mounted in pockets around the inside of the wall of a hollow base. Paddles carried by a rod supported in a bracket fixed to the base project into, and pass between, paddles fixed to the inner wall of the container.—J. H. P.

*Agitator. Agitating means.* N. D. Nielsen, Elyria, Ohio. U.S. Pats. (A) 1,268,601 and (B) 1,268,602, June 4, 1918. Dates of appl., Sept. 4, 1917, and Jan. 9, 1918.

(A) A TANK is constructed with its bottom dished inwards, and a horizontal shaft projects through the side wall and carries a propeller in the angle formed by the side walls and bottom. A stream of liquid is thus directed against the inclined bottom and then deflected against the opposite side walls which are inclined to one another in such a way that the liquid is divided into two streams moving in opposite directions back to the propeller. (B) In a modification of the apparatus described in (A), the bottom of the tank is dished outwards and the propeller is provided with substantially straight radial blades, so that the liquid streams are directed radially in a vertical plane. The liquid is deflected by the side walls and bottom on to the walls opposite the propeller, and then deflected back to the propeller.—W. F. F.

*Refrigerating composition.* F. G. Keyes, Boston, Mass. U.S. Pat. 1,267,772, May 28, 1918. Date of appl., Aug. 7, 1917.

THE volatile refrigerant, e.g., ammonia, is absorbed in an aqueous solution that has a lower vapour pressure than water, e.g., a mixture of 60% of ammonium nitrate, 25% of glycerin, and 15% of water.—W. H. C.

*Separating liquids from solids; Process of and apparatus for —.* A. M. Nicholas, Bismuth, N.S.W. U.S. Pats. 1,268,273 and 1,268,274, June 4, 1918. Date of appl., Nov. 24, 1916.

A SHALLOW cylinder open at one end is rotated about its horizontal axis. The interior of the cylinder is covered with filtering material on a suitable perforated support spaced away from the wall, and the space between the two is divided into separate compartments by partitions placed parallel to the axis of the cylinder. As the cylinder rotates these compartments are successively connected with a source of vacuum and with a supply of fluid under pressure. The mixture of liquid and solid is fed on the lowest part of the interior which is in communication with the vacuum and the liquid portion is sucked through. As the cylinder rotates the spaces come successively into the position opposite the supply of compressed air or other fluid, and the solids are blown off into a discharge shoot.

—W. H. C.

*Aerating device.* J. P. Ruth, jun., Denver, Colo. U.S. Pat. 1,268,630, June 4, 1918. Date of appl., May 31, 1917.

LIQUID to be aerated is contained in a tank provided with a central vertical pipe having an enlargement at the bottom with radial passages which communicate with the atmosphere through the central pipe. Another set of radial passages in the enlargement communicates with a short central depending pipe opening into the liquid near the bottom of the tank. The central pipe carries a pulley at the top, and when rotated liquid and air are circulated through the passages in the enlargement at the bottom.—W. F. F.



*Still or retort.* H. MacRae, Wilmington, N.C. U.S. Pat. 1,268,742, June 4, 1918. Date of appl., Mar. 28, 1918.

A VERTICAL cylindrical retort or still having a central tube is divided into compartments by horizontal partitions provided with openings and means for closing the same. The lowest compartment serves as a cooler for the residue from the upper compartments, which are each provided with a separate internal heating coil and with separate offtake pipes for the distillate.—W. H. C.

*Treating certain mineral and chemical products: Apparatus for —.* C. J. Leyes, Jersey City, N.J. U.S. Pat. 1,270,307, June 25, 1918. Date of appl., Feb. 20, 1917.

A SERIES of inclined conveyor tubes pass through a furnace chamber and are connected at their upper ends with a common feed hopper and at their lower ends with a common receiver. The tubes, hopper, and receiver are supported by a frame so that the whole can be tilted in a vertical plane independently of the furnace.—W. H. C.

*Fire-extinguishing composition having a low freezing-point.* G. E. Ferguson, New York, Assignor to Pyrene Manufacturing Co. U.S. Pats. (A) 1,270,392, (B) 1,270,394, (C) 1,270,395, (D) 1,270,396, and (E) 1,270,397, June 25, 1918. Dates of appl., (A) Aug. 12, (B) to (E) Oct. 20, 1914.

FIRE-EXTINGUISHING compositions of low freezing-point are composed of: (A) carbon tetrachloride, trichloromethane, and pentachloroethane; (B) a non-inflammable solvent and acetophenone dissolved therein, the solution having a freezing-point substantially lower than that of the solvent; (C) a stable fire-extinguishing solvent, e.g., carbon tetrachloride, with oil of peppermint dissolved therein; (D) a non-inflammable solvent, e.g., carbon tetrachloride with a camphor substitution product dissolved therein, the composition having a lower freezing-point than the fire-extinguishing component; (E) a carbon and chlorine compound, e.g., carbon tetrachloride, with an organic acid, e.g., propionic acid, dissolved therein.—A. de W.

*Composition having a low freezing-point.* G. E. Ferguson, New York, Assignor to Pyrene Manufacturing Co. U.S. Pat. 1,270,393, June 25, 1918. Date of appl., Oct. 20, 1914.

A SOLUTION comprising carbon tetrachloride and anisol dissolved therein.—A. de W.

*Kneading and mixing of plastic substances: Jacketed troughs for machines for — and for like purposes.* J. E. Pointon, and Perkins Engineers, Ltd., Peterborough. Eng. Pat. 117,005, Oct. 24, 1917. (Appl. No. 15,462 of 1917.)

*Furnaces for heating air.* J. Garner and R. H. Posnett, Runcorn. Eng. Pat. 117,165, Aug. 8, 1917. (Appl. No. 11,377 of 1917.)

*Furnace.* C. Zulver, London, Assignor to L. E. Smith, South Shields. U.S. Pats. 1,270,679 and 1,270,680, June 25, 1918. Date of appl., June 25, 1917.

SEE Eng. Pats. 106,704 and 106,703 of 1916; this J., 1917, 857.

*Use of solid fuel for large scale power production.* Eng. Pat. 117,920. See II A.

## II A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Air from a mine fire; Analyses of —.* A. G. Blakely and H. H. Geist. J. Ind. Eng. Chem., 1918, 10, 552–553.

FIRE broke out in an anthracite coal mine on November 18, 1916, and barriers were erected for the purpose of cutting off all supply of air and smothering the fire. Pipes were extended through the barriers and samples of the gases obtained at intervals from the fire zone. The quantity of oxygen present decreased gradually from 18.00% on December 1, 1916, to 4.80% on July 26, 1917; during this period the carbon dioxide increased, but not regularly, from 0.99 to 4.47%, whilst the methane increased from 2.40 to 10.9%. Carbon monoxide increased from 0.008% on December 1 to a maximum of 0.01% on December 26, then decreased to 0.001% on February 22, after which date none could be detected in the gases.—W. P. S.

*Gas producers from which slag is tapped off: Comparison of — with those of the water seal type.* H. Markgraf. Stahl u. Eisen, 1918, 38, 703–707.

Two producers working with liquid slag have given complete satisfaction for 4 years and another, capable of gasifying 100 tons per 24 hours, is now in course of erection. The producer is charged with dry coke of large size, together with open-hearth or other fusible slag and blown with air only. A gas practically free from hydrogen is produced which, since it yields little or no moisture on combustion, is well suited to the heating of open-hearth furnaces. The sulphur in the gas varies from 0.126 to 0.468%, compared with a variation from 0.49 to 2.0% in gas from the Würth producer. The slag added with the charge readily dissolves the residue from the coke which is thus prevented from interfering with the continuous working of the producer. The addition of excess of a highly phosphoric slag to the coke charged into the producer yields a highly phosphoric pig iron of low melting point. The efficiency of this producer is 23% less than that of the Würth producer, since the slag additions have to be melted, but compensation for this is found in the fact that no heat is used up in the production of water-gas. The gas is very free from dust. Coke very high in ash, useless for the blast furnace, can be used in this producer, with or without slag additions. The life of the producer is from 9 months to a year. The gas produced contains 34.3% CO, and small quantities of hydrogen and methane, the residue being practically all nitrogen. The construction is similar to that of an ordinary producer, there being in addition slag holes below the air tuyères.—F. C. Th.

*Ammonium sulphate: "Direct process" of making — in gasworks.* 54th Annual Report on Alkali Works (Year 1917), by the Chief Inspector, 32–47.

ATTENTION in 1917 was mainly directed to the yield of ammonia from retorts under varying seasonal conditions, with a close watch on the loss of ammonia in the spent liquors from the still. Loss of ammonia at the bubbler has been observed, due to the removal of the tarry scum from the surface of the liquor in the effluent tank. Investigations on the poor yield by the "direct process" show that improvement must now be attained in the carbonising house, rather than in the bubbler, as the process is capable of effecting a recovery of approximately 95% of the ammonia leaving the retort. Chemical processes for the production of ammonia from hydrogen and nitrogen are making rapid progress, but the recovery of ammonia as a

by-product is capable of extended application, and important changes in gasworks practice are indicated. The use of steam in vertical retorts will materially increase the yield of ammonia and an increased use of an economical heat-giving gas is to be anticipated.—C. A. K.

*Oil shales; Destructive distillation of* — J. C. Morrell and G. Egloff. Chem. and Met. Eng., 1918, 19, 90–96.

EXPERIMENTAL distillation of an oil shale from the Green River formation gave as products per ton of shale: oil, 20.3 galls.; permanent gases, 1947 cub. ft.; ammonium sulphate, 25.4 lb. Evolution of ammonia was first noted at 365° C., and increased at an unrecorded temperature above 400° C. The distillate was collected in 100 c.c. fractions; the sp. gr. of the oil portion in the first fraction was higher than in the second or third, with subsequent increase again in later fractions. The ammonia content of the aqueous distillate increased to a maximum. Benzene, toluene, and xylene were present in small amounts in the oil distillate, but difficulty in working up the oil into a motor fuel will be experienced owing to the presence of sulphur, nitrogen, unsaturated hydrocarbons of the terpene type, and phenolic compounds. The higher boiling fractions are lubricating oils of similar properties to those obtained from California and Russian petroleum, and are probably highly naphthenic in character. A shale oil industry will require high technical skill and large capital and competition with petroleum wells in the near future is questionable.—C. A. K.

*Lubricating oils; Dubrisay's method of testing [viscosity of]* — P. Nicolardot and P. J. Masson. Ann. Falsif., 1918, 11, 77–79.

THE authors consider that the results given by the Dubrisay viscometer (this J., 1917, 1123) are materially affected by the density and surface tension of the oils tested, and that at temperatures other than atmospheric the thermal conditions are unsatisfactory. Tests made on different oils at various temperatures show that while a constant relationship exists between the results given by the Ostwald viscometer and the Barbey ixomètre, the relationship between the results given by the Barbey and the Dubrisay instruments varies with different oils.—T. St.

*Analysis of ammoniacal liquors.* See VII.

*Manufacture of amyl acetate and similar solvents from petroleum pentane.* Brooks and others. See XX.

#### PATENTS.

*Coal-washing apparatus.* L. Llewellyn, Assignor to Pittsburgh Coal Washer Co., Pittsburgh, Pa. U.S. Pat. 1,267,565, May 28, 1918. Date of appl., Jan. 5, 1918.

A WASHING tank is provided with a vertical transverse screen terminating below the top, and the coal is received on a perforated platform at one side of the screen and slightly inclined downwards towards its upper end. A similar delivery platform oppositely inclined is provided on the other side of the screen. A horizontal reciprocating shaft extends transversely through the tank below the platforms and carries three plungers of different sizes, one in each compartment below the platforms, and one moving centrally through the screen. A circulation of liquid through the perforated platforms is thus obtained. The receiving platform ter-

minates short of the central screen and the washed out material is discharged into the tank through the space between them. This space may be closed by a gate.—W. F. F.

*Alcohol; Solid — and process of producing same.* A. Strobl, Assignor to S. Sternau and Co., New York. U.S. Pat. 1,266,080, May 14, 1918. Date of appl., Feb. 11, 1915.

CLAIM is made to products consisting of alcohol, metallic soaps, and free fatty acid, and in particular to products containing alcohol, sodium stearate, and stearic acid. For example, alcohol at 140°–160° F. (60°–71° C.) may be treated with 2–3% of stearic acid and the latter subsequently nearly neutralised by addition of sodium hydroxide.

—J. H. L.

*Solid fuel; Process of burning* — O. B. Evans, Lansdowne, Pa., Assignor to The United Gas Improvement Co., Philadelphia, Pa. U.S. Pat. 1,267,646, May 28, 1918. Date of appl., Jan. 3, 1916.

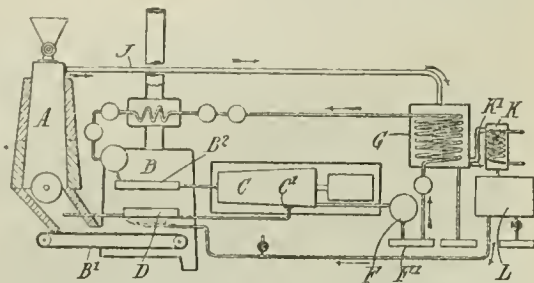
THE fuel is fed into the top of a vertical cylindrical furnace which is constricted near the bottom. The air blast is regulated so that the ash is fused above the constriction and a combustible mixture of clinker and carbon passes downwards. Below the constriction an annular space is left between the wall of the furnace and the mixture of clinker and carbon moving downwards, and air for burning out the carbon from the clinker is injected into this space.—W. F. F.

*Coke-oven.* F. Peiter, Cleveland, Ohio. U.S. Pat. 1,267,332, May 21, 1918. Date of appl., Sept. 11, 1916.

THE combustion flues of a coke oven are arranged in groups of four, and in series in each group. Air for combustion is preheated by passing through a regenerator, and delivered to the top of the first flue of each group. Combustible gas is supplied to the top of the first and third flues, baffles are arranged in the second and third flues, and the hot gases from the fourth flue are led to the regenerator.—W. F. F.

*Power production; Use of solid fuel for large scale* — Merz and McLellan, and A. C. Michie, Newcastle-on-Tyne, and E. G. Weeks, Monk-seaton. Eng. Pat. 117,290, July 6, 1917. (Appl. No. 9817 of 1917.)

COAL is fed continuously into a retort, A, and the coke residue continuously discharged on to the grate, B<sup>1</sup>, of a boiler, B. High-pressure steam from the boiler passes through a high-pressure superheater,



B<sup>2</sup>, to a turbine, C, from which partially expanded "bleed" steam is taken at the point, C<sup>1</sup>, and fed through a low-pressure superheater, D, combined with the superheater, B<sup>2</sup>, in the boiler furnace, to

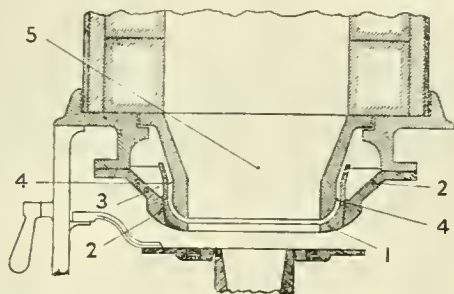


the retort, A, for the low temperature distillation of the coal. The exhaust steam from the turbine passes to a condenser, F, and the condensed water is pumped from the well, F<sup>1</sup>, through the retort gas condenser, G, to cool the latter, and then through the usual feed-water heaters back to the boiler, B. The retort gases pass by the pipe, J, through the water-cooled condenser, G, and pipe, K<sup>1</sup>, to a cooler, K, and then to a stripping plant, L, to recover light oils and other by-products. The gas may then be used for any power purposes, *e.g.*, for heating the boiler, B, as shown, or in other steam generators or internal combustion engines.

—W. F. F.

*Gas producers; Vaporising apparatus of* —. Le Gaz Pauvre Economique, Nantes, France. Eng. Pat. 112,282, Dec. 28, 1917. (Appl. No. 19,166 of 1917.) Under Int. Conv., July 19, 1915.

The base, 5, of the producer is surrounded by an annular water vessel, 3, formed by two members, 1, 2, the member, 1, having a conical seating on the



member, 2. The water is evaporated at a rate corresponding to the temperature of the fire, and the steam is superheated and the fire temperature lowered by passing the steam through the annular space, 4, to the producer.—W. F. F.

*Coal gas; Manufacture and treatment of* —. G. Helps, Nuneaton. Eng. Pat. 117,308. (Appl. Nos. 10,224, July 14, and 12,043, Aug. 22, 1917.)

ALL the gas retorts in a bed are charged simultaneously, and maintained at a pressure above the external pressure in the furnace to prevent admission of inert gases. The gases from several retort beds are led to a common receiver at a temperature sufficiently low to prevent interaction between them. The discharge main from the receiver is provided with one or more nozzles facing upstream, through which air, producer-gas, or water-gas is injected to dilute the coal gas to any desired calorific value.—W. F. F.

*Gas generators or the like; Apparatus for feeding fuel to* —. G. H. Bentley and E. G. Appleby, London. Eng. Pat. 117,348, Sept. 20, 1917. (Appl. No. 13,553 of 1917.)

A ROTARY feeding drum above the generator is operated by a reciprocating rod through a pawl and ratchet wheel, and the fuel falls on to an adjustable distributing cone in the mouth of the retort. By means of an additional ratchet wheel and reverse pawl, and cams, the direction of rotation of the feeding drum is reversed at intervals in order to maintain a level surface of the fuel in the generator. To facilitate access to the distributing cone this is suspended from a balanced lever pivoted on a detachable frame secured to a frame supporting the feeding drum.—W. F. F.

*Gas-producer.* J. W. Horusey, Summit, N.J., Assignor to General Reduction Gas and By-Products Co. U.S. Pat. 1,267,410, May 28, 1918. Date of appl., July 3, 1914. Renewed Aug. 17, 1917.

For the continuous manufacture of producer gas, a horizontal rotating cylinder with a refractory lining is provided internally with sets of lifting blades or pockets, each set being out of alignment with adjacent sets. The material is admitted at one end of the cylinder and its longitudinal movement is obstructed by circumferential rings at intervals. A gas outlet is provided at the charging end of the cylinder, for gas and gaseous by-products, and an outlet at the other end for gas free from tar. Means are provided for directing the flow of gas towards either outlet as desired.

—W. F. F.

*Gas; Manufacture of* —. J. M. Rusby and O. B. Evans, Assignors to The United Gas Improvement Co., Philadelphia, Pa. U.S. Pat. 1,268,628, June 4, 1918. Date of appl., Oct. 11, 1913.

THE charge of coal or the like in a vertical retort is divided horizontally by layers of porous refractory material, and the gas is delivered through a central perforated vertical pipe. The gas is distilled at a pressure sufficient to force it through the refractory layers and also through the coal layers from the hot circumference to the cooler centre.

—W. F. F.

*Natural-gas substitute [water-gas]; Process of and apparatus for making* —. C. S. Palmer, Pittsburgh, Pa. U.S. Pat. 1,268,763, June 4, 1918. Date of appl., Oct. 5, 1917.

FUEL is charged into a retort formed of a metal in the same periodic group as iron but having a greater atomic weight and not readily oxidised, the thickness of the walls being not greater than 1.5 in. The retort is relatively narrow and is strongly heated externally without an internal air blast. Steam is injected to produce water-gas.

—W. F. F.

*Desulphurisation of gases and other fluids containing sulphuretted hydrogen.* H. W. Hemingway, London. Eng. Pat. 117,387, Nov. 21, 1917. (Appl. No. 17,165 of 1917.)

IN the process for desulphurising coal and other gases, or ammoniacal sulphide liquors, with ferrous carbonate, which is described in Eng. Pat. 12,093 of 1901 (this J., 1902, 906), the ferrous carbonate is recovered for use. Coal gas is passed through scrubbers and the ammoniacal liquor then treated with ferrous carbonate. The liquor is run off to a still or returned to the scrubbers, and the precipitated ferrous sulphide is converted into sulphate by any known method. The ammoniacal liquor is added to the ferrous sulphate and the precipitate converted into ferrous carbonate by absorption of carbon dioxide. The ammoniacal liquor is transferred to a still and the cycle may then be repeated.

—W. F. F.

*Gas-washer.* H. A. Dreffein, Chicago, Ill. U.S. Pat. 1,268,100, June 4, 1918. Date of appl., May 3, 1913.

TWO concentric pipes, tapering slightly towards the bottom, are mounted one within the other on a vertical axis. The two surfaces bounding the annular space are constructed with complementary helical corrugations and the distance between them may be varied by moving the inner pipe vertically by a screwed vertical rod. The outer pipe is

mounted in a casing, and gas admitted to the upper part of the casing must pass downwards through the tortuous passage between the pipes. Means are provided to supply jets of water or steam to the corrugated surfaces.—W. F. F.

*Gas process.* F. P. Peterson, Tulsa, Okla., Assignor to Consolidated Liquid Gas Co., Chicago, Ill. U.S. Pat. 1,266,621, May 21, 1918. Date of appl., Jan. 5, 1914. Renewed Jan. 26, 1918.

CRUDE natural gas is compressed and then passed through a cooler. The condensed liquid is passed back through the cooler and the most volatile constituents removed by allowing them to vaporise and expand at a lower pressure, whereby the gas and liquid are cooled. The process may be used to separate several liquefiable constituents by liquefying them in the inverse order of their volatility and treating each condensed fraction as above. The volatile portions removed are returned to the unliquefied gas for further treatment.

—W. F. F.

*Hydrocarbons; Treatment of liquid — for the transformation thereof.* C. E. and N. C. Cassal, and B. H. Gerrans, London. Eng. Pat. 117,087, June 29, 1916. (Appl. No. 9178 of 1916.)

A LIQUID hydrocarbon, such as petroleum oil, is passed through a horizontal tube filled with fragments of graphite or alumina and heated to 700° C. The resulting product contains benzene and toluene.

—W. F. F.

*Crude oil; Process of and apparatus for preparing — for distillation.* J. O. Jensen, New York. U.S. Pat. 1,268,721, June 4, 1918. Date of appl., Feb. 24, 1917.

A CURRENT of crude oil is heated to above 400° F. (above 200° C.) and the water and low-boiling fractions are removed. The oil then passes into a tank in which it is cooled, by means of cold oil circulating through a number of ducts within the tank, to a temperature above the boiling point of water but below the flashing point of the oil. From the tank the oil is conveyed into the still.

—L. A. C.

*Coke, coal, and other material; Machines for cutting, crushing, or breaking —.* A. G. Sims, Reading. Eng. Pat. 117,585, Mar. 11, 1918. (Appl. No. 4266 of 1918.)

*Gas generators or the like; Automatic removal of ashes from —.* G. H. Bentley and E. G. Appleby, London. Eng. Pat. 117,534, Sept. 20, 1917. (Appl. No. 13,554 of 1917.)

*Gasifying liquid hydrocarbons; Apparatus for —.* A. W. Southey, London. Eng. Pat. 116,970, Aug. 23, 1917. (Appl. No. 12,108 of 1917.)

*Fuel blocks or briquettes; Manufacture of —.* E. R. Sutcliffe, Leigh, Assignor to Pure Coal Briquettes, Ltd., Cardiff. U.S. Pat. 1,267,711, May 23, 1918. Date of appl., Mar. 27, 1915.

SEE Eng. Pat. 20,679 of 1914; this J., 1916, 297.

*Gas-cashbox.* C. S. Chrisman, West Chester, Pa., Assignor to The United Gas Improvement Co., Philadelphia, Pa. U.S. Pat. 1,267,513, May 28, 1918. Date of appl., Jan. 8, 1916.

SEE Eng. Pat. 114,397 of 1917; this J., 1918, 262 A.

*Petroleum or other hydrocarbon distillates or mixtures of volatile liquids; Method and apparatus for continuous fractionation and continuous dephlegmation of —.* J. Hamilton, Glasgow. From H. L. Allan, Rangoon, Burmah. Eng. Pat. 117,277, Apr. 10, 1917. (Appl. No. 5001 of 1917.)

SEE Fr. Pat. 481,134 of 1916; this J., 1917, 127. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 1269 and 16,713 of 1914; this J., 1914, 345; 1915, 1045.)

*Apparatus for separating dust from ores, coal, and the like.* Eng. Pat. 113,780. See 1.

*Manufacture of glycols [from oil-gas].* U.S. Pat. 1,270,759. See XX.

*Method of determining the viscosity of fluids.* Eng. Pat. 117,234. See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Destructive distillation of oil shales.* Morrell and Egloff. See IIA.

*Determination of acetone [in methyl alcohol].* Field. See XX.

### PATENTS.

*Distillation of wood or other similar material. Apparatus for the distillation of wood or other material.* W. J. Mellersh-Jackson, London. From Seaman Waste Wood Chemical Co., New York. Eng. Pats. 116,939 and 116,940, June 30, 1917. (Appl. Nos. 9485 and 9487 of 1917.)

SEE U.S. Pats. 1,236,881 and 1,236,885 of 1917; this J., 1917, 1041.

[*Electric lamps, etc.; Leading-in conductor [for —].* W. L. Van Keuren, North Bergen, N.J., Assignor to General Electric Co. U.S. Pat. 1,268,647, June 4, 1918. Date of appl., Dec. 29, 1913. Renewed Feb. 7, 1918.

SEE Eng. Pat. 4235 of 1914; this J., 1915, 317.

*Still or retort.* U.S. Pat. 1,268,742. See 1.

## III.—TAR AND TAR PRODUCTS.

*Viscous tar, etc.; Determining specific gravity of — at different temperatures.* A. R. Myhill. Gas J., 1918, 143, 254.

A THIN glass tube about 2 ft. in length and  $\frac{3}{8}$  in. internal diameter, sealed at one end, is weighed with a thermometer inside and after removing the thermometer, placed in a vertical position in a glass water-jacket of about 2 in. diameter and 2 ft. in height, containing water at or near the boiling point. A small portion of the tar, previously heated to about 100° C. by immersing the vessel containing it in boiling water, is then poured into the tube, any adhering to the sides being removed. The level of the tar is marked with a file and its temperature taken. The tube is again weighed with the thermometer inside, and the weight of the tar determined. The volume of the tar is then obtained by cleaning out



the tube and running in water from a burette, the water as it is run in being warmed up to the temperature of the tar at the time of making the file mark, by again immersing the tube in the water-jacket. By repeating the readings at two or three different temperatures, say  $20^{\circ}$  to  $30^{\circ}$  apart, a curve can be constructed for converting the volume of the tar at any temperature to that at a standard temperature.—L. A. C.

*Analysis of ammoniacal liquors.* See VII.

#### PATENTS.

*High boiling phenols; Method of producing* —. Sir R. A. Cooper, Berkhamsted, and K. B. Edwards, Glasgow. Eng. Pat. 116,905, Mar. 27, 1917. (Appl. No. 4430 of 1917.)

Oils obtained from shales, brown coal, or lignite are treated with caustic soda solution to obtain "soda tar." The "soda tar" is diluted with water to separate neutral non-phenolic hydrocarbons and then decomposed to recover the phenols.—W. H. C.

*Apparatus for solidifying or concentrating liquid or semi-solid materials on a revolving cylinder.* Eng. Pat. 117,560. See I.

*Treatment of liquid hydrocarbons for the transformation thereof.* Eng. Pat. 117,087. See IIA.

### IV.—COLOURING MATTERS AND DYES.

#### PATENTS.

*Dyestuffs; Manufacture of — and processes of dyeing therewith.* N. Malcolmson and F. W. V. Fitzgerald, London. Eng. Pat. 117,095, Apr. 4, 1917. (Appl. No. 4856 of 1917.)

WOOLLEN yarn or fabric is dyed brown to black by the use of a soluble or insoluble lead compound, such as litharge, in conjunction with a mordant composed of sodium or calcium hydroxide, or mixed with sodium carbonate and calcium oxide. When calcium hydroxide is used, a small quantity of ammonium chloride is added to prevent the formation of insoluble calcium carbonate, and sodium chloride may be added to retard the formation of the colour. A soluble lead salt may be combined with nitrate, sulphate, or other salt of copper or iron, by first rendering the copper or iron salt anhydrous, and then replacing the water of crystallisation by a saturated solution of the lead salt, this being used in conjunction with a mechanical mixture of sodium carbonate and calcium oxide. The mixture containing copper produces a khaki and that containing iron a brown shade on wool. Sulphur may be added to the foregoing mixtures, either mechanically or by dissolving the sulphur in heated paraffin, and adding the lead, copper, or iron salt to the dissolved sulphur, the dyestuff being then suitable for dyeing cotton yarn or fabric a deep greyish black.—B. N.

*Anthracene [vat] dyes and process of making them.* H. Wolff and F. Kafer, Mannheim, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,266,896, May 21, 1918. Date of appl., Dec. 1, 1913.

SEE Eng. Pat. 10,291 of 1913; this J., 1914, 544.

*Sulphur dye.* O. Silberrad, Buckhurst Hill. U.S. Pat. 1,268,803, June 4, 1918. Date of appl., Mar. 1, 1918.

SEE Eng. Pat. 111,738 of 1916; this J., 1918, 52 A.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Lignin; Researches on —. I. Lignosulphonic acids.* M. Hönig and J. Spitzer. Monatsh. Chem., 1918, 39, 1—14.

By the careful evaporation of sulphite-cellulose waste liquor it is possible to obtain a solution of "lignosulphonic acid" which by conversion into the calcium or barium salt and subsequent fractional precipitation from the aqueous solution by alcohol, can be separated into several fractions of different composition. The barium salt obtained in this way can be separated into fractions of the composition  $C_{43}H_{10}O_{38}S_2Ba$ , containing four methoxyl groups,  $C_{40}H_{14}O_{36}S_2Ba$ , containing one methoxyl group, and  $C_{74}H_{14}O_{38}S_2Ba$ , containing three methoxyl groups, the last formula referring to the portion which remains unprecipitated. The salts thus obtained are amorphous brown solids whilst the corresponding free acids are soluble in water, ethyl alcohol, or acetic acid and liberate carbon dioxide from carbonates.—D. F. T.

#### PATENTS.

*Paper; Apparatus for colouring* —. J. Galloway and A. Stratton, Portobello, Scotland. Eng. Pat. 117,428, Apr. 8, 1918. (Appl. No. 5958 of 1918.)

AN overhead tank containing dye liquor is provided with a pipe delivering the dye through a horizontal, perforated, spray pipe, arranged parallel to a calender roll, on to the outer surface of the paper on the roll. A "doctor" or distributing box, having a felt strip engaging the paper, collects the surplus dye, and the latter is passed to a receiver from which it is pumped to the overhead tank.—B. N.

*Porous fabrics [paper-board]; Saturation of — [with asphalt].* E. J. Cady, Chicago, Ill. U.S. Pat. 1,268,430, June 4, 1918. Date of appl., Apr. 2, 1917.

ONE surface of a travelling length of paper-board is subjected to the weight of a body of asphalt solution, while the opposite surface is maintained under reduced air pressure and out of contact with the solution.—W. E. F. P.

*Cellulose film; Composite* —. J. E. Brandenberger, Thaon-les-Vosges, Assignor to Soc. La Cellophane, Paris. U.S. Pat. 1,266,766, May 21, 1918. Date of appl., May 26, 1914.

SEE Fr. Pat. 458,638 of 1913; this J., 1913, 1063. The outer films may be of nitrocellulose, and small particles of solid material may be incorporated with the plastic material in the intermediate layer.

*Paper machines; Fourdrinier* —. A. J. Davies, Kenogami, Quebec. Eng. Pat. 107,579, May 25, 1917. (Appl. No. 7569 of 1917.)

SEE U.S. Pat. 1,231,717 of 1917; this J., 1917, 960.

*Paper; Coated* —. H. R. Rafsky, Lawrence, Mass., U.S.A. Eng. Pat. 117,414, Feb. 23, 1918. (Appl. No. 3235 of 1918.)

SEE U.S. Pat. 1,260,448 of 1918; this J., 1918, 297 A.

*Paper; Apparatus for making* — G. Schenck, jun., Assignor to Great Northern Paper Co., Millinocket, Me. U.S. Pat. 1,267,701, May 28, 1918. Date of appl., Mar. 18, 1915.

SEE Eng. Pat. 8838 of 1915; this J., 1915, 1007.

*Pulp; Apparatus for washing or similarly treating* — S. Milne, Edinburgh. U.S. Pat. 1,267,683, May 28, 1918. Date of appl., Dec. 26, 1916.

SEE Eng. Pat. 107,410 of 1916; this J., 1917, 959.

*Process of producing 1,2,4-methylhydroxyisopropylbenzene [carvacrol]*. U.S. Pat. 1,265,800. See XX.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Dyeing textile fabrics and apparatus therefor*. T. Glaister and J. K. Hoyle, Bolton, Lancs. Eng. Pat. 117,039, Jan. 10, 1918. (Appl. No. 566 of 1918.)

THE dyeing machine is provided with a pair of driven pressure bowls or rollers, the lower one forming one side of the dye-box (through which the fabric is led after being saturated by submersion in the dye liquor. The latter is sprayed through a perforated pipe in the first instance upon the surface of the bottom bowl, and then transferred in a film to the fabric as it passes through the pressure nip of the pair of driven bowls, thus ensuring thorough impregnation and even dyeing.

—B. N.

*Cleansing of linen, woollen, cotton, and other goods*. F. L. Bartelt, Bristol. U.S. Pat. 1,267,737, May 28, 1918. Date of appl., Dec. 10, 1914.

SEE Eng. Pat. 28,500 of 1913; this J., 1915, 134. A soluble zinc salt is incorporated with the mixture of sodium carbonate and glycerin.

*Cotton material with white-effect threads and process of making it*. H. Walther, Leverkusen, Germany, Assignor to Synthetic Patents Co., New York. U.S. Pat. 1,270,348, June 25, 1918. Date of appl., Nov. 5, 1914.

SEE Eng. Pat. 2396 of 1915; this J., 1915, 1205.

*Manufacture of dyestuffs and processes of dyeing therewith*. Eng. Pat. 117,095. See IV.

*Apparatus for colouring paper*. Eng. Pat. 117,428. See V.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Thiosulphuric, sulphurous, trithionic, and sulphuric acids; Estimation of mixtures of* — O. Billeter and B. Wavre. *Helvetica Chim. Acta*, 1918, 1, 174—180.

THE solution containing the acids is warmed with bromine water, whereby the acids are converted entirely into sulphuric acid, which is estimated as barium sulphate. The sum of the sulphite and thiosulphate is determined by titration with standard iodine solution. If no trithionate is present the sulphite may then be estimated by titration with standard sodium disulphide, when the change

$\text{Na}_2\text{S}_3 + \text{Na}_2\text{SO}_3 = \text{Na}_2\text{S} + \text{Na}_2\text{S}_2\text{O}_3$  takes place. This estimation is carried out in the presence of ammonium chloride at the boiling point, the end of the reaction being indicated by the persistence of the yellow colour of the standard solution. If trithionate is present alone, the reaction is  $\text{Na}_2\text{S} + \text{Na}_2\text{S}_3\text{O}_6 = 2\text{Na}_2\text{S}_2\text{O}_3$ ; the estimation is carried out by adding sodium sulphide to the solution and boiling for a few minutes, an excess of sodium acetate and some acetic acid is then added and the solution again boiled to decompose the excess sulphide; after cooling, the thiosulphate produced is titrated with standard iodine. Should both sulphite and trithionate be present, the sodium disulphide titration is carried out as described above, the end point being indicated both by the yellow colour and the formation of a turbidity due to sulphur, and then the trithionate titration proceeded with as above described. The method gives good results.—J. F. S.

*Hydrocyanic acid; Decomposition of solutions of* — W. Lewcock. *Pharm. J.*, 1918, 101, 50—51.

ADDITION of sodium carbonate to a 30% solution of sodium cyanide causes, in time, the formation of a brown deposit, whereas caustic soda has not the same action. The brown decomposition product appears to be formed by the action of the alkali on free hydrocyanic acid produced by the hydrolysis of the cyanide, whilst the presence of caustic soda suppresses the hydrolysis. The decomposition of dilute solutions of hydrocyanic acid on storage may be attributed to the action of small quantities of basic substances principally derived from the glass vessels, possibly also from the use of hard water in the preparation of the solutions. Addition of bases or salts with alkaline reaction greatly accelerated the decomposition of hydrocyanic acid solutions. Substances such as alcohol, glycerin, and mannitol did not prevent the rapid decomposition of the hydrocyanic acid, but sulphuric acid, tartaric acid, and even carbon dioxide had a protective action depending on the concentration. An 8% solution of hydrocyanic acid in distilled water showed a yellow coloration in 2 days but in presence of 1% of sulphuric acid, calculated on the weight of hydrocyanic acid, the solution remained colourless for 23 days. It is calculated that the addition of 10% of sulphuric acid on the weight of hydrocyanic acid would preserve the solution indefinitely. (See also *J. Chem. Soc.*, 1918, i., 382.)—J. P. B.

*Acetic acid; Determination of* — by distillation with phosphoric acid. W. P. Munn. *J. Ind. Eng. Chem.*, 1918, 10, 550—552.

FIVE grms. of the sample is placed in a round-bottomed flask attached to a condenser by means of a three-bulb still-head which is made in one piece with the condenser tube. The receiver consists of a conical suction flask containing a known excess of standardised barium hydroxide solution. Dilute phosphoric acid solution (40 c.c. of 85% phosphoric acid solution and 20 c.c. of water) is added to the sample in the distillation flask, a gentle current of air is drawn through the apparatus, the inlet tube for the air being provided with a soda-lime tube, and the contents of the flask are boiled. Dilute phosphoric acid is then admitted to the flask through a tapped funnel at the same rate at which the liquid distils. At the end of the distillation, which usually requires about 90 mins., the contents of the receiver are titrated with N/10 hydrochloric acid, using phenolphthalein as indicator. N/1 hydrochloric acid is then added to dissolve any barium carbonate present, and the excess of acid is titrated



with N/1 sodium hydroxide solution using methyl orange as an external indicator. The latter titration gives the correction to be applied for carbonate. —W. P. S.

*Nitric oxide and oxygen; Velocity of reaction between* —. M. Bodenstein. Deuts. Bunsen-Ges., Apr., 1918. Z. Elektrochem., 1918, 24, 183—201.

OVER the temperature range  $0^{\circ}$ — $90^{\circ}$  C. the reaction between nitric oxide and oxygen proceeds according to the equation:  $dx/dt = kC_{O_2}C_{2NO}$ , that is the reaction is of the third order. The velocity falls with increase in temperature and is not affected by the presence of nitrogen peroxide or water vapour. Sulphur dioxide is also without influence on the reaction, and hence, conversely, the oxidation of sulphur dioxide cannot be catalysed by oxides of nitrogen, and in the sulphuric acid chamber nitrogen peroxide acts not in the gas phase but only after condensation to  $HSNO_2$ . The controversy between Lunge and Raschig (this J., 1904, 934, 1213; 1905, 131, 923; 1906, 533; 1907, 965) is considered and Lunge's view of the mechanism of the oxidation of nitric oxide is confirmed. Raschig's experimental methods are shown to be faulty for mixtures in which less than 50% of the nitric oxide has been converted into nitrogen peroxide. In the case of such mixtures the nitrogen peroxide and an equivalent quantity of nitric oxide would dissolve as  $N_2O_3$  in the absorption medium, the equilibrium  $NO + NO_2 \rightleftharpoons N_2O_3$  being attained practically instantaneously, and any remaining nitric oxide would be evolved. Hence in such circumstances the fact that the N:O ratio in the products absorbed is 1:1, as found by Raschig, affords no support for his view that nitric oxide is oxidised rapidly to  $N_2O_3$ , and the latter then slowly oxidised to  $NO_2$ . (See also J. Chem. Soc., Sept., 1918.)—J. F. S.

*Nitric oxide; Velocity of oxidation of* —. E. Briner and E. Fridöri. Helvetica Chim. Acta, 1918, 1, 181—185.

THE velocity of oxidation of nitric oxide in mixtures of air and nitric oxide such as are obtained in the manufacture of nitric acid from the atmosphere, takes place with a measurable velocity and conforms to the equation for a reaction of the third order. Lowering of the temperature by  $10^{\circ}$  over the interval  $0^{\circ}$ — $60^{\circ}$  C. increases the velocity by 10–20%.—J. F. S.

*Gas; Ionisation of* — during chemical reaction. [Interaction of nitric oxide with oxygen and with chlorine.] A. Pinkus. Helvetica Chim. Acta, 1918, 1, 141—145.

THERE is no ionisation of the reacting gases when oxygen and nitric oxide are allowed to react under a pressure of  $\frac{1}{4}$ — $\frac{1}{2}$  atm., nor is there any ionisation when chlorine reacts with nitric oxide, provided that the chlorine is not in excess. If, however, the chlorine is in excess, there is a marked ionisation. (See also J. Chem. Soc., Sept., 1918.)—J. F. S.

*Ammonia and hydrochloric acid; Estimation of* — by weighing as ammonium chloride. A. Villiers. Bull. Soc. Chim., 1918, 23, 306—308.

THE author reaffirms the accuracy of his method (compare Bull. Soc. Chim., 1900, 23, 253) for the estimation of ammonia or hydrochloric acid by weighing as ammonium chloride. Ammonium chloride did not lose weight when heated for 3 days and 3 nights at  $105^{\circ}$  C.; the small, gradually diminishing losses which have been observed by other workers were probably due to the slow elimination of the last traces of moisture.—W. G.

*Ammoniacal liquors; Analysis of* —. 54th Annual Report on Alkali Works (Year 1917), by the Chief Inspector, 22—31.

THE difficulty in the complete analysis of ammoniacal liquors is again noted and revision of certain methods (this J., 1910, 943) is necessary. In the estimation of thiocyanate the use of lead carbonate to remove sulphide leads to unreliable results unless precautions are taken to exclude air during the operation, and lead carbonate is regarded as an unsuitable agent, particularly if cyanides be present. Boiling is preferred with minimum exposure of the solution to air. 60–70 c.c. of distilled water is boiled to expel dissolved oxygen and 50 c.c. of liquor run into the boiling water, the boiling being continued to expel ammonium cyanide and sulphide. 10 c.c. of a saturated solution of ammonium iron alum is added, and the solution separated by decantation and filtration. Cuprous thiocyanate is then precipitated by adding sodium bisulphite and an excess of copper sulphate solution, and after standing for 30 mins. the solution is filtered and the precipitate washed, digested with 25 c.c. of a 4% solution of sodium hydroxide at  $30^{\circ}$ — $40^{\circ}$  C., acidified with nitric acid, and titrated with N/10 silver nitrate solution, using a solution of iron alum as indicator. A sharper end-point is obtained by adding an excess of silver nitrate and titrating back with ammonium thiocyanate. Cyanide in liquors of high strength is most conveniently determined by the "polysulphide" method, in which cyanide is converted into thiocyanate by the action of ammonium polysulphide and titrated with silver nitrate. The pre-existing thiocyanate and chloride in the liquor are determined by a blank experiment. Attempts to estimate polysulphide in liquors by a simple method have only met with partial success. The liquor is mixed with an excess of ammonium cyanide solution of known strength in a stoppered vessel and the thiocyanate formed is determined. An alternative method is to determine the excess of cyanide by distillation. Both these methods are, however, subject to interference from dissolved oxygen.—C. A. K.

*Bicarbonates and carbonates, or alkali carbonates and hydroxides; Method of analysing mixtures of* —. J. Clarens. Ann. Chim. Analyt., 1918, 23, 148—152.

THE method depends on the determination of the quantity of carbon dioxide liberated when the carbonate mixture is titrated, the operation being carried out in a flask provided with a mercury manometer.—W. P. S.

*Lime and magnesia; Latent heats of fusion of* —. E. W. Washburn. Trans. Amer. Ceram. Soc., 1917, 19, 195—200.

THE author has calculated the latent heat of fusion of lime and magnesia from the freezing point-solubility formula for solutions. The value for lime is 28,000 ( $\pm 20\%$ ) calories per gram-mol. or 490 calories per gram., and that of magnesia 28,000 ( $\pm 15\%$ ) calories per gram-mol. or 700 calories per gram. Similar values were obtained for various silicate minerals. Hence, the low fusibility of these materials is due, in considerable degree, to their latent heat of fusion.—A. B. S.

*Alumina; Preparation of* — in the aluminium industry with special reference to rotary kilns. W. von Escher. Chem.-Zeit., 1918, 42, 333—355.

GREAT difficulty is experienced in obtaining calcined alumina suitable for the production of aluminium and free from such impurities as iron, silica, and titanium oxide. Most of the methods used for purifying the alumina are based

on the solution of the aluminium as aluminate, the other constituents of the ore remaining undissolved. The ore is washed to remove sand, crushed to the size of hazel nuts, and calcined at  $450^{\circ}\text{C}$ . Calcining at a much higher temperature reduces the yield of alumina and makes the subsequent grinding more difficult. A rotary kiln is used, connected with a rotary cooler. The calcined material is ground until a sample leaves only 15% residue on a 180-mesh sieve; the finer the material the greater is the yield of alumina. The ground material is stored in bins, at least a week's supply being kept in stock. It may be treated by any of the five following methods. *Bayer's wet process* (Ger. Pats. 43,977 and 65,604; see also this J., 1888, 625, 748; 1893, 263, 353, 925). The ground calcined material is thoroughly mixed with 40–45% soda-lye in the proportion of 1 mol.  $\text{Al}_2\text{O}_3$  to 1.8 mols.  $\text{Na}_2\text{O}$ . The formation of sodium aluminosilicates is prevented by the addition of quicklime to the crude ore before grinding it. The mixture of calcined ore and lye is heated under pressure for 5 hrs. in a wrought iron autoclave fitted with a mechanical stirrer to convert the alumina into soluble sodium aluminate, a yield of 80% being obtained. The "red lye" thus produced is run into large tanks fitted with stirring mechanism and means for indirect heating, in which it is diluted with wash-water to  $25^{\circ}\text{B}$ . (sp. gr. 1.21). It is then filtered and the residue washed until free from alkali. The use of great pressure in the filter-presses causes the finer particles to pass through. The solution which runs from the filters is re-filtered in separate filters. The residue may be used for purifying coal-gas or as iron-ore. The filtrate—which contains 90–160 grms.  $\text{Al}_2\text{O}_3$  and 100–110 grms.  $\text{Na}_2\text{O}$  per litre—is pumped into tanks 20–30 ft. high and 10–20 ft. diameter in which it is subjected to constant agitation for 5–7 days to precipitate aluminium hydroxide; the addition of aluminium hydroxide facilitates the precipitation. The well-stirred liquid should contain up to 6 mols.  $\text{Na}_2\text{O}$  to each 1 mol.  $\text{Al}_2\text{O}_3$ , i.e. about 30 grms.  $\text{Al}_2\text{O}_3$  remains in each litre of solution. The aluminium hydroxide is filter-pressed, the filtrate evaporated to one-third of its volume and used for treating further quantities of material in the autoclave. The wash-water, of sp. gr. exceeding 1.075 ( $10^{\circ}\text{B}$ ), is also evaporated, but that of lower density is used for diluting the "red lye." The press-cakes, which consist of friable granular aluminium hydroxide showing a loss on ignition of 45–50%, are afterwards calcined. *Dry process (treatment with soda)* (Ger. Pats. 93, 1650, 19,784, 25,777, and 70,175). This process is not suitable for silicious bauxites unless they have previously been washed. A high iron-content is less injurious than in Bayer's process. The ground, calcined bauxite is mixed with sodium carbonate (1.2–1.5 mols.  $\text{Na}_2\text{O}$  for each 1 mol.  $\text{Al}_2\text{O}_3$ ) and a little coal and is then passed through a damping mixer into a rotary kiln in which it is heated until the mass slinters, and all the carbon dioxide has been expelled. The sintered mass is extracted as rapidly as possible with water above  $80^{\circ}\text{C}$ ., about 2.5 cub. m. of liquid being used for each 1000 kilos. of product. Caustic soda is added to bring the  $\text{Al}_2\text{O}_3$ : $\text{Na}_2\text{O}$  ratio to 1:1.8; if the extraction is unduly prolonged aluminium hydroxide will be precipitated and lost. The product is filtered, and the filtrate (sp. gr. 1.26–1.32,  $30^{\circ}$ – $35^{\circ}\text{B}$ ), at a temperature of  $80^{\circ}\text{C}$ ., is run into tall cylindrical "carbonators" in which the aluminium hydroxide is precipitated by carbon dioxide. The wash-water is used for extracting a further quantity of slintered material. In order to obtain the aluminium hydroxide in a granular form, attention must be paid to the concentration of the solutions. The aluminium hydroxide is calcined. The filtrate and wash-water are evaporated *in vacuo* in order to recover the sodium carbonate. *Peniakoff's pro-*

*cess* (Ger. Pats. 80,063, 93,952, and 247,762; see also this J., 1891, 947; 1896, 808; 1910, 1451). The ground, calcined bauxite is mixed with sodium sulphate and coal, moistened, and heated in a rotary kiln to  $1400^{\circ}\text{C}$ . or until the mass slinters. Sulphur dioxide and carbon dioxide are expelled. The proportions of the various ingredients have an important influence on the yield. The formation of sulphides is prevented by passing a current of air through the kiln. The sintered mass is treated as in the soda process; the gases from the kiln are treated by Hargreaves' process in order to convert the sulphur dioxide into sodium sulphate. *Process of Chem. Fabr. Griesheim-Elektron* (Ger. Pat. 232,563; this J., 1911, 621). The clay or bauxite is boiled with sulphuric acid and the solution is allowed to crystallise. Aluminium sulphate separates out, whilst iron sulphate remains in solution. Any traces of iron in the crystals may be separated by dissolving them in hot sulphuric acid and recrystallising. *Serpuk's process* (Patents owned by Soc. Gén. des Nitrures; see this J., 1906–1917). A mixture of bauxite and coal is heated in a current of producer gas in a rotary kiln, with an electrically heated zone, to between  $1400^{\circ}$  and  $1850^{\circ}\text{C}$ . until the alkalis, alkaline-earths, and silica compounds are volatilised and aluminium nitride is formed. On treating the nitride with water, ammonia is formed and recovered as sulphate and this important by-product reduces the cost of the alumina. This process was not used commercially before the war and little is known about it. In comparing the foregoing processes, attention must be paid to the composition of the raw bauxite. A material rich in iron but low in silica is preferably treated by a dry process, but silicious bauxites should be purified by Bayer's process. Very hard bauxites, such as those in Hungary, are best treated by heating with soda to a slintering temperature. The relative costs of using caustic soda, sodium carbonate, and sodium sulphate must also be considered. Bayer's process has been the most extensively used and appears to require less power and labour than the others.—A. B. S.

*Arsenious oxide; Preparation and testing of pure* — R. M. Chapin. J. Ind. Eng. Chem., 1918, 10, 522–525.

To 2 litres of boiling water is added 150 grms. of powdered commercial arsenious oxide mixed with water, the whole is boiled for about 90 mins., or until the volume has been reduced to 1600 c.c., then allowed to settle, and the clear liquid filtered. If the liquid is not clear, it is heated to boiling with 2 grms. of calcium oxide, and then filtered. The filtrate is concentrated until 4 or 5 grms. of arsenious oxide has separated, and then filtered rapidly while boiling. A portion (equal to 1 gm. of dry substance) of the precipitate is tested for antimony by dissolving it in 10 c.c. of a mixture of equal vols. of ammonia (sp. gr. 0.9) and water, adding 10 c.c. of hydrogen sulphide solution, heating to boiling, and allowing to cool; antimony, if present, separates out as sulphide. Should the test for antimony be positive, successive crops of crystals must be tested until a negative test results: the antimony is practically all adsorbed by solid arsenious oxide. The antimony-free solution is then mixed with rather more than its own volume of concentrated hydrochloric acid, concentrated to saturation, filtered hot, and the filtrate cooled. The crystals formed are collected and sublimed. —W. P. S.

*Ceric oxychloride, produced by the electrolysis of cerous chloride.* H. Arnold. Z. Elektrochem., 1918, 24, 137–138.

A dark brown amorphous deposit is produced on the cooler parts of the anode in the electrolysis of



fused cerous chloride. This powder is strongly hygroscopic and somewhat like ferric chloride, and consists of a mixture of ammonium chloride, ceric oxychloride ( $\text{CeOCl}_2$ ), and water. Attempts to obtain the pure oxychloride failed. The oxychloride is only produced so long as the melted cerous chloride contains water. (See also J. Chem. Soc., Sept., 1918.)—J. F. S.

*Nitrogen chloride: Convenient method of preparation of* —. H. Rai. Chem. News, 1918, 117, 253.

NITROGEN chloride can readily be prepared by the action of a solution of bleaching powder on a saturated solution of ammonium chloride, at about  $0^\circ\text{C}$ ., without the use of hydrochloric acid or a solvent such as benzene. The apparatus must be absolutely free from grease and direct sunlight should be entirely excluded. (See also J. Chem. Soc., Sept., 1918.)—C. S.

*Evaporator for acid liquids.* Hart. See I.

"Direct process" of making ammonium sulphate in gasworks. See IIA.

[Aluminium and chromium] carbides. Ruff. See X.

*Use of nitre-cake in the manufacture of superphosphate.* See XVI.

*Composition of lime-sulphur solutions.* Winter. See XIXb.

*Estimation of phosphoric acid as ammonium phosphomolybdate.* Villiers. See XXIII.

*The bisulphate method of determining radium.* Barker. See XXIII.

#### PATENTS.

*Sulphuric acid: Method of and apparatus for making* —. L. A. Thiele, Columbus, Ohio. U.S. Pat. 1,267,012. May 21, 1918. Date of appl., Feb. 12, 1917.

A GLOVER tower is connected with an acid chamber by a number of pipes which enter the chamber tangentially and which are of different lengths and sectional areas, the smallest pipe having the greatest length and the largest pipe having the shortest length. A number of outlet conduits are arranged concentrically in the bottom of the chamber "on a diameter approximately half the diameter of the chamber."—L. A. C.

*Catalytic substances; Preparation of* — from natural ores containing iron oxide which contains water of hydration. H. Fritzweiler, Stolberg, B. C. Stuer, and Chem. Fabr. Rhenania, Aachen, Germany. Eng. Pat. 109,056, Aug. 22, 1917. (Appl. No. 12,072 of 1917.) Under Int. Conv., July 27, 1916.

THE crude ore, e.g., bog iron ore, bauxite, or the like, is finely powdered and compressed, with or without preliminary heating and without any binding substance. Disintegration of the catalyst in use is thereby prevented.—W. F. F.

*Magnesium from compounds and mixtures of magnesium and calcium carbonate: Separation of* —. H. W. C. Annable, Egham, Surrey. Eng. Pat. 117,483, July 13, 1917. (Appl. No. 10,202 of 1917.)

A MIXTURE of calcium and magnesium carbonates suspended in water is boiled in an autoclave under pressure (preferably about 4 atmos.) with just sufficient calcium chloride (or barium or strontium chloride) to convert the magnesium carbonate into magnesium chloride. The product is filtered and the magnesium precipitated from the filtrate by the addition of milk of lime, the calcium chloride produced being used for the treatment of fresh material. By passing carbon dioxide through a suspension of the precipitated magnesium oxide in water, a hydrated magnesium carbonate may be obtained, a different compound being produced according to whether the gas is passed into a cold or boiling solution. In treating mixtures in which the carbonates occur together as complex molecules (e.g., dolomite), it is necessary first to calcine the material in a current of air or steam, and precipitate the carbonates by passing carbon dioxide through a suspension of the product in water.

—L. A. C.

*Carbon dioxide and solid sodium sulphite; Process of making* —. H. Howard, Brookline, Mass., Assignor to General Chemical Co. U.S. Pat. 1,266,595, May 21, 1918. Date of appl., Nov. 22, 1915.

SODIUM bisulphite solution is introduced beneath the surface of a column of liquid containing sodium carbonate in suspension, the amount of liquid being insufficient to dissolve the sodium carbonate. Normal sodium sulphite and carbon dioxide are formed, the carbon dioxide being purified from sulphur dioxide in its passage through the liquor. (See also U.S. Pat. 1,164,649; this J., 1916, 254.)

—L. A. C.

*Lead arsenate; Manufacture of* —. I. P. Lihme, Lakewood, Assignor to The Grasselli Chemical Co., Cleveland, Ohio. U.S. Pat. 1,267,428, May 28, 1918. Date of appl., July 25, 1916.

LEAD oxide and arsenic acid in the proportion of their combining weights are caused to react in the presence of an amount of an acid-halogen derivative of silicon (hydrofluosilicic acid) less than 1% of the arsenic acid.—L. A. C.

*Aluminium chloride; Process of making* —. G. H. King and G. I. Roberts, Port Arthur, Tex., Assignors to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,268,015, May 28, 1918. Date of appl., June 15, 1917.

A PREHEATED suspension of finely divided alumina and carbon in chlorine is passed through a heated reaction chamber, and the aluminium chloride produced is condensed.—W. E. F. P.

*Potassium sulphate and alumina; Method of recovering* — from alunite. H. F. Chappell, Assignor to Mineral Products Corporation, New York. U.S. Pat. 1,268,433, June 4, 1918. Date of appl., July 23, 1913.

HOT, calcined alunite is brought into contact with an aqueous solvent, and the undissolved alumina is separated from the hot solution of potassium sulphate produced. The latter is then cooled to effect deposition of potassium sulphate, and the mother liquor is used as solvent for a further quantity of calcined alunite.—W. E. F. P.

*Potassium sulphate; Process of treating potassium-bearing silicates to obtain —.* G. Sterling, Salt Lake City, Utah. U.S. Pat. 1,268,508, June 4, 1918. Date of appl., Sept. 17, 1917.

A FINELY-DIVIDED mixture of the silicate (3 parts), limestone (2 parts), and sodium sulphate (1 part) is heated to just below the temperature of vitrification, and stirred meanwhile, whereby potassium sulphate is produced and sublimed.—W. E. F. P.

*Potassium and phosphorus compounds; Process of producing —.* E. W. Hinslup, Bronxville, N.Y., and B. A. Peacock, Philadelphia, Pa. U.S. Pat. 1,270,515, June 25, 1918. Date of appl., Oct. 29, 1917.

A MIXTURE of finely-divided potassium-bearing silicate and phosphate rocks is treated with sulphuric acid in a closed, heat-insulated vessel until the mixture attains a temperature above 150° C. and a substantial portion of the silicate is decomposed. The mass is then treated with water and the liquid neutralised, to precipitate alumina and produce a neutral solution of the desired compounds.—W. E. F. P.

*Alumina; Treatment of insoluble —.* H. F. Chappell, Assignor to Mineral Products Corporation, New York. U.S. Pat. 1,270,266, June 25, 1918. Date of appl., Mar. 1, 1916.

THE alkali-insoluble alumina of calcined alunite is converted into water-soluble compounds by mixing 100 parts with 210 parts of sodium carbonate and 110 parts of calcium oxide, and calcining the mixture at about 900° C.—W. E. F. P.

*Brine [containing calcium and magnesium chlorides]; Method of treating —.* E. O. Barstow, Assignor to The Dow Chemical Co., Midland, Mich. U.S. Pat. 1,270,589, June 25, 1918. Date of appl., May 12, 1917.

BRINE containing both calcium and magnesium chlorides is treated with excess of lime, and the precipitate, consisting of magnesium and calcium hydroxides, is separated, leaving substantially pure calcium chloride solution. The precipitate, in the form of a sludge, is then caused to react with a further quantity of the brine, whereby the calcium hydroxide is converted into magnesium hydroxide. The resultant liquor is then treated as before.—L. A. C.

*Air or other gaseous mixtures; Process for separating the constituents of —.* Ges. für Linde's Eismaschinen A.-G., Hölriegelskreuth, Germany. Eng. Pat. 109,789, July 12, 1917. (Appl. No. 10,114 of 1917.) Under Int. Conv., Sept. 18, 1916. Addition to Eng. Pat. 21,735, Jan. 29, 1914.

AS an improvement of the process described in the chief patent (this J., 1915, 871) for separating the constituents of air or other gaseous mixtures containing, besides argon, two other gases which condense with difficulty, one of which boils more easily and the other less easily than argon, the rectifying column is cooled to about the boiling point of the more easily boiling liquefied gas under the pressure, above atmospheric pressure, prevailing in the column. For example, in the case of air, liquid nitrogen may be used as the cooling agent, being caused to evaporate under the pressure in the cooler. Liquid air or oxygen may also be used as the cooling agent, in which case, either the pressure is lowered by suction to attain the necessary temperature, or the pressure in the rectifying column is raised to that at which pure nitrogen liquefies at the temperature prevailing in the cooler.—L. A. C.

*Nitrogen; Production of — from the air.* C. Andreucci, Rome. Eng. Pat. 117,333, Aug. 21, 1917. (Appl. No. 12,179 of 1917.)

ATMOSPHERIC air and hydrogen in theoretical proportions are exploded in an internal combustion engine; the exhaust gases consist of nitrogen and water vapour, from which the nitrogen is recovered.—C. A. K.

*Bromine; Process of producing —.* R. Datta, Calcutta. U.S. Pat. 1,267,638, May 28, 1918. Date of appl., Aug. 17, 1916.

THE process consists in oxidising nitrosyl bromides and separating bromine from the products.—W. E. F. P.

*Oxides of nitrogen; Process of producing — and apparatus therefor.* C. Rossi, Legnano, Italy. U.S. Pat. 1,266,717, May 21, 1918. Date of appl., Oct. 22, 1913.

SEE Fr. Pat. 463,821 of 1913; this J., 1914, 421.

*Nitrogen compounds; Manufacture of metal —.* R. Grenlich, Ludwigshafen, Germany, and H. Lickfett, Rjukan, Norway, Assignors to Gebr. Giulini, Ludwigshafen. U.S. Pat. 1,268,240, June 4, 1918. Date of appl., Nov. 28, 1913.

SEE Fr. Pat. 463,232 of 1913; this J., 1914, 421.

*Alkali perborates; Electrolytic process of making —.* O. Liebknecht, Frankfurt, Germany, Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,268,369, June 4, 1918. Date of appl., Oct. 23, 1915.

SEE Eng. Pat. 14,292 of 1915; this J., 1916, 839.

*Desulphurisation of gases and other fluids containing sulphuretted hydrogen.* Eng. Pat. 117,387. See IIa.

*Production of ammonium chloride during the working of blast furnaces.* Eng. Pat. 102,146. See X.

## VIII.—GLASS; CERAMICS.

*Glass-tank furnace operation.* R. L. Frink, Trans. Amer. Ceram. Soc., 1917, 49, 331—338.

THE production of seedy glass of low quality and erratic colour was traced to alterations to the checkerwork and to the partial collapse of a side flue, which had diminished the flow of air to the furnace. On re-setting the checkers and increasing the height of the chimney, good glass was produced. When the damper is lowered to "cut" the steam, the furnace temperature is also lowered, stiff, coloured, and seedy glass being produced. The correct procedure is to open both chimney- and air-dampers, and, if necessary, to supply more gas, so as to increase the temperature and so effect combination between the various constituents of the glass-batch. If the oil or gas used as fuel is allowed to impinge on the batch, glass, or port blocks before combustion is complete, carbon will be deposited and absorbed by the molten glass, with consequent discoloration. This can be avoided by mixing the air and gas thoroughly, previous to their combustion. It is important to maintain the checkerwork at a sufficiently high temperature, with proper regulation of the burner, pressure, air, and dampers; the top checker may be at 2200° F. (1200° C.), the bottom one at 1500° F. (815° C.), the flue at 900° F. (480° C.), and the chimney at 700°—800° F. (370°—430° C.).—A. B. S.



*Glass; Formation and removal of bubbles in molten* —. R. M. Howe. Trans. Amer. Ceram. Soc., 1917, 19, 201–208.

THE removal of bubbles from glass depends on the frictional resistance of the glass and on the size of the bubbles, in accordance with the equation:  $x = Ar^2/K$  where  $x$  = the rate of removal,  $A$  is a constant,  $r$  = the radius of the bubbles, and  $K$  is a constant. As  $K$  can never be made negligibly small, it is impossible to remove all the small bubbles by raising the temperature of the glass. The most effective means of removing small bubbles is to introduce larger ones into the glass, *e.g.*, by dropping a potato into the molten glass; the small bubbles merge into the large ones and so are carried to the surface. The large bubbles are under a lower pressure than the smaller ones, so that the latter penetrate readily into them.—A. B. S.

*Kaolins; Use of sulphuric acid in the sedimentation of* —. H. J. Schurecht. Trans. Amer. Ceram. Soc., 1917, 19, 130–145.

KAOLINS are purified by agitating them with a very dilute solution of sodium hydroxide, decanting the fluid with the clay in suspension, and adding just enough sulphuric acid to neutralise the alkali previously added and to facilitate the deposition of the kaolin. The treatment sometimes increases the plasticity of the kaolins, but this may be prevented by adding less acid. The formation of a black scum or discoloration is due to the use of an excess of sulphuric acid. The most suitable amount of acid must be ascertained by trial, as it differs with each clay. Usually it is desirable to employ rather less acid than is equivalent to the soda used.

—A. B. S.

*Clays; Relation of salt to clay in the purification of* —. H. G. Schurecht. Trans. Amer. Ceram. Soc., 1917, 19, 460–478.

CLAYS may be purified by mixing them with very dilute solutions of certain electrolytes which cause the clay particles to remain in suspension whilst the coarser impurities settle out. If no electrolyte is added, some of the clay is carried down by the coarser particles. The lower the viscosity the greater the purification effected, but in the absence of electrolytes the proportion of clay kept in suspension is very low. To maintain a minimum viscosity in clay slips, it is more important to keep constant the ratio of electrolyte to clay than that of electrolyte to water. The author confirms Bleininger's statement (this J., 1915, 906) that sodium silicate and sodium hydroxide are more effective than sodium carbonate; they also have a larger range of action. Viscosity tests show that 50–58% more clay can be separated from the crude material when the above-mentioned electrolytes are added than when plain water is used.—A. B. S.

*Clays; Burning properties of some American refractory* —. H. G. Schurecht. Trans. Amer. Ceram. Soc., 1917, 19, 248–253.

TEST-PIECES of four clays were fired at the rate of 20° C. per hour and the pieces drawn from the kiln at intervals between 1210° C. and 1410° C. The porosities and volume-shrinkages were determined; also the temperature at which the burned pieces (each 7 in. × 1 in. × 1 in. and supported on fireclay knife edges 6 in. apart) broke under a load of 3 lb. applied transversely. It was found that the load-carrying capacity of refractory clays can be increased by burning them to higher temperatures, though vitrifiable clays are not improved so much as those of more open texture. The shrinkage in use is reduced by burning the ware at as high a temperature as possible.—A. B. S.

*Refractory clays; Relation between the fusion point and composition of* —. R. J. Montgomery and C. E. Fulton. Trans. Amer. Ceram. Soc., 1917, 19, 303–311.

ON plotting the ratio of alumina to alumina plus silica of 57 clays as abscissae and their softening points as ordinates, the authors found that the points all lie below the eutectic curve found by Sosman (this J., 1917, 136) for mixtures of pure alumina and silica. The vertical distance between such points and the curve depends primarily on the percentage of fluxes present in the clays. The maximum activity of the fluxes present is shown in a clay containing about 14% of alumina (based on alumina plus silica = 100). With high silica, beyond the eutectic point, the activity of the fluxes diminishes rapidly.—A. B. S.

*Tiles; Effect of variation of pressure in forming dust-pressed* —. F. K. Pence. Trans. Amer. Ceram. Soc., 1917, 19, 94–101.

THE chief defects in dust-pressed tiles are due to unequal distribution of the pressure, to insufficient pressure, or, rarely, to excessive pressure. Tiles with high porosity and shrinkage have not been pressed sufficiently. Tiles which have been subjected to excessive pressure have a low porosity and low shrinkage. The use of excessive pressure in manufacture may cause vitrified tiles to blister or swell and glazed tiles to craze. The effect of pressure on the shrinkage of the tiles is important. The linear shrinkage and porosity curves correspond fairly closely. The minimum pressure likely to prove satisfactory under commercial conditions is 1000 lb. per sq. in.; 1800 to 2000 lb. per sq. in. is recommended as a satisfactory working mean.

—A. B. S.

*Tiles; Effect of variation in pressure and moisture upon the forming of dust-pressed* —. F. W. Walker, jun., and E. G. Kerr. Trans. Amer. Ceram. Soc., 1917, 19, 409–427.

THE effect of a variable percentage of moisture and different pressures in making tiles of constant body-composition and fired at a uniform temperature was investigated. The pressure was applied by means of a large hand-press, the personal factor being eliminated by standardising the drop of the plunger and working the press only under the influence of gravity. Variations in pressure were secured by altering the area of the tiles. The plunger was first allowed to enter the box very slowly so as to compress the dust just sufficiently to eliminate most of the air. It was then raised to a constant height and allowed to drop under its own weight and momentum, two to ten blows being given. The tiles were burned at Mayer cone 10. Each blow after the second caused a constant decrease in the porosity of the tile, if the pressure remained the same. When the pressure per sq. in. was low, there was a considerable variation in porosity for any slight change in pressure, but with high pressures this variation was of less importance than a constancy of moisture content in the tiles. The effect of a variation in the moisture content was important in all cases. Representing the total pressure applied by  $K$ , it was found that at pressures below 0.05  $K$  per sq. in. an increase in moisture content caused a decrease in porosity, but at higher pressures caused an increase in porosity. The practical limit of pressure for any moisture content is 0.2  $K$ . The shrinkage increased with increase of pressure for all dusts up to 0.25  $K$ ; beyond this the shrinkage decreased. The shrinkage at any given pressure above 0.05  $K$  decreased with a decrease in the moisture-content.—A. B. S.

*Tunnel kiln; Heat balance of a continuous —.*  
C. B. Harrop. Trans. Amer. Ceram. Soc., 1917,  
19, 216—247.

The kiln investigated is of the moving-car type, 197 ft. long, 4 ft. 4 in. wide, and 8 ft. high from the top of the rails to the centre of the crown. It is heated by four hand-fired furnaces with horizontal grates, two on each side of the kiln and 108 ft. from the inlet end. The grate area of each furnace is 16½ in. by 39 in. The ash-pits are depressed and kept full of water. The waste gases pass out of the kiln, at a point 18½ ft. from the inlet end, to two vertical flues—one in each side wall of the tunnel—and down into the main flue which runs transversely underneath the kiln, and leads to a chimney, 126 ft. high by 5 ft. 6 in. diameter inside, at the base, and 5 ft. 6 in. by 15 in. at the top. Each car normally carries about 900 firebricks, weighing nearly 3 tons when burned, but, during the test, refractory ware of various sizes was burned along with the bricks and the average load was only 2.58 tons. The kiln holds 36 cars: 17 heating up, 4 in the high fire zone, and 15 cooling. The firebricks are burned to cone 14. One car is removed every two hours, another car of fresh bricks then enters the kiln, and all the remaining cars are advanced one car-length. The furnaces are usually fired immediately after moving the cars, but occasionally at shorter intervals. The grates are cleaned twice each 24 hours. The test covered a period of only six hours. The following results—expressed as percentages of the heat in the fuel supplied—were obtained:—3.27% in unconsumed combustible, 23.30% leaving kiln in dry combustion gases and dry excess air, 0.33% leaving kiln in the moisture which came in with the excess of air and air required for combustion, 1.79% leaving kiln in moisture evaporated from ash-pits, 3.67% leaving kiln in vaporised mechanical and hygroscopic water in combustion gases, 4.22% consumed in dehydration of clay and leaving in vaporised chemical water in combustion gases, 2.52% leaving kiln in unconsumed combustible matter in combustion gases, 17.07% leaving kiln in burned ware and clay in car structure, 0.20% leaving kiln in ironwork of car structure, 45.00% radiation and convection loss. The average excess of air in the flue gases was 352%, and by reducing it to 100% about 13.3% of the fuel would apparently be saved. If the greater part of this excess of air comes through the combustion-zone of the kiln, a reduction in its amount would lower the chimney-temperature, which is scarcely permissible. If, on the contrary, the greater part of the excess of air enters at the inlet end of the kiln, a reduction of the amount of air would result in a higher chimney-temperature, but would show no saving in fuel. About 203% of air leaked through the inlet end of the kiln. The total fuel consumption was 11.15% of the weight of the goods burned, or 725 lb. per thousand bricks.—A. B. S.

*Porcelain bodies; Temperature-porosity-volume changes in —.* G. A. Loomis. Trans. Amer. Ceram. Soc., 1917, 19, 636—641.

The author recommends that temperature-porosity and temperature-shrinkage graphs of chemical and other hard porcelain bodies in commercial use should be plotted, so that their behaviour in burning may be accurately studied and loss in manufacture prevented. The maturing-temperature, the temperature at which over-firing begins, and the permissible range of temperature can be ascertained from these graphs. A body which remains constant in volume during a long temperature-interval, whilst the porosity remains at a minimum, is certain to be superior to a body with a shorter range.—A. B. S.

*Bone-ash [in pottery]; Apatite, a substitute for —.* N. B. Davis. Trans. Amer. Ceram. Soc., 1917, 19, 125—129.

PRELIMINARY tests indicate that natural apatite of great purity may be used as a substitute for bone-ash in the manufacture of china ware. Commercial precipitated calcium phosphate is unsatisfactory on account of the impurities present, but this objection does not appear to apply to selected pieces of the mineral phosphate.—A. B. S.

*Marquardt porcelain and magnesium aluminate; Production of special refractories, viz., —.*  
F. H. Riddle. Trans. Amer. Ceram. Soc., 1917,  
19, 397—408.

A CONTINUATION of an investigation on the manufacture of porcelain tubes for pyrometers (Howat, this J., 1917, 715). The materials are blunged with water to form a slip, which is passed through a 120-mesh sieve and then filter-pressed. For making pressed tubes, a solution of 0.16 part of gum tragacanth per 100 parts of dry body is added and the mass is thoroughly pugged in a pill-mixer for about 20 mins. It is afterwards forced through a small hydraulic pipe-press. The collars and closed ends must be fixed on afterwards. For casting, sufficient water is added to the filter-press cakes to make 60% of the dry weight and, after thorough blunging, sufficient of a solution of 3½% sodium silicate and 10% sodium carbonate is added to thin the slip so that 200 c.c. will run through a Pleininger viscometer in 2 mins. (Technical Paper 51, U.S. Bureau Standards; see also this J., 1917, 875). The plaster moulds are cast around a cold-drawn steel rod with a rounded end, in which a hole ⅛ in. diameter and ⅜ in. deep is drilled. A steel pin, about 3 in. long is fitted loosely into the hole, and when withdrawn provides an air-vent at the end of the mould. When using the mould this air-vent is plugged with a small piece of wire which projects about ½ in. into the mould and is kept in place by soft clay. The mould is filled with the slip, which is allowed to stand for 1—4 mins. (according to the diameter and thickness of the tube); afterwards the wire is drawn out whilst the mould is turned round and the surplus slip is poured out. If the wire is drawn too soon, the vent will be blocked with slip; if it is left in after the slip is poured out, the tube will collapse. The tubes remain in the moulds for 24 hrs.; they are then withdrawn and laid close together on sheets of plate glass and dried for 24 hrs. The tubes are suspended by their flanged heads in tall saggars, each 14 in. × 3 in. and containing only one row of tubes, and burned at cone 1. They are then sprayed with a glaze-mixture corresponding to  $\text{CaO}, \text{Al}_2\text{O}_3, 4\text{SiO}_2$  and re-burned at cone 17 in 5 or 6 hrs., rapidity of heating being important. During the burning, the tubes must swing freely and be heated uniformly or they will warp. If two rows of tubes are burned in a saggar they will warp. The tubes produced were similar in character to the German tubes, but the latter are rather thicker and sag slightly less when pieces 8 in. long are placed on supports 7 in. apart and are heated to cone 22.

Magnesium aluminate (spinel,  $\text{MgO}, \text{Al}_2\text{O}_3$ ) is the most stable compound of the system lime-alumina-magnesia. Its fusion point is 2135° C., as compared with 1816° C. for  $\text{Al}_2\text{SiO}_5$ , 1387° C. for  $5\text{CaO}, \text{Al}_2\text{O}_3$ , 1587° C. for  $\text{CaO}, \text{Al}_2\text{O}_3$ , 1725° C. for  $3\text{CaO}, 5\text{Al}_2\text{O}_3$ . It may be prepared by grinding a mixture of 45.2% of flocculent magnesium carbonate or Californian magnesite with 54.8% of calcined alumina in a ball-mill, tempering the product with water, making it into balls of about 1½ in. diameter, and calcining these at cone 22—30. The calcined



material is ground dry to three sizes (40F, 20F and 20—40). The material is non-plastic and cannot be cast satisfactorily. If made into a paste with water, it can be shaped by means of a "jolley" in moulds coated with oil in which fine graphite is suspended. The ware should be removed from the moulds as soon as possible, dried, and burned. At cone 17, the material forms a cream-white, porous, biscuit ware, but at cone 35 it vitrifies to a dense, translucent body, not easily attacked by molten slag or iron, sensitive to exceedingly rapid cooling, but withstanding ordinary usage. It should be useful for furnace linings, crucibles, tubes, combustion-boats, and similar products.

—A. B. S.

*Silica bricks; Volume-changes of some commercial — on heating.* D. W. Ross. Trans. Amer. Ceram. Soc., 1917, 19, 83—93. (See also this J., 1917, 718.)

Silica bricks which have not been heated sufficiently during manufacture increase both in porosity and "outside volume" when reheated to 1400° C. 1450° C., or 1500° C., the increase in porosity being roughly proportional to the increase in the volume of the solid material. With material which has attained equilibrium at 1450° C. there is very little change on reheating at 1500° C., and there may even be a slight decrease in the outside volume, due to an increase in the density of the solid material. By comparing the percentage increase in volume which occurs on a single reheating to each of three higher temperatures or on repeated heatings at a single temperature, an idea can be obtained as to how far each test-piece was from equilibrium at the reheating temperature. The amount of skin surface or surface of the original brick on the test-pieces did not affect the results. Test-pieces of less than 150 c.c. volume tend to give inaccurate results and still larger pieces are preferable. Increase in pore-space is usually caused by and is proportional to an increase in the volume of the solid material. The influence of small cracks on the increase in volume is usually negligible. An effect of minor importance is the fluxing and consequent shrinkage of some particles which tend to counteract any increase of pore-space. The extremes of porosity in the original bricks were 22.64% and 31.96% respectively. A volume-expansion of 1.45% at 1400° C., 1.38% at 1450° C., and 5.66% at 1500° C. may be expected when commercial (American) silica bricks are reheated at these temperatures. When such bricks are built into a furnace, a somewhat greater expansion may be anticipated. The specific gravity of a silica brick also affords an easy and accurate means of determining the degree to which it has been burned, raw silica having sp. gr. 2.59—2.66 and silica completely inverted to tridymite and cristobalite sp. gr. 2.27 and 2.33 respectively.—A. B. S.

*Grog in fireclay bodies; Effect of size of —.* F. A. Kirkpatrick. Trans. Amer. Ceram. Soc., 1917, 19, 268—300.

Grog was made by crushing high-grade firebricks (made from a mixture containing 50% flint fireclay) in a wet-pan and then screening it through sieves so as to obtain the following sizes: (a) 4—8 mesh, (b) 8—12 mesh, (c) 12—20 mesh, (d) 20—40 mesh, (e) 40—80 mesh, (f) 80-mesh—dust. The various fractions were mixed with clay in different proportions and the mixture was wetted and pugged. Test-pieces were made by hand-moulding, drying in air for at least 3 days, then at 65° C. for one day, and finally at 100° C. for 2 days. Some of the test-pieces were burned at cones 10, 12, and 14. *Tests using grog particles of one size.*—The body with grog of size *f* vitrified more rapidly than the others.

The 80-mesh formed a dividing line between the coarse or slowly vitrifying and the fine or rapidly vitrifying grog sizes. Some fine grog was necessary for development of high strength by vitrification. From sizes *a* to *e*, the strength increased with the porosity, probably due to drying cracks; the size of these cracks decreased with the coarser grog. From sizes *a* to *e*, the strength increased with the increase of porosity; it also increased with the surface factor of the grog. Size *e* gave the most porous bodies. Specimens containing the larger sizes of grog gave the best quenching tests. *Tests using grog of various sizes.*—The strength of raw bodies varied as the surface-factor in the mixtures containing grog sizes *a*, *b*, and *c*, and *b*, *c*, and *d*, respectively, but not in those containing grog sizes *c*, *d*, and *e*, and *d*, *c*, and *f*. The strength varied directly with the porosity in the first three mixtures mentioned, but inversely as the porosity in the fourth mixture. Mixtures of grog of different sizes gave denser bodies than single sizes of grog, provided the smaller grog-particles packed well into the voids between the larger ones. Grog of size *e* fits into the voids between either size *c* or *d*, and a mixture containing 25—66.7% of size *d*, 0—25% of size *c*, and 33.3—66.7% of size *f* approached the theoretical ratio and gave the strongest (unburned) product, though most of the particles in size *e* were too large. Mixtures of equal parts of sizes *a*, *b*, and *c*, and of equal parts of sizes *d* and *e*, and size *f* alone gave the densest, but not the strongest bodies. If a mechanical analysis of the grog mixtures is made, that one will give the strongest test-pieces which yields a graph the lower part of which is parabolic and the upper part is a straight line. In the burned state, the surface-factor affords an accurate means of judging the strength of the material, but the porosity is often misleading. The strength increased with a higher burning temperature. Mixtures containing grog sizes *b*, *c*, and *d* gave the best quenching tests. Grog for glass-pots and similar bodies should not contain too much fine material or that of intermediate sizes, as this is a source of low strength and cracking during drying. The best bodies for this purpose have compositions corresponding closely to the parabola previously mentioned, and have a modulus of rupture of 275—300 lb. per sq. in. in the dried state and 3500—4400 lb. per sq. in. in the burned state. For saggers having walls and bottoms 1 in. in thickness, a mixture of 1 part of grog of size *a*, 3 parts of size *b*, and 1 part of size *c* is recommended where resistance to repeated heating and cooling is desired more than strength, whilst a mixture of two parts of size *c* and 1 part of size *d*, or the use of size *c* alone, will give greater strength at some sacrifice of durability. Both very large and very fine grog are alike undesirable in saggers.—A. B. S.

*Firebricks; Modified load-test for —.* J. B. Shaw. Trans. Amer. Ceram. Soc., 1917, 19, 498—506.

In order to classify high-grade firebricks more closely than is possible when a constant load is applied, the author applies the pressure by means of a spring so that the load diminishes when the brick begins to yield or is crushed. It is suggested that a test under diminishing pressure bears a closer resemblance to furnace conditions than a test with a constant load.—A. B. S.

*Lead glazes; Cost of raw —.* H. F. Staley and L. C. Hewitt. Trans. Amer. Ceram. Soc., 1917, 19, 659—673.

A SERIES of 108 glazes was composed of the following ingredients: white lead at 3½d. per lb., felspar.

whiting, clay, and flint each at  $\frac{1}{2}$ d. per lb. Replacement of lead oxide by felspar did not affect the texture, but made the glazes more refractory. Increasing the lime and reducing the lead oxide and felspar in equal proportions made the glazes more refractory and tended to make them matt. An increase in the clay content made the glazes more refractory and dull. An increase in flint usually made the glazes more fusible and glossy. Increasing the temperature increased the number of bright glazes, especially those rich in lead oxide. For use at cones 05–3, the cheapest bright glazes cost 1-4d. –2-25d. per lb. The cheapest silky matt glazes cost 1-65d. –2-25d., and the cheapest watery matt glazes 1-65d. –2-25d. per lb. In the discussion, it was pointed out that the cost of the glaze materials is so small a part of the total cost of the ware, that the production of a minimum amount of defective ware, rather than the cost of the glaze, should have the chief consideration.—A. B. S.

*Lead glazes; Physical and chemical properties of lead compounds and their use in raw* —. F. A. Morgan. Trans. Amer. Ceram. Soc., 1917, 19, 442–448.

Litharge and red lead may be substituted for white lead (usually with a saving in cost) if the necessary adjustment is made in the viscosity of the glaze and the same equivalent of PbO is used. The "fumed oxides" are equal in purity to white lead and though they have a higher sp. gr. they will remain in suspension longer on account of their great fineness. When dry, the oxide glazes are harder and so produce less dust than those containing white lead.—A. B. S.

*Lead glazes; Study of three-component normative systems in raw* —. W. G. Whitford. Trans. Amer. Ceram. Soc., 1917, 19, 312–330.

The compositions of various raw glazes were represented in triangular diagrams with the following end-members:—Anorthite series: orthoclase (potash felspar), anorthite (made from whiting and kaolin), and lead metasilicate (made from white lead and flint). Wollastonite series: orthoclase, wollastonite (made from whiting and flint), and lead metasilicate. Zinc silicate series: orthoclase, zinc metasilicate (made from zinc oxide and flint), and lead metasilicate. The glazes were applied to white, biscuit wall-tiles and burned at cones 05, 02, 1, and 3 respectively for from 12 to 16 hrs. When the end-members are properly chosen, the normative system of glaze calculation and blending (this J., 1912, 1128) gives series of glazes whose physical properties vary in a regular manner. The glazes in the anorthite series are more refractory than the others and contain a larger proportion of matt glazes. Mattiness is a function of temperature and composition; it is determined by the anorthite content and bears no relation to the oxygen ratio. Most of the glazes of the wollastonite series were bright, the division between these and the dull glazes being fixed at each temperature by the wollastonite content. Most of those in the zinc silicate series were bright glazes and stoneware enamels, but a few matt and some truly crystalline glazes were produced.—A. B. S.

*Fritted glazes; Control of fusibility in* —. H. F. Staley and R. J. Riley. Trans. Amer. Ceram. Soc., 1917, 19, 642–652.

WITHIN fairly large limits, equal melted weights of potassium, sodium, calcium, magnesium, zinc, lead, and boron oxides have about the same effect on the fusibility of glazes. Two-thirds of a pound of flint increases the refractoriness of a frit rather more

than 1 lb. of felspar. Enamels high in felspar are better in every way than those high in flint. Enamels rich in lime are pink in colour.—A. B. S.

*Matt glazes; Effect of porosity of the body and moisture content of the glaze in the application of* —. F. S. Hunt. Trans. Amer. Ceram. Soc., 1917, 19, 428–441.

THREE representative raw lead matt glazes, with different moisture contents, were applied to tiles of a constant composition, but with porosities varying from 14 to 31%. It was found that the weight of glaze per unit area determines the texture of the glaze surface. The best matt effects were produced when the burned glaze had a weight of 0.100 to 0.135 grain per sq. cm. of tile area.—A. B. S.

*Glazes; Crawling of matt* —. C. F. Binns and M. E. Saunders. Trans. Amer. Ceram. Soc., 1917, 19, 597–600.

THE factors which chiefly affect the tendency of matt glazes to crawl are the burning of the body and the grinding of the glaze. The glaze fell off some short-fired pieces in large flakes, even when mucilage had been added to the glaze-slip. With over-ground material, the addition of mucilage is useful, especially when all the clay in the glaze is calcined. The best results were obtained with a glaze made from calcined clay, ground for half an hour, mixed with mucilage, and applied to a well-fired body which had previously been soaked to saturation.—A. B. S.

*Bristol glazes compounded on the eutectic basis; Practical application of* —. A. S. Watts. Trans. Amer. Ceram. Soc., 1917, 19, 301–302.

THREE glazes were selected from the eutectic area mentioned in a previous paper (see this J., 1917, 716), having the following compositions:—

		A	B	C
Felspar	...	60.87	57.07	50.10
Whiting	...	6.25	5.86	9.01
Zinc oxide	...	7.76	10.69	9.73
China clay	...	7.06	8.15	9.07
Ball clay	...	7.06	8.15	9.07
Flint	...	11.00	10.08	13.02

They were mixed with water to form a slip weighing 22 oz. per pint. When properly ground and mixed, these glazes are satisfactory between cones 3 and 14 on a large variety of clays. In no case was crawling or crazing noted. At cones 3, 4, and 5, these glazes produce fine smooth enamels, suitable for novelty pottery and cooking ware; at cone 10 they are opaque enough for use on buff-burning clay, and at cones 12 to 14 are useful for electrical porcelain.—A. B. S.

*Terra-cotta; Pinholing and peeling on* —. H. Wilson. Trans. Amer. Ceram. Soc., 1917, 19, 209–215.

"PINHOLES" are caused by small tongues of under-slip which rise through the glaze and leave a clean hole with a distinct projection. They are not merely small blisters or holes in the glaze. Pinholes are produced when a second coating of under-slip or a coating of glaze is sprayed on to dry terra-cotta, when no barium salt is added to a body containing soluble salts, when an excess of barium carbonate is added to the clay, and when the dry ware is brushed over with a 10% solution of barium chloride or fluoride previous to slip-coating and glazing. In the case of all glazed ware, pinholing is worse in glazes which flow least in firing. Pin-



holes are not prevented by rubbing the dried and glazed surface, by avoiding the use of oil, or by brushing away all dust before coating the ware. They are not caused by particles of plaster from the mould or by the slip- or glaze-coatings being too dry before a further coating is applied. They are partially prevented by removing the surface of the ware with sand-paper, by firing the underslip with no glaze covering, and by adding 2% of a mixture of barium carbonate and fluoride. They are wholly prevented or remedied by re-spraying the burned pieces with glaze and reburning or, preferably, by adding 2% "gum arabic sorts" to the underslip and 3% to the glaze.—A. B. S.

*Magnesia-alumina-silica mixtures; A deformation study of —.* A. S. Watts. Trans. Amer. Ceram. Soc., 1917, 19, 453–456.

THE most fusible mixture of magnesium carbonate, china clay, and flint contained 20% MgO, 20%  $\text{Al}_2\text{O}_3$ , and 60%  $\text{SiO}_2$ , and had a deformation temperature of cone 12. The deformation was very rapid. Mixtures containing less than 40%  $\text{SiO}_2$  curled forward at cone 8, but this was not true deformation. Mixtures containing 35% MgO or more shrank considerably at cone 10.—A. B. S.

*Baryta-alumina-silica mixtures; A deformation study of —.* A. S. Watts. Trans. Amer. Ceram. Soc., 1917, 19, 457–459.

THE most fusible mixtures of barium carbonate, china clay, and flint contained 35% BaO, 10%  $\text{Al}_2\text{O}_3$ , 55%  $\text{SiO}_2$ , and 40% BaO, 10%  $\text{Al}_2\text{O}_3$ , 50%  $\text{SiO}_2$  respectively. They both deformed completely at cone 6. Five other mixtures deformed at cone 7, ten at cone 8, seven at cone 9, and four at cone 10. No mixture of barium carbonate and flint deformed at or below cone 10. The ternary mixtures containing 10%  $\text{Al}_2\text{O}_3$  fused to clear glasses; those with only 5%  $\text{Al}_2\text{O}_3$  were opaque.—A. B. S.

*[Crystalline glasses.] Investigation of the ternary system willemite-tephroite-soda glass.* H. C. Arnold. Trans. Amer. Ceram. Soc., 1917, 19, 674–695.

WILLEMITE ( $2\text{ZnO} \cdot \text{SiO}_2$ ), tephroite ( $2\text{MnO} \cdot \text{SiO}_2$ ), and soda glass ( $\text{Na}_2\text{O} \cdot 2\text{SiO}_2$ ) were prepared synthetically, and mixtures of these in various proportions were heated to 1175° C. in  $3\frac{1}{2}$  hrs., the results being plotted on a triangular diagram. The most promising specimens were re-melted at cone 12 in 24 hrs. and cooled for 62 hrs. Long acicular crystals of willemite grow most readily in a mixture of equal parts of willemite and soda glass. A larger proportion of willemite produces crystals which are too small to be satisfactory. Crystallisation is less definite in the presence of manganese than in that of zinc. Three compounds of manganese are produced: hausmannite,  $\text{Mn}_3\text{O}_4$ , is first formed, its development being greatly increased on prolonged heating at a temperature corresponding to moderate viscosity of the glaze. If the mass is free to attack silicious material, rhodonite (manganese trisilicate) is formed, but otherwise tephroite (manganese orthosilicate). Evidence of immiscible liquids was found in various parts of the field. The material with the lowest melting point (772° C.) is opposite the glass-willemite eutectic and close to the glass-tephroite eutectic in the triangular diagram.—A. B. S.

*Chrome-tin colours at cone 9.* R. H. Minton. Trans. Amer. Ceram. Soc., 1917, 19, 378–396.

To develop a pink colour underglaze at cone 9, the glaze must contain tin oxide. The composition of

the glaze appears to be more important than that of the stain. Re-firing weakens the colour and may destroy it. Strontium chromate and barium chromate produce stronger colours than any other chromate used, with chromic oxide as the next best. Zinc chromate also produces a strong colour. Lead chromate does not yield dark reds and causes milkiness. Potassium bichromate is the best source of chromic oxide for clear reds. Calcium fluoride is superior to calcium carbonate or sulphate. Milkiness appears to be the result of incomplete reaction between the chromic oxide and the lime (depending on the source of each) and on insufficient lime. Unwashed stains are preferable for the stronger colours.—A. B. S.

*Overglaze colours at cone 6–7.* H. Wilson. Trans. Amer. Ceram. Soc., 1917, 19, 653–658.

To glaze a large number of pieces of ornamented terra-cotta in two colours, the raised work being white on a coloured background, the whole of the pieces were first sprayed with white glaze slip containing gum arabic, and a coloured overglaze stain was then applied with a fine air brush. Any stain which had fallen where it was not required was removed (when dry) with a stiff brush, the gum in the glaze making it sufficiently hard to resist this treatment. A final coat of a dilute solution of gum arabic was applied in some cases to prevent the overglaze from "dusting." The pieces were then fired in the usual way at cone 6–7. For yellow backgrounds, a mixture of a commercial yellow stain, powdered rutile, and uranium sulphate or nitrate, ground with water, was used; for pink or rose backgrounds, a mixture of tin oxide, whitening, potassium bichromate, and flint; and for brown backgrounds a mixture of this or a chrome green stain with the yellow mixture mentioned above. For violet backgrounds, a mixture of china clay, zinc oxide, flint, sodium nitrate, and cobalt oxide was used, and for green backgrounds, a mixture of cobalt oxide and uranium nitrate stains.

—A. B. S.

*Enamels; American clays for floating —.* J. B. Shaw. Trans. Amer. Ceram. Soc., 1917, 19, 339–360.

A TENNESSEE and a Florida ball clay were each found to be satisfactory substitutes for the Valendar (German) clay commonly used for keeping the non-plastic ingredients of an enamel slip in suspension and for making the slip of suitable consistency. For measuring the viscosity of the slips a viscometer was used which has two outlets  $\frac{3}{32}$  in. and  $\frac{5}{32}$  in. diameter respectively. The viscosity is found by dividing the volume of slip flowing through one outlet by that which flows simultaneously through the other, and so avoids referring the viscosity to water as unity. Enamel slips of high viscosity and low sp. gr. give the best results. By keeping the consistency of the slip (*i.e.*, the viscosity divided by the sp. gr.) constant, more uniform results are obtained in enamelling. The quantity of flocculating agent (aluminium or magnesium sulphate) present in the slip is unimportant so long as sufficient is added to flocculate the clay; 0.5% of magnesium sulphate, as ordinarily used in commercial practice, was found to be better than stannic chloride (0.5%) or borax (3%). The use of a flocculating agent prevents the enamel from flowing down the sides of the ware in an undesirable manner, even when the slips have a low sp. gr. The maximum viscosity obtained was 6.5, but a value as low as 4.5 is permissible if the volume-weight of the slip is about 150 grms. per 100 c.c.

—A. B. S.

*Enamel surfaces under the microscope.* E. P. Poste. Trans. Amer. Ceram. Soc., 1917, 19, 146—159.

In continuation of previous work on the resistance of enamels to acids (this J., 1917, 879), various pieces of enamelled ware (including pieces fired above and below the usual temperature) were examined under a microscope with a magnification of 75 diameters. The results showed that marked variations in the physical condition of the enamel are brought about by different heat-treatments. Similar variations were observed in samples of commercial enamelled ware. In under-burned pieces, the enamel consists chiefly of a glassy matrix containing fine bubbles and particles of unfused material. In normally burned enamels, the unfused matter is absent and the bubbles run together forming larger ones. In over-burned enamels, the bubbles continue to combine until they produce surface-pitting visible to the naked eye. Enamels which had been treated with warm hydrochloric acid for several hours were also examined. The acid tends to soften the enamel, to dissolve some of the constituents, and to leave behind a fine white powder which can be rubbed off when the surface is dry. The paper is illustrated with 14 photomicrographs.—A. B. S.

*Latent heats of fusion of lime and magnesia.* Washburn. See VII.

*Instrument for measuring plasticity.* Emley. See IX.

#### PATENTS.

*Glass furnace; Electric* —. J. O. Erskine, Steubenville, Ohio. U.S. Pat. 1,267,317, May 21, 1918. Date of appl., Nov. 21, 1917.

Two electrodes are partially immersed in the glass-producing materials and an electric current is passed so as to melt the materials and produce glass continuously. The charge forms the sole resistance for generating the heat by which melting is accomplished. The melting and working chambers of the furnace may each be provided with a separate set of partially immersed electrodes.—A. B. S.

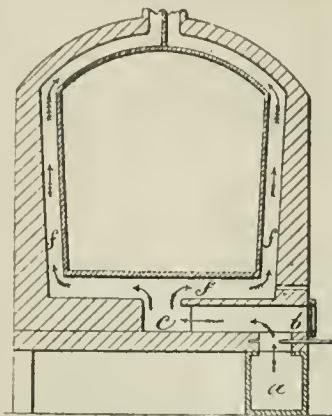
*Sheet glass; Method of and apparatus for drawing* —. T. H. Chadwick, Arnold, Pa. U.S. Pat. 1,267,849, May 28, 1918. Date of appl., Nov. 23, 1916.

A continuous sheet of glass is drawn from the molten "metal" by means of a gripping device and elevator. The edges of the sheet are cooled by an air-blast and the glass is then cut into individual sheets by a travelling oxy-acetylene flame, the severed sheets being moved at a more rapid rate than the continuous one.—A. B. S.

*Kilns for earthenware, tiles, and other articles.* W. Bennett, Stoke-on-Trent. Eng. Pat. 117,147, July 16, 1917. (Appl. No. 10,257 of 1917.)

A GAS-FIRED kiln or oven in which the flame is equally distributed over the kiln bottom and through the walls is constructed with a gas flue, *a*, extending longitudinally at one side of the kiln, and connected with a series of transverse conduits, *b*, which lead to a combustion chamber, *c*, placed midway in the kiln and extending the whole length of the latter. Parallel to the transverse gas con-

duits and alternating with them are air passages. The products of combustion from the chamber, *c*,



pass up around the sides of the kiln through flues, *f*, to the chimney. Means of regulating the gas and air are provided.—A. B. S.

*Tunnel kilns [for pottery]; Continuous* —. G. H. Benjamin, New York. Eng. Pat. 117,383, Nov. 12, 1917. (Appl. No. 16,592 of 1917.)

The kiln is heated by a series of internal horizontal flues or combustion chambers placed end to end and built up in sections to allow them to expand or contract without injury. Air and gas are supplied to these chambers under pressure. The products of combustion are withdrawn and used in an external air-heater, to heat the air required for the combustion of the gas used as fuel and also for supplying a current of air to the interior of the oven. The circulation of the air is effected by means of an external pump.—A. B. S.

*Glaze used in the manufacture of pottery and like articles.* W. P. and W. H. Edwards and W. Mollart, Stoke-on-Trent. Eng. Pat. 117,047, Feb. 13, 1918. (Appl. No. 2579 of 1918.)

CALCINED shells are ground to a very fine powder, mixed with water, and added to ordinary potter's glaze in the proportion of 2 oz. to 1 cwt. of glaze. The mixture is agitated for about 5 mins., when it thickens, but may be diluted with water to a convenient consistence. It is claimed that the use of the powder produces a better distribution of the glaze and reduces the amount of glaze material (particularly lead compounds) used.—A. B. S.

*Platinizing earthenware, pottery, and the like.* F. J. Kettel, Beckenham, Kent, A. Gasch and T. A. Dean, London. Eng. Pat. 117,432, May 13, 1918. (Appl. No. 8003 of 1918.)

The article is covered with a solution or emulsion of platinum chloride, phenylhydrazine, and glycerol, heated sufficiently (e.g., to 850°–900° C.) to reduce the platinum to the metallic state, and then allowed to cool gradually. The solution or emulsion is prepared by dissolving 3 grms. of platinum chloride in the least possible quantity of water, adding 12 c.c. of glycerol, and heating the mixture to 100° C., at which temperature it is maintained for about 1½ hrs. The phenylhydrazine is then added, drop by drop, stirring after each addition, and after settling, the clear liquid is decanted.

—A. B. S.



*Refractory furnace lining and process of making.* S. B. Newberry, Cleveland, Ohio. U.S. Pat. 1,267,686, May 28, 1918. Date of appl., June 25, 1917.

LIMESTONE or magnesian limestone is ground and mixed with 2–10% of oxides of the aluminum-iron group, capable of forming fusible compounds with lime and substantially free from silica. The mixture is calcined to form a dense clinker.—A. B. S.

*Aluminous abrasives.* T. B. Allen, Toronto, Canada, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pats. (A) 1,268,532 and (B) 1,268,533, June 4, 1918. Dates of appl., (A) Nov. 11, 1916; (B) Aug. 7, 1917.

(A) An aluminous ore containing 15% or more of iron oxide and silica is heated to fusion in a reducing atmosphere and is then allowed to cool and crystallise. (B) A crystalline fused aluminous abrasive material containing more than 60% of crystalline alumina and more than 10% of silica, iron oxide, and titanium oxide, the major portion of the crystals being less than 0.01 mm. in diameter.

—A. B. S.

## IX.—BUILDING MATERIALS.

*Paving bricks from blast-furnace slag.* J. B. Shaw. Trans. Amer. Ceram. Soc., 1917, 19, 507–522.

THE author claims that the possibility of using American slags for the manufacture of paving bricks has been conclusively proved. The characteristics of the bricks depend more on the manner in which the slag is cooled than on its composition. The following process is recommended: The slag from the furnace is poured into a tilting frit-furnace and any additional materials (such as 10–20% of silica or iron oxide) required to bring it to a desired composition are then added. The mixture is brought to a quiet fusion, and run into moulds. The moulds with their contents are immediately placed in an annealing furnace at the softening temperature of the bricks, and are maintained at this temperature until the bricks are uniform throughout; or the bricks are buried in sand. The rate and mode of annealing are of great importance and determine whether tough, good bricks or brittle ones are produced.—A. B. S.

*Gypsum: Measurement of the time of set of calcined* —. W. E. Emley. Trans. Amer. Ceram. Soc., 1917, 19, 573–584.

THE normal consistency of the plaster is first determined, using the Southard instrument as recommended by the American Society for Testing Materials. From this is ascertained the amount of water to be added to 300 grms. of the sample. This quantity of water is placed in a casserole and 300 grms. of the sample placed in it, without splashing, in less than 2 secs. The plaster is allowed to soak quietly for 58 secs., the mixture is stirred vigorously for 1 min., and is then poured into a mould made of hard rubber and dipped in melted paraffin. At 1-minute intervals it is tested with a standard Vicat needle. The time which elapses between the adding of the sample to the water and that when the needle no longer penetrates to the bottom of the mass is recorded as the time of final set. The mould can be cleaned readily, if it is first heated very gently. No method has been found for determining the time of initial set, but as this does not appreciably affect the ultimate properties of the plaster, it is unimportant.—A. B. S.

*Plasticity [of wall-plasters]; Instrument for measuring* —. W. E. Emley. Trans. Amer. Ceram. Soc., 1917, 19, 523–533.

As the plasticity of a wall-plaster is usually judged by means of a trowel held at an angle of 10°–15° to the wall, an instrument was devised in which the trowel is replaced by a cone mounted point downwards, the side of which makes an angle of 10° with the horizontal, and the wall is represented by a disc of plaster of Paris of known absorption mounted on a vertical shaft, below and concentric with the axis of the cone; the shaft is threaded and runs through a fixed nut. The material of which the plasticity is to be measured is placed on the disc and the latter is forced upwards against the cone with a spiral motion at a constant speed (this being more convenient than moving the "trowel" over the disc). The pressure exerted is analogous to that component of the force exerted by the trowel which acts normally to the wall; the tangential force of the turning movement of the disc over the face of the cone brings into play that component of the force which is parallel to the wall. The cone is so mounted that either of these forces is able to cause motion independently of the other, this motion being transmitted through a system of levers to one of two bars suspended like pendulums. When a constant force moves the cone, the bars swing out until in equilibrium with the force of gravity acting on them; both the cone and bars then become stationary and the force acting on the cone is directly proportional to the size of the angle through which the bars have moved. The machine is run at a constant speed for a definite time—say 3 mins. A tangential force of (say) 2 grms. per sq. cm. is arbitrarily assumed as the starting point and a boundary ordinate erected through this point, the other being at a point representing 3 mins. later. The position of the first ordinate depends on the consistency of the paste under examination, but the average value of the tangential force between the two ordinates (which is the plasticity-figure) is independent of the consistency. The plasticity-figure for limes lies between 5 and 20. The ratio of the tangential to the vertical force with clays is so much greater than with limes that the machine would have to be modified to make it suitable for use with clays.—A. B. S.

*Volume changes of some commercial silica bricks on heating.* Ross. See VIII.

## PATENTS.

*Concrete and the like.* W. J. Stewart, London. Eng. Pat. 117,504, July 25, 1917. (Appl. No. 10,690 of 1917.)

AN aggregate of low specific gravity is made by moulding clay into spheres of various sizes (e.g.  $\frac{1}{4}$  in.,  $\frac{1}{2}$  in., and  $\frac{3}{4}$  in. diameter), burning these at a suitable temperature, and glazing them. Before the fused glaze has become hard, the spheres are caused to pick up sand so as to give them a rough surface. If a vitreous clay (such as that used for making blue bricks) is employed, the glazing and sanding are unnecessary. The aggregate thus prepared may be made into concrete by arranging it in a mould in a series of layers of different sizes and then filling the mould with the cement matrix from below upwards.—A. B. S.

*Binder for roads, etc., and process for making same.* E. A. Paterson, Thorold, Canada, U.S. Pat. 1,266,618, May 21, 1918. Date of appl., Jan. 5, 1916.

A BINDER for stony agglomerates is made by treating a solution of an alkali silicate (sodium silicate)

with a soluble haloid salt of an alkaline-earth (calcium chloride) and with an acid capable of combining with the alkali-metal base of the silicate to form a salt which, in solution, is inert to organic substances, and is gradually removed by weathering. Part of the silicic acid is liberated in the free state and part forms insoluble calcium silicate.

—A. B. S.

*Fibrous composition and process of making the same.* G. A. Henderson, St. Albans, W. Va. U.S. Pat. 1,270,732, June 25, 1918. Date of appl. July 30, 1917.

VEGETABLE material (such as hard wood) consisting of fibres of exactly uniform thickness but of irregular lengths and breadths, is passed through an open-ended vat containing a hot binding and preservative agent so as to expand the pores of the fibres, vaporise the moisture in them, and fill the pores with a predetermined amount of preservative. The steam produced is withdrawn from the vat and the material is compressed into shape.

—A. B. S.

*Timber; Kilns and chambers for drying* —. M. Halkes, Dunston, Lincoln. Eng. Pat. 117,040, Jan. 11, 1918. (Appl. No. 702 of 1918.)

*Saturation of porous fabrics [paper-board] with asphalt.* U.S. Pat. 1,268,430. See V.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Blast-furnaces; Hot blast for* —. O. Simmersbach. Stahl u. Eisen, 1918, 38, 697–703.

IN the Cowper hot-blast stove the combustible gases are distributed uniformly over the whole of the brickwork from the centre to the walls and pass through all the channels with a uniform velocity, usually about 1 metre per sec. With low velocities the gas passes straight up the openings and gives up a relatively small part of its heat, but as the velocity is increased the gas passes up spirally with much better results. The proportion of heat given up by the hot gases to the walls is equal to  $C \cdot v$ , where  $C$  is a constant and  $v$  is the velocity with which the gases pass up the stove. This relationship holds only so long as the critical velocity of 2 metres per sec. is surpassed. With a velocity of 2–3 metres per sec., the transfer of heat is as great in 1 hour as in 3 hours with a velocity of 1 metre per sec. By increasing the height of the stove from 26 to 40 metres, the temperature of the hot blast is raised from 823° to 946° C., while the efficiency is raised from 51 to 66%. The replacement of the usual hexagonal brick with a circular aperture by an octagonal one which, when packed in the stove gives rectangular channels between the bricks as well as the circular ones within each brick, has resulted in a marked improvement. The time required to heat the stove is reduced to one-third and the increase in efficiency is 14%, while the temperature of the gas as it leaves the stove is reduced from 400° C. to 250° C.—F. C. Th.

*Sulphur in iron and steel; Standard apparatus for the determination of* — by the evolution method. H. B. Pulsifer. J. Ind. Eng. Chem., 1918, 10, 545–550.

The apparatus consists of a 500 c.c. conical flask provided with a side tube and a glass stopper through which passes the stem of a tapped 60 c.c.

funnel and the inlet and outlet tubes of a small spiral condenser arranged in the flask just below the stopper. The neck of the funnel carries a short length of glass tube forming a hollow ground-in stopper. Concentrated hydrochloric acid is used for the decomposition, and a current of hydrogen or carbon dioxide is used to remove the liberated hydrogen sulphide, the latter being collected in ammoniacal cadmium chloride solution and determined iodometrically. The condenser in the upper part of the flask maintains the hydrochloric acid at a maximum strength.—W. P. S.

*Steel; Rapid determination of carbon in* — by the barium carbonate titration method. J. R. Cain and L. C. Maxwell. J. Ind. Eng. Chem., 1918, 10, 520–522.

THE method (see this J., 1914, 696, 966) may be simplified and rendered more rapid if carried out as follows. The furnace is heated at 1063° C. and 2 grms. of the sample is placed in the boat, filled with alundum sand, which has been in the hot furnace and then cooled to about 400° C.; the boat is immediately replaced in the furnace, and left for 1 min. with no oxygen passing. Oxygen is then admitted at the rate of 10 to 15 litres per minute, the outlet being constricted so that the rate of flow of gas at this end of the tube does not exceed 225 c.c. per minute. A roll of copper gauze is placed in the front end of the tube and heated at 200° to 300° C. Barium hydroxide solution in a Meyer absorption tube is used to absorb the carbon dioxide, and the barium carbonate formed is collected in a Buchner funnel, washed, and titrated. There should be vivid incandescence while the sample is burning and complete combustion of a 2-grm. sample is usually attained in 2 mins. Closed glass tubes may be inserted in the front end of the combustion tube to reduce the dead space.

—W. P. S.

*Steels; Effect of annealing on the electrical resistance of hardened carbon* —. I. P. Parkhurst. J. Ind. Eng. Chem., 1918, 10, 515–518.

THE total change in resistance increases with the carbon content of the steel; the change is rapid at the beginning of the annealing and becomes slower as the resistance decreases. Since the resistance of a steel changes with the hardness, resistance measurements are a fair indication of the varying rates at which hardened steels are softened by annealing at constant temperature. The larger part of the change occurs within a few minutes, but the change is not complete in 113 hrs.

—W. P. S.

*Electric furnace for annealing and forging of steel; Development of an* —. W. S. Scott. Assoc. Iron and Steel Elect. Eng., Apr. 20, 1918. Chem. and Met. Eng., 1918, 19, 86–89.

EXPERIMENTS with resistor beds of granular graphite, coke, charcoal, and combinations of these showed that all were unsuitable for an electric forging furnace. Ashes or slag from commercially obtainable carbon cause clinkering of the bed with a rapid increase in resistance, and the great temperature difference in different parts of the resistor due to the electrical characteristics of carbon exceed the safe limits of refractory materials. Silicon carbide proved suitable when properly bonded, as the decomposition temperature is above 4000° F. (2200° C.) and blocks can be made of high tensile strength. With increase in temperature its conductivity increases rapidly, as it has a large negative temperature resistance coefficient. In a furnace with a radiating surface of



81 sq. ft., two resistor blocks,  $9 \times 4\frac{1}{2} \times 2\frac{1}{2}$  in., were used, embedded in a refractory lining of crucible clay and graphite. Electrode chimneys or wells packed with granular graphite connected the resistors to external terminal connections. A forging temperature of  $1800^{\circ}\text{F.}$  ( $980^{\circ}\text{C.}$ ) was attained in two hours and the power at full load was 10 kilowatts (300 amp. at 33 volts), though for high-speed steel a preheating chamber is advisable. A uniform temperature together with a neutral atmosphere is obtained in all parts of the heating chamber, with complete control of the rate of heating, thereby eliminating many of the difficulties in reheating high-class steels.—C. A. K.

*Gold precipitation from aurocyanide solution by charcoal.* A. W. Allen. Met. and Chem. Eng., 1918, 18, 642–644.

PRECIPITATION by lump charcoal has never become a commercial proposition owing to the excessive quantity of charcoal necessary, but the Moore-Edmands process makes use of charcoal slime and effects a great improvement in this direction. A special form of extractor is used consisting of a small vacuum filter through which solution and charcoal pulp are pumped; fresh solution is added concurrently with the formation and maintenance of the charcoal cake. The extractors are worked in series, so that one containing nearly exhausted charcoal receives the richest solution. When finally exhausted the charcoal is washed, dried, and incinerated in braziers and the ash is smelted to recover the gold. Charcoals from different woods show widely differing precipitating powers, and the fineness of grinding is also an important factor. The principal drawback appears to be the amount of material to be smelted, though the actual operation entails no special difficulties. Edmands (this J., 1918, 183 A) gives the percentage of gold in the calcined ash as from 10 to 14%, and Walton (Rept. Dept. Mines, W. Australia, 1916) states that 1 ton of charcoal will precipitate 770 oz. of gold with production of about 6 cwts. of ash. Various theories have been brought forward to account for the action, *e.g.*, occluded oxygen, electro-deposition, carbon monoxide, but it is thought that simple adsorption of aurocyanide ( $\text{KAu}(\text{CN})_2$ ) has the most evidence in support.—C. A. K.

*Plating and refining baths; Rôle of complex salts as electrolytes in —.* R. S. Dean and M. Y. Chang. Chem. and Met. Eng., 1918, 19, 83–85.

It has long been known that smoother and more adherent deposits of a metal are produced during electroplating from complex salts than from simple salts at equivalent concentrations, and experiments made on simple salts favour the theory previously proposed by Bancroft (J. Phys. Chem., 1905, 9, 290; see also this J., 1904, 1098) that the crystalline character of a deposit decreases with decreasing concentration of the ion being discharged, *i.e.*, with increasing potential difference between the deposit and the bath. Whether this conclusion can be extended to solutions containing so little silver ion as the complex cyanides cannot be definitely stated. Experimenting on well agitated suspensions of silver oxide, chromate, chloride, thiocyanate, and sulphide, concentrations of the silver ion comparable with that of the cyanide solution were maintained. A deposit was obtained from the oxide suspension, but not from the less soluble salts. The deposition of silver from complex cyanide solutions is due to the discharge of potassium ion, and is secondary. The necessary conditions under which a complex solution will give an amorphous or a smooth deposit are that another metal with a lower solution-tension than

the one being deposited is present, and that a sufficiently complex salt is present that the concentration of the ion being deposited will be so low that its discharge voltage will be above that of the secondary metal.—C. A. K.

*Copper minerals in partly oxidised ores; Determination of —.* F. Cremer. Met. and Chem. Eng., 1918, 18, 644–646.

It is customary to determine the copper content of an ore in two forms, *e.g.*, sulphide copper and "oxide" copper. The usual method for the determination of oxide copper by extraction with a 5% solution of sulphuric acid, when tried on an oxidised ore containing about 20% cuprite and 10% chrysocolla resulted in some metallic copper from the cuprite being left undissolved, so that this, together with all native copper, would appear as sulphide copper. Again, few samples were found which did not contain metallic iron and by deposition of copper the error would be further increased. Phosphoric acid in a solution of ammonium chloride is suggested as a reagent; satisfactory results are obtained if the sample is finely ground before treatment. One gram of the sample is boiled gently with 20 c.c. of a 15% solution of phosphoric acid and an equal volume of a 20% solution of ammonium chloride. When cooled somewhat a small amount of freshly burnt lime and 25 c.c. of concentrated ammonia solution are added, and the flask is well shaken. After filtering, the ammoniacal copper solution is titrated in the ordinary way with cyanide solution. A second extraction is advisable in the case of high-grade oxide ores (say 30%).—C. A. K.

*Aluminium and aluminium alloys; Chemistry of —.* J. G. A. Rhodin. Faraday Soc., July 23, 1918. [Advance proof.] 12 pages.

The specific gravity of copper-zinc-aluminium alloys containing copper and zinc in equal amounts increases as the aluminium content is reduced. An alloy with 96.2% Al has sp. gr. 2.767 and one with 76.9% Al, 3.101. At about 76% Al a point of inflection occurs, the increase of density being then somewhat more rapid; an alloy with 62.6% Al has sp. gr. 3.519. Above 90% Al the sp. gr. falls rapidly. Porosity of the samples or the presence of heavy constituents, such as nitrides, may result in abnormal densities. The specific gravity is markedly influenced by the oxide which the metal can retain in solution. Virgin aluminium or its copper or copper-zinc alloy may normally contain  $1\frac{1}{2}\%$  or so of oxide calculated as  $\text{Al}_2\text{O}_3$ . "Burnt" samples may, as a result of repeated remeltings, contain 9% or more. The following method is given for determining the metallic aluminium directly:—The metal is treated with 10% sodium hydroxide solution and filtered, the undissolved residue containing the oxide present. A part of the filtrate is evaporated with strong hydrochloric acid to dryness, all free acid being removed by heating on a water-bath for at least 1 hour more. The residue is redissolved, sodium carbonate and barium chloride solutions are added, the precipitate is filtered off, washed, and redissolved in hydrochloric acid. The solution is heated to remove all carbon dioxide, and aluminium hydroxide precipitated with ammonia in the presence of ammonium chloride at  $90^{\circ}\text{C.}$  The precipitate is washed with a 5% ammonium nitrate solution, again dissolved in hydrochloric acid, the solution evaporated to dryness, and the residue kept at  $100^{\circ}\text{C.}$  for 2 hours. After removing the silica, the aluminium hydroxide is re-precipitated, filtered off, and washed. The residue insoluble in 10%

sodium hydroxide is dissolved in the minimum quantity of nitric acid, and the copper precipitated with hydrogen sulphide. The filtrate is concentrated, oxidised with bromine, and iron and aluminium hydroxides precipitated. The iron is determined by fusion with potassium bisulphate, reduction with zinc, and titration with permanganate. It is believed that when burnt in air, *e.g.*, by heating the powdered metal in an open crucible, aluminium is converted into  $Al_2O_3$ , which is soluble in the unoxidised metal. The existence of an oxide lower than  $Al_2O_3$  is strongly suggested by the fact that an energetic "thermit" reaction can be induced between alumina and the metal, a grey powder being formed which is insoluble in a 10% sodium hydroxide solution and slowly soluble in nitric acid. The existence of an oxide  $Al_2O_3$  is unconfirmed, the substance having this composition being believed to be a solution of  $Al_2O_3$  in aluminium. The specific heat of commercial aluminium of 98.6% purity between 20° C. and 100° C. is 0.2081, of an alloy of 90% Al with 10% Cu or Zn 0.1918, and of 80% Al and 20% Zn 0.1844.—F. C. Th.

*Chromium; Passivity of* —. A. H. W. Aten. Proc. K. Akad. Wetensch. Amsterdam, 1918, 20, 1119–1134.

When electrolytic chromium in contact with a solution of chromous sulphate is anodically polarised by the passage of a sufficiently strong current, the metal assumes the potential which is characteristic of the passive condition. When the current is stopped, the metal becomes active and its potential is more negative than before polarisation. The activity of the metal after anodic polarisation increases with the strength of the polarising current. Chromium, prepared by Goldschmidt's process, which has been activated by treatment with molten zinc chloride or a mixture of sodium and potassium chlorides, does not readily assume the passive condition when subjected to anodic polarisation in a solution of potassium chloride. The resistance to passivation is greater when the strength of the polarising current is slowly increased, than when this is increased rapidly. If the metal has been previously subjected to anodic polarisation, its resisting power is reduced. If the chromium has been made passive by anodic polarisation, the active condition is restored at once when the solution in contact with the metal is heated. The activity remains on cooling, even when the polarising current is continued, provided that the strength of the current is not too great. (See also J. Chem. Soc., Sept., 1918.)—H. M. D.

[Aluminium and chromium] carbides. O. Ruff. Z. Elektrochem., 1918, 24, 157–162.

ALUMINIUM carbide ( $Al_4C_3$ ) sublimes at temperatures up to 2200° C. without melting and with some decomposition. The melting point of aluminium carbide lies higher than this. At 2200° C. aluminium carbide is in equilibrium with graphite, molten aluminium saturated with graphite, and vapour. The corresponding equilibrium mixture of chromium carbide, chromium, and graphite boils at 2270° C., the vapour consisting entirely of chromium. Between 2250° and 2050° C. the melt consists of  $Cr_3C_2$ . Other carbides shown to be formed are  $Cr_4C_2$  and  $Cr_5C_2$ .—J. F. S.

"Stellite" [cobalt-chromium alloy]. L. Guillet and H. Godfroid. Rev. Mét., 1918, 15, 339–346.

"STELLITE" (this J., 1915, 1058) is a complex alloy of carbon, cobalt, chromium, tungsten, and, at times, molybdenum, etc., used for tools. The Brinell hardness in the cold is 495, which falls to 332 at 800° C. A good high-speed steel had a hard-

ness of 652 at ordinary temperatures, which had fallen to 112 at 800° C. Reheating "stellite" to temperatures up to 800° C. does not affect the hardness after cooling again. The microstructure in the cast condition shows the typical dendritic pattern which is not modified by reheating the material to 1150° C. and quenching it. When heated for 6 hours at 1000° C., the dendritic structure is obliterated. The following analyses illustrate the variations which are found in the composition of the alloy:—

Fe	C	Si	Mn	S	P	Co	V	Cr	W	Mo
10.00	1.79	0.78	0.72	—	—	34.6	1.0	26.4	12.7	9.4
—	1.48	0.17	—	0.01	traces	55.6	—	33.6	9.2	—

In the lathe tools made of "stellite" act at least as well as most of the high-speed steels on the market, at any rate on the softer materials; it is, however, somewhat uneven and the results obtained are not uniform.—F. C. Th.

*Sulphide precipitation of group 2a metals.* J. Shibko. Chem. News, 1918, 117, 253.

In the analysis of a brass or bronze the amount of copper found, even when the tin and the lead had been previously removed, was always slightly higher with ammonium sulphide as precipitant than with hydrogen sulphide, the solution being kept slightly acid. The error is due to small quantities of zinc brought down with the copper. A 60:40 brass, after removal of tin and lead, gave with hydrogen sulphide, Cu 59.86, Zn 0.0, with ammonium sulphide, Cu 60.12, Zn 0.25%. (See also J. Chem. Soc., Sept., 1918.)—C. S.

*Comparison of gas producers from which slag is tapped off with those of the water-seal type.* Markgraf. See IIA.

*Preparation of alumina in the aluminium industry with special reference to rotary kilns.* Von Escher. See VII.

*Paving bricks from blast-furnace slag.* Shaw See IX.

#### PATENTS.

*Blast furnaces; Production of ammonium chloride during the working of* —. A. Riedel, Koessern, Saxony. Eng. Pat. 102,146, Oct. 28, 1916. (Appl. No. 15,385 of 1916.) Under Int. Conv., Nov. 8, 1915.

In the zone of the blast furnace in which ammonia may be produced (500°–900° C.) no water vapour is present to aid this formation, all materials in normal working having been thoroughly dried in the upper part of the furnace. A uniform supply of water vapour is obtained by the introduction of a highly hydrated chloride with the furnace charge. The salt should be one which only parts with its water of crystallisation at a temperature at which ammonia is formed. The chlor-ion possesses the property of protecting ammonia from dissociation at even higher temperatures than water vapour does, and the conversion of fixed nitrogen into free nitrogen is restricted. Of the chlorides available, calcium chloride offers special advantages as the lime produced allows of a diminution of the normal quantity of lime in the blast-furnace charge.

—C. A. K.

*Steel; Manufacture of* —. H. A. Greaves and H. Etchells, Sheffield. Eng. Pat. 117,286, June 8, 1917. (Appl. No. 8175 of 1917.)

STEELS containing from 2 to 15% Mn and not more than 0.40% C may be made in the electric furnace



by adding ferromanganese to the liquid bath of metal and oxidising the excess carbon by manganese oxides, by adding previously refined ferromanganese, or by adding manganese ore, together with finely divided carbon, to the slag covering the steel. In plain carbon steels the addition produces metal from which plates with a very smooth surface, suitable for ship plates, may be obtained, and also tough, non-magnetic material for electrical machinery. In alloy steels a substitution of the cheaper manganese for the special elements produces economies and provides new special steels for armour and armour-piercing shells.—F. C. Th.

*Zinc-bearing materials; Refining metallic — by the electrolytic process.* Electrolytic Zinc Co., Inc., New York, Assignees of C. H. Aldrich and J. K. Bryan, Colgate, Md., U.S.A. Eng. Pat. 108,312, July 12, 1917. (Appl. No. 10,073 of 1917.) Under Int. Conv., July 26, 1916.

ZINC-BEARING minerals are refined electrolytically by the use of an acid electrolyte, containing from 0.1 to 0.5% of free acid, at about 40° C. or lower. The anode slimes are maintained substantially in contact with the anode, by means of a bag or the like surrounding the latter, so as to keep the more electro-negative metals, such as cadmium, from the disintegrated anode in contact with the excess of zinc in the anode, and thus prevent solution of the more electro-negative metal. If cadmium passes into the solution, it may be removed by passing the electrolyte through a number of tanks in series, provided with insoluble anodes, and after removal of the cadmium the electrolyte is returned to the main circuit.—B. N.

*Separating metals [lead and zinc] from their ores; Process for —.* A. L. J. Queneau, Newcastle-on-Tyne. Eng. Pat. 116,943, July 4, 1917. (Appl. No. 9674 of 1917.)

THE CRUSHED ore containing lead and zinc sulphides is mixed with excess of molten chloride of zinc and sodium (60%  $ZnCl_2$  and 40% NaCl). Metallic zinc is then added to the hot mass to the extent of one-third by weight of the lead present, and the lead bullion separated and leached with water to remove the chlorides and the zinc sulphide formed. A flotation process allows the latter to be separated in a form which will yield a very high-grade zinc. —F. C. Th.

*Crucible furnaces.* H. T. Lancey, Southampton. Eng. Pat. 116,946, July 5, 1917. (Appl. No. 9756 of 1917.)

TO FACILITATE the slagging out of crucible furnaces of the tilting type, the bottom of the furnace is made as a pair of concave doors hinged to the furnace wall or fastened on by cotters. These doors can be opened and slag readily removed. To prevent spilled metal, etc., from corroding the iron, this is covered with a refractory lining of firebrick, etc. Loose trays may be inserted into the furnace above the doors and removed and replaced by new ones when full of slag.—F. C. Th.

*Crucible furnaces; Tilting —.* J. Gaunt, D. Brookfield, and J. Tylor and Sons, Ltd., London. Eng. Pat. 117,054, Sept. 27, 1917. (Appl. No. 5152 of 1918.)

CRUCIBLE furnaces of the pattern described in Eng. Pats. 114,684 and 115,222 (this J., 1918, 308 A, 426 A) are mounted on an axis and tilted by means of a chain or wire rope attached to a winding drum. A balance weight may also be used to facilitate the operation.—C. A. K.

*Crucible furnaces for melting metals.* T. W. Aitken, Luton. Eng. Pat. 117,180, Sept. 17, 1917. (Appl. No. 13,306 of 1917.)

GREATER protection is afforded to the hearths of crucible furnaces, particularly of the type described in Eng. Pat. 107,468 of 1916 (this J., 1917, 967), by covering the floor of the furnace with a layer of sand, and by the provision of front and back doors above the sand covering, or of a tapping hole at this level.—C. A. K.

*Furnace rakes for mechanical roasting furnaces.* E. E. Brand, Helsingborg, Sweden. Eng. Pat. 116,974, Sept. 1, 1917. (Appl. No. 12,592 of 1917.)

THE blades of the rakes are inserted directly in longitudinal grooves in the radial arms fixed to a central vertical rotary shaft. The blade, which may be plane, bent, or curved, forms an obtuse angle with the furnace bottom and is also inclined in relation to the longitudinal direction of the supporting arm.—F. C. Th.

*Heat treatment of metals; Gas heated and similar furnaces for —.* C. R. Inman, Sheffield. Eng. Pat. 117,301, July 12, 1917. (Appl. No. 10,064 of 1917.)

EXTENDING longitudinally below the floor of the furnace is a combustion chamber into which nozzles for the supply of a mixture of gas and air pass. These nozzles are fitted with flanges below the floor of the chamber to prevent access of air and may be removed for inspection or cleaning. The hot gases from the combustion chamber pass up side flues into the furnace and escape through holes in the centre of the arched roof into a clear space above the furnace which they heat up before passing to the chimney. The heating is claimed to be rapid and economical, and since all the openings can be readily closed, the furnace can retain its heat for a long time.—F. C. Th.

*Waste metal; Apparatus for recovery of —.* G. W. Wardle, Birmingham. Eng. Pat. 117,321, July 31, 1917. (Appl. No. 10,990 of 1917.)

WHEN pouring aluminium or other metal covered with slag or dross, the metal remaining in the latter at the end of pouring may be caused to collect and separate from the impurities by the action of superheated steam at 300° F. (150° C.). The best means for doing this is to pour the dross and metal downwards through a hollow cone formed by superheated steam issuing from a series of holes in the under side of a circular pipe, the holes being directed inwards.—F. C. Th.

*Metal; Apparatus for extracting — from ores.* J. N. Lewis, Assignor to W. L. Wilson, Detroit. Mich. U.S. Pat. 1,265,459, May 7, 1918. Date of appl., Oct. 28, 1916. Renewed Mar. 8, 1918.

A CONTAINER for the solvent and ore is provided with a combined filtering and precipitating unit, fitted as a removable portion of the wall. Fibrous material forms the filtering medium and this is clamped to a porous container for the precipitating agent.—C. A. K.

*Annealing and other furnaces; Means for feeding goods into and removing goods from —.* Gibbons Bros., Ltd., and M. van Marle, Lower Gornal, Staffs. Eng. Pat. 116,949. (Appl. Nos. 9924, July 10, 1917, and 2283, Feb. 8, 1918.)

*Crucible furnaces and the like; Operating mechanism of tilting* —. Aldays and Onlons Pneumatic Engineering Co., Ltd., A. Nicholson, and H. Hodkinson, Birmingham. Eng. Pat. 117,212, Nov. 29, 1917. (Appl. No. 17,660 of 1917.)

*Distilling ores and metal-bearing products; Apparatus for* —. D. B. Jones, Chicago, Assignee of C. H. Fulton, St. Louis, U.S.A. Eng. Pat. 111,835, Aug. 10, 1917. (Appl. No. 11,497 of 1917.) Under Int. Conv., Dec. 7, 1916.

SEE U.S. Pat. 1,242,337 of 1917; this J., 1917, 1240.

[Copper] *slags; Recovery of values from metalliferous* —. J. B. Herreshoff, jun., New York. Eng. Pat. 117,470, Apr. 14, 1917. (Appl. No. 5298 of 1917.)

SEE U.S. Pat. 1,231,319 of 1917; this J., 1917, 929.

*Zinc; Method of converting zinc powder into liquid* —. S. Hultdt, Stockholm, Sweden, Assignor to Norsk Elektrisk Metallindustri Aktieselskap, Sarpsborg, Norway. U.S. Pat. 1,256,808, May 21, 1918. Date of appl., Apr. 3, 1917.

SEE Eng. Pat. 105,558 of 1917; this J., 1918, 271 A.

*Apparatus for separating dust from ores, coal, and the like.* Eng. Pat. 113,780. See I.

*Slag-heated steam generator.* U.S. Pat. 1,267,022. See I.

*Hardness testing machines.* Eng. Pat. 117,526. See XXIII.

## XI.—ELECTRO-CHEMISTRY.

*Ceric oxychloride produced by the electrolysis of cerous chloride.* Arnold. See VII.

*Anodic formation of esters, simultaneously with nitrobenzene and o-nitrophenol from mononitrobenzoic acids.* Schall. See XX.

*Method for making electrometric titrations of solutions containing protein.* Baker and Van Slyke. See XXIII.

### PATENTS.

*Separators or non-conducting supports for accumulator plates.* H. Lefluer, London. Eng. Pat. 117,121, July 3, 1917. (Appl. No. 9573 of 1917.)

Wood separators, to be used as supports for accumulator plates, are subjected to the action of sodium chloride solution, which has been or is being electrolysed to form chlorine, hypochlorites, etc., and are then treated with dilute sulphuric acid.—B. N.

*Electrode for use in electric furnaces and for other purposes.* H. Nathusius, Kreis Beulhen, Germany. U.S. Pat. 1,268,483, June 4, 1918. Date of appl., July 19, 1916.

Pieces of metal are inserted in a mass of dolomite and after adding suitable binders and substances containing carbon, the mass is stamped in moulds, baked, and the metal melted by means of an electric current.—J. H. P.

*Electric insulating materials and the like; Manufacture of* —. W. E. W. Richards, London. U.S. Pat. 1,267,696, May 28, 1918. Date of appl., Dec. 22, 1915.

SEE Eng. Pat. 3364 of 1915; this J., 1916, 608.

*Electric glass furnace.* U.S. Pat. 1,267,317. See VIII.

*Method and apparatus for treating grain [with ozonised air].* Eng. Pat. 116,952. See XIXa.

## XII.—FATS; OILS; WAXES.

*Fatty oils of Sambucus racemosa, L.* H. J. Zellner. Monatsh. Chem., 1918, 39, 87–94.

The European elder, *Sambucus racemosa, L.*, and the North American *Sambucus racemosa, var. arborescens*, although very similar, exhibit distinct but slight differences in nature; this probably accounts for the difference between the characteristics of the oil from the flesh of the red berries of these varieties (Zellner, this J., 1903, 101, and Byers and Hopkins, this J., 1902, 1238, respectively). The oil from the former when kept for 15 years in a stoppered flask undergoes marked alteration with increase in the proportion of free acid and decrease in the iodine value. The elder seeds also yield an oil, of sp. gr. 0.931 at 15° C.,  $[\alpha]_D^{20} = +1.485$ , saponification value 190.8, and iodine value (Höbl), 162.0; in its drying properties and other characteristics this oil resembles linseed oil.—D. F. T.

*Cholesterol and the higher fatty acids; Nephelometric values of* —. F. A. Csonka. J. Biol. Chem., 1918, 34, 577–582.

The turbidities produced by the addition of acid to solutions of cholesterol and various soaps differ in intensity and stability. Estimations of fat by nephelometric methods are therefore open to error unless the nature of the fat is known and allowed for in calculating the results. (See also J. Chem. Soc., Sept., 1918.)—H. W. B.

*Dubrisay's method of testing [viscosity of] lubricating oils.* Nicolardot and Masson. See IIa.

### PATENTS.

*Hydrogenation; Process of* —. C. Ellis, Montclair, N.J. U.S. Pat. 1,266,782, May 21, 1918. Date of appl., Nov. 23, 1914.

The material to be hydrogenated, together with hydrogen, is subjected to the action of a catalytic body comprising nickel and silica, both in a non-abrasive, e.g. colloidal, condition, and associated with another metal having catalytic properties, e.g., a metal of the palladium group.—A. de W.

*Oils and the like; Method of extracting* —. R. Wells, Homer, N.Y., Assignor to Cobwell Corporation, Cleveland, Ohio. U.S. Pat. 1,267,611, May 28, 1918. Date of appl. Mar. 11, 1915. Renewed Oct. 22, 1917.

To extract oils or the like from a material also containing water, the material, in a relatively thin layer, is immersed in a water-insoluble solvent having a higher boiling point than water. Means are provided for moving an arm underneath the layer of material in order to impart an undulatory



movement thereto without bodily moving the same, whilst simultaneously stirring its upper surface. The temperature of the mass is then raised to the joint boiling point of the solvent and the water and as evaporation of both proceed, more solvent is added. After expelling water from the material in this way, the oil or the like material is extracted by means of a solvent of lower boiling point than water.—A. de W.

*Catalysts [for hydrogenation of oils]; Preparation of* — J. Dewar, London, and A. Liebmann, Weybridge, Assignors to The Procter and Gamble Co., Cincinnati, Ohio. U.S. Pat. 1,268,692, June 4, 1918. Date of appl., June 2, 1914.

SEE Eng. Pat. 12,981 of 1913; this J., 1914, 797.

*Method of determining the viscosity of fluids.* Eng. Pat. 117,234. See XXIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Resins; Constituents of* — I. Siarensinol from Siamese gum benzoin. A. Zinke and H. Lieb. Monatsh. Chem., 1918, 39, 95–106.

On treatment with hot sodium hydroxide solution Siamese gum benzoin yields the sodium derivative,  $C_{30}H_{47}O_2Na \cdot SH_2O$ ,  $[\alpha]_D^{21} = +34.97^\circ$ , of siarensinol, from which siarensinol,  $C_{30}H_{48}O_4$ ,  $[\alpha]_D^{22} = +27.31^\circ$  (in methyl alcohol) is liberated by hydrochloric acid, this substance being probably identical with the benzoresinol of Lüdy (this J., 1893, 946; compare Reinitzer, this J., 1915, 681). With acetic acid siarensinol yields an additive compound,  $C_{30}H_{48}O_4 \cdot C_2H_4O_2$ , crystallising in prisms, m.pt.  $288^\circ - 281.5^\circ C.$ , whilst the sodium derivative reacts with benzoyl chloride giving a benzoyl derivative,  $C_{37}H_{52}O_5$ , crystallising in needles, m.pt.  $182^\circ - 183^\circ C.$ ,  $[\alpha]_D^{20} = +30.0^\circ$  in ethyl alcohol.—D. F. T.

#### PATENTS.

*[Titanium dioxide] pigments; Manufacture of white or light coloured* — B. E. D. Kilburn, London. From Det Norske Aktieselskab for Elektrokemisk Industri, Norsk Industri-Hypotekbank, Christiania. Eng. Pat. 110,535, May 8, 1917. (Appl. No. 7214 of 1917.)

A WHITE pigment of high opacity combined with an absence of externally crystalline outline in the composing particles such as is possessed by pulverised titanium dioxide crystals of high refractive index, e.g., rutile, although of internally crystalline structure, is obtained by converting an amorphous titanium oxygen compound, such as titanium dioxide or titanic acid hydrate, into an internally crystalline titanium dioxide by means of heat. A halogen compound may be added to act as a catalyst of the crystallisation process. The progress of the crystallisation on heating may be determined by observing the increasing double refraction of the particles under the microscope between crossed nicols. The titanium pigment may also if desired be associated with barium sulphate and/or calcium sulphate.—A. de W.

*Carbon-pigment base and process of making same.* D. J. Ogilvy, Cincinnati, Ohio. U.S. Pat. 1,268,142, June 4, 1918. Date of appl., July 21, 1916.

CARBON pigments for printing inks, paints, or the like are obtained by impinging a carbonaceous flame against a cool high "fire test" oily surface until the oil is charged with carbon.—A. de W.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Sulphur in soft rubber compounds; Determination of free* — H. S. Upton. J. Ind. Eng. Chem., 1918, 10, 518–520.

Two grms. of the rubber compound is extracted with acetone until all the free sulphur is dissolved, the acetone solution is then evaporated, and the residue dried at  $100^\circ C.$  This residue is boiled for 1 hour with 50 c.c. of 5% alcoholic potassium hydroxide solution, then cooled, and treated with 50 c.c. of ammonium zinc chloride solution (10 grms. of zinc oxide dissolved in hydrochloric acid, neutralised with ammonia, 50 c.c. of ammonia of sp. gr. 0.9 added in excess, and the solution diluted to 1 litre), neutralised with acetic acid, 3 c.c. of the latter is added in excess, the mixture is diluted to 200 c.c., and titrated with N/20 iodine solution; the titration gives the quantity of sulphur which has been converted into thiosulphate. The portion of sulphur converted into sulphide is found by treating the titrated solution with ammonia, then acidifying with hydrochloric acid, and again titrating the mixture with iodine solution. The sum of the two titrations is a measure of the free sulphur originally present. A correction for other reducing substances is found by treating similarly a rubber compound of the same character but containing no free sulphur.—W. P. S.

#### PATENTS.

*Rubber; Compounds for use in lieu of* — J. Flint, Rushcutters Bay, N.S.W. Eng. Pat. 105,912, Apr. 10, 1917. (Appl. No. 5012 of 1915.) Under Int. Conv., Mar. 21, 1916.

SEE U.S. Pat. 1,233,459 of 1917; this J., 1917, 1019.

*Recovery by condensation of volatilised solvents.* Eng. Pat. 116,590. See I.

### XV.—LEATHER; BONE; HORN; GLUE.

*Gelatin; Influence of neutral salts, bases, and acids on the precipitation of* — by alcohol. J. Loeb. J. Biol. Chem., 1918, 34, 489–501. (Compare this J., 1918, 383 A, 384 A.)

GELATIN powder which has been treated with salt solution and, after washing, dissolved in water, is not reprecipitated by alcohol when the salt employed contains univalent anions and cations above M/128 concentration. For salts with divalent cations, the limiting concentration is M/256. The anion is without action. These and similar results are explained by the assumption that salts of gelatin with univalent cations are readily ionised, whilst those with divalent cation are less or not at all ionisable. The anion of the salt behaves as if it did not combine with the gelatin at all.—H. W. B.

#### PATENTS.

*Glue or gelatin; Manufacture of substances capable of precipitating* — J. Y. Johnson, London. From Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 116,933, June 29, 1917. (Appl. No. 9380 of 1917.)

WATER-SOLUBLE products capable of precipitating gelatin and having valuable tanning properties are prepared by treating with sulphonating agents a naphthylsulphone (dinaphthylsulphone or its derivatives containing at least one naphthalene residue free from hydroxyl) which can be produced from naphthalene by suitable treatment with sulphuric

acid. The products so obtained may be condensed with formaldehyde, when useful tanning substances are produced, or skins treated with formaldehyde may be tanned by the original substances.—F. C. T.

*Tanning, and manufacture of agents to be used therein.* J. Y. Johnson, London. From Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 116,934, June 29, 1917. (Appl. No. 9381 of 1917.)

EASILY soluble condensation products capable of tanning leather are obtained by heating together a phenolsulphonic acid and a sulphonated hydrocarbon such as benzenesulphonic acid or "sulphonated rock oil." For example, the product obtained by heating Caucasian "naphtha" with sulphuric acid at 100° C., is mixed with phenol-sulphonic acid and heated for 20 hours at 130° C. The product is diluted with water, filtered, and concentrated.—F. C. T.

*Tanning.* J. Y. Johnson, London. From Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. Eng. Pat. 116,935, June 29, 1917. (Appl. No. 9382 of 1917.)

HIDES or skins may be tanned by aqueous solutions of sulphonic acids of aromatic hydrocarbons, or sulphonated derivatives of these, not containing hydroxyl groups, e.g., sulphonic acids of anthracene, phenanthrene, etc., and their derivatives. Such sulphonic acids used singly or in mixture can be used for tanning either with or without the addition of natural tanning materials. The dark colour produced by some natural tanning materials can be cleared by the use of sulphonic acids.

—F. C. T.

*Hides, skins, and leather; Machines for treating* —. J. W. H. Hall, Leeds. Eng. Pat. 117,358, Oct. 9, 1917. (Appl. No. 14,621 of 1917.)

*Process of making fertiliser [from leather].* U.S. Pat. 1,268,683. See XVI.

## XVI.—SOILS; FERTILISERS.

*Protozoa in the soil; Method for the counting of certain* —. A. Itano and G. B. Ray. Soil Sci., 1918, 5, 303—310.

A WHIPPLE cell (compare "Microscopy of Drinking Water," 1914, p. 34) was thoroughly cleaned with water and then with xylene. The bottom of it was covered by means of a platinum loop with a thin layer of phenolsulphonaphthalein solution, and when this had dried, the culture, previously diluted with a 4% gelatin solution, adjusted to P<sub>h</sub> 8, was run in as described by Whipple (*loc. cit.*). The number of protozoa was then counted and from these data the total number of organisms per c.c. of suspension was calculated. The soil culture was prepared by adding 5 grms. of soil to 100 c.c. of a mannitol solution, gently shaking, and allowing to stand at the ordinary temperature for 2 or 3 weeks. The results indicate that the use of a vital stain aids materially in defining the organism. The results are seriously affected when the soil is allowed to settle, but rapid motion is inhibited by the use of the semi-solid diluent. The probable error seems to vary inversely with the size of the organisms and directly with the magnification.

—W. G.

*Sulphofication in relation to nitrogen transformations [in soils].* J. W. Ames and T. E. Richmond. Soil Sci., 1918, 8, 311—321.

OXIDATION of free sulphur in soils devoid of bases depresses the activities of nitrifying organisms, and even when calcium carbonate is added in excess of the lime requirement of the soil the yield of nitric nitrogen from casein is considerably decreased by oxidation of added sulphur. The depression increases as the acidity decreases. There is an increase of ammonia as the yield of nitrates decreases when sulphur is oxidised, this being due probably to the deficiency of calcium carbonate necessary for the neutralisation of the sulphuric acid formed. The ammonia formed neutralises the sulphuric acid and remains as ammonium sulphate.

—W. G.

*Sulphur: Oxidation of — by micro-organisms in its relation to the availability of phosphates.* H. C. McLean. Soil Sci., 1918, 5, 251—290.

THE most economical combination for the production of available phosphoric acid from rock phosphate appears to be 100 parts of soil, 120 parts of sulphur, and 400 parts of rock phosphate, although an appreciable amount of phosphoric acid is rendered available even if the soil is omitted. Of the different "floats" tried, Florida soft rock phosphate gave the best yield of available phosphoric acid. Fineness of grinding is an advantage provided that it is not carried to such an extent as to inhibit biological activities owing to lack of aeration. Micro-organisms are influential in the oxidation of sulphur and the rendering available of the phosphorus in rock phosphate, and as these organisms are apparently mainly aerobic, aeration is of great importance. Of the various catalysts tried, the sulphates of ammonium, calcium, magnesium, and zinc exerted no influence on the process, whilst sodium nitrate, potassium iodide, and copper sulphate exerted a depressing action on the organisms. When present in small amounts (0.4 lb. per ton) in composts, ferrous sulphate and aluminium sulphate or a mixture of the two, exerted a marked stimulating action on sulphur oxidation processes and on the rendering available of the phosphorus. The presence of organic substances such as peat, fresh horse manure, old composted manure, or peptone is not desirable, and calcium carbonate brings about a decrease in available phosphorus when added to a sulphur-rock phosphate-soil compost.—W. G.

*Nitrogen in nitrate of soda, ammonium sulphate, dried blood, and farm manures; Twenty years' work on the availability of —.* J. G. Lipman and A. W. Blair. Soil Sci., 1918, 5, 291—301.

OVER a period of twenty years in a five-course rotation sodium nitrate at the rate of 320 lb. per acre per annum gave better crop yields than an equivalent amount of either ammonium sulphate or dried blood. During the first ten years the two latter fertilisers gave results about on a par with those from the former, but after that they showed a considerable falling off. The average nitrogen recovery in the crops over the twenty years from each fertiliser was: sodium nitrate, 62.42%; ammonium sulphate, 47.48%; dried blood, 38.69%; cow manure, 32.69%. Cow manure at the rate of 16 tons per acre gave somewhat larger crop yields than sodium nitrate at the rate of 320 lb. per acre, but the increased yields were not sufficient to justify the increase in the cost of nitrogen.—W. G.



*Liquid manure; Decomposition and preservation of* —. E. Blanek. Landw. Versuchs-Stat., 1918, 91, 253—269, 271—290, 309—343.

Liquid manure (urine) decomposes rapidly when applied to soils, the loss of nitrogen depending on the rate of evaporation of the liquid and on bacterial action. Treatment of the liquid manure with sulphuric acid prevents loss of nitrogen in so far that it fixes, up to a certain limit, the ammonia formed. Formaldehyde preserves the urine, but its use is not recommended, as it interferes with plant growth.—W. P. S.

*Dicyanodiamide; Constitution of* —. W. J. Hale and F. C. Vibrans. J. Amer. Chem. Soc., 1918, 40, 1046—1063.

From the facts that (1) three-fourths of the nitrogen in dicyanodiamide can be expelled by 20% sodium hypochlorite solution, (2) nitrous acid, acting on a solution of dicyanodiamide in aqueous acetic or phosphoric acid at the boiling point, expels from one to four atoms of nitrogen according to the concentration of the acid, (3) the condensation of dicyanodiamide and acetylacetone by aqueous sodium hydroxide yields 2-imino-1-cyano-4,6-dimethyl-1,2-dihydropyrimidine,  $C_8H_8N_4$ , colourless needles, m.p. 225° C., the authors are of opinion that the constitution of dicyanodiamide must be represented by the cyanoguanidine structure,  $NH_2C(:NH).NH.CN$ , originally proposed by Bamberger (Ber., 1883, 16, 1074). (See also J. Chem. Soc., 1918, i., 350.)—C. S.

*Superphosphate; Use of nitre-cake in the manufacture of* —. 51th Annual Report on Alkali Works for 1917 by the Chief Inspector, 19—20.

A SUCCESSFUL process is in use for the manufacture of superphosphate from Tunisian phosphate and nitre-cake. The nitre-cake is ground in an ordinary disintegrator and if ground phosphate is gradually mixed with it during the grinding clogging of the machine is obviated. It has been found necessary to add to the mixing about 10% of hot water more than is present when sulphuric acid of 118° Tw. (sp. gr. 1.59) is used. Comparatively little heating or evolution of gas occurs. Analysis of the product (after two weeks) gave 16.36% of water-soluble phosphate.—C. A. K.

*Seeds; Prejudicial effects of treatment with formalin upon the germination of* —. L. Kiessling. J. Landw., 1918, 66, 7—51.

It has been customary to treat seed corn with formaldehyde solutions to get rid of bunt in oats and rust in wheat, and this treatment has been observed to influence germination. Culture and germination experiments extending over two years showed that the immersion of seed in a solution containing 0.1 per 1000 of formaldehyde for a quarter of an hour retarded the onset of germination and also the total time of germination, although the number of plants obtained was about the same as from untreated seed. When artificially damaged seeds were submitted to treatment, they appeared to undergo no greater injury than sound seeds. Some of the injurious effects were due to the presence of impurities, such as methyl alcohol, in the solutions used, which are customarily prepared from formalin, and it was proved that it would be better to employ pure solutions prepared from paraformaldehyde. The use of formalin to protect seeds against mildew is not to be recommended, unless a pure solution of formaldehyde is used or part of the formalin is replaced by another fungicide.—J. H. J.

*Determination of ammonia in urine, serum, etc.* Wiessmann. See XXIII.

#### PATENTS.

*Fertiliser purposes; Process of treating organic matter for* —. A. J. Grinberg, New York, and B. Field, Plymouth, Mass. U.S. Pat. 1,268,563, June 4, 1918. Date of appl., Apr. 27, 1917.

ORGANIC matter is treated with acid producing and denitrifying bacteria, with or without the addition of a phosphate compound and a potash compound, and the whole allowed to "work" by growth of bacteria and then dried.—W. G.

*Fertiliser; Process of making* — [from leather]. D. E. Clark, Lynn, Mass. U.S. Pat. 1,268,683, June 4, 1918. Date of appl., July 18, 1917.

THE fertiliser is produced by treating leather with the liquor known as "dunder."—W. G.

*Fertiliser.* H. Blumenberg, jun., Oro Grande, Cal., Assignor to Golden State Portland Cement Co., Los Angeles, Cal. U.S. Pat. 1,270,688, June 25, 1918. Date of appl., Mar. 4, 1918.

THE fertiliser consists of calcium carbonate, 3 parts, and calcium sulphate, 1 part.—W. G.

*Phosphate manure; New soluble and assimilable — and process for making it.* E. Stoppani, Bologna, and V. Volpato, Milan, Italy. U.S. Pat. 1,267,473, May 28, 1918. Date of appl., Oct. 5, 1916.

SEE Fr. Pat. 480,697 of 1916; this J., 1917, 152.

#### XVII.—SUGARS; STARCHES; GUMS.

*Dextrose; Rate of production of colour in alkaline solutions of — and pierate.* T. Addis and A. E. Shevsky. J. Biol. Chem., 1918, 35, 43—51.

THE results show that under the conditions described in most methods for the colorimetric estimation of dextrose by pierate and alkali, an increase in the concentration of dextrose does not lead to an exactly proportionate increase in colour. The most satisfactory conditions are found to be a concentration of 10% of sodium carbonate and the heating continued for 45 minutes at 100° C. in the presence of 0.8% of pieric acid.—H. W. B.

*Starch; Bulrushes as a source of* —. L. Kofler. Z. Unters. Nahr. Genussm., 1918, 35, 266—272.

ATTENTION is directed to the high starch content of bulrush roots, the quantity present being about 46% of the dry substance. The powdered root has a characteristic appearance under the microscope and the starch grains are of two sizes, the larger being about 13  $\mu$  and the smaller 3.5  $\mu$  in diameter.—W. P. S.

*Law of action of sucrase [invertase].* Colin and Chaudun. See XVIII.

#### PATENTS.

*Sugar cane; Process for extracting sucrose from* —. A. Adams, Kahuku, Hawaii. U.S. Pat. 1,265,582, May 7, 1918. Date of appl., Aug. 25, 1915.

THE cane, after passing through a crusher and shredder, is conveyed to a diffusion cell. The juice

from the shredder passes to a liming tank and thence through a heater to the cell. The bagasse from the cell is conveyed to a mill and the juice from the latter can be returned to the liming tank and diffusion cell.—J. H. L.

*Bagasse; Method of treating — for the recovery of its values.* M. W. Marsden, Assignor to Sugar Cane By-Products Co., Philadelphia, Pa. U.S. Pat. 1,265,694, May 7, 1918. Date of appl., Nov. 11, 1915.

BAGASSE is reduced to a uniformly divided state and freed from all but about 10% of its natural moisture-content. The pith is then separated from the fibre and ground to a flour, which after being boiled with water and washed, is obtained in the form of a paste. This paste may be employed for the production of glucose.—J. H. L.

*Glue or adhesive; Vegetable —.* R. W. Tunnell, Philadelphia, Pa. U.S. Pat. 1,266,354, May 14, 1918. Date of appl., Nov. 9, 1917.

AN adhesive is formed from hydrolysed starch and zinc chloride, with or without an excess of caustic alkali; e.g., zinc chloride may be added to a viscid mixture of starch, water, and caustic alkali.

—J. H. L.

## XVIII.—FERMENTATION INDUSTRIES.

*Barleys; Institute of Brewing method for the determination of the moisture-content of —.* Committee of the Institute of Brewing. J. Inst. Brew., 1918, 24, 234.

THE following standard method for determining moisture in barley has been adopted by the Institute. About 10 grms. of the well mixed sample is ground as finely as possible in a coffee mill previously rinsed with the same sample. 4–5 grms. is weighed out in a weighing bottle about 2 ins. in diameter and 1 in. high, provided with a cover, and this is then placed in a boiling water oven for 5 hours, the cover resting on the top of the oven meanwhile. After 5 hours, the vessel is covered, transferred to a desiccator and weighed as soon as cool. The percentage moisture-content is calculated to the nearest first decimal. The oven should contain nothing but barley samples, and these should be placed not on the shelf but on the floor of the oven, and not close to the door. The oven door must not be opened during the drying. The ventilator in the door must remain open and the orifice at the top of the oven kept free. To prevent furring, it is advisable to use distilled water for heating the oven. With each batch of barleys a determination should be made on a stock sample, as control. Careful adherence to the method should ensure agreement to within 0.5%.—J. H. L.

*Barley; Determinations of acidity in — by titration in stages.* A. Relehard. Z. ges. Brauw., 1918, 41, 57–60, 65–68, 75–77, 83–81, 89–90.

IN continuation of previous work (this J., 1917, 399), aqueous extracts of barley were made, under conditions excluding enzyme action, and titrated with alkali, first in presence of litmus and then in presence of phenolphthalein. The most satisfactory method of preventing enzymic action was found to consist in heating the ground barley with alcohol (cp. Lüers and Adler, this J., 1915, 1158), and evaporating off the latter, after which the material was digested with cold water for at least 3 hours, and titrated. With freshly harvested barley the acidity determined in presence of phenolphthalein was much greater than that determined

in presence of litmus, sometimes twice as great. During storage of the grain the former value gradually diminished and finally coincided with the latter. This diminution of the "phenolphthalein acidity" is attributed to the disappearance of amino-acids (acid towards phenolphthalein but not towards litmus), a change which, according to Schjerring (this J., 1906, 1109) and others, is characteristic of the process of "after-ripening" of barley. The identity of the "litmus acidity" and the "phenolphthalein acidity" might be regarded as a sign that barley is completely matured and ready for malting. In exceptional cases, however, barleys which do not conform with this analytical standard give good results on germination.—J. H. L.

*Brewing material; Sudan durra as a —.* I. Briant and H. Harman. J. Inst. Brew., 1918, 24, 209–214.

DURRA forms the staple food of the population of the Sudan and is the subject of a growing export trade. At the suggestion of the Imperial Institute, the authors had placed at their disposal 5–6 tons of the material for the purpose of investigating its suitability for brewing purposes. The composition of a sample of durra analysed at the Imperial Institute was as follows:—moisture 8.45%, proteins 13%, fat 3.30%, fibre 1.03%, ash 1.71%, carbohydrates (by difference) 72.45%; nutrient ratio 1:6.1; food units 113.3. With the collaboration of various manufacturers, large-scale experiments were made on the conversion of the material into malt, roasted grain, flaked products, and glucose. The results of the maltings were not promising, for although the product was moderately friable and possessed a fair diastatic power, it yielded only about one-third as much extract as barley malt. By roasting the grain, products were obtained having a satisfactory aroma and flavour, comparing favourably with those of roasted barley. Experimental brews for porter showed that 7 parts of roasted durra may replace 10 parts of roasted barley without interfering with the character of the beer. The malting of the grain before roasting is not recommended. Amber durra malt of fine flavour was prepared, but its yield of extract was very low. Satisfactory flaked products were prepared from durra without difficulty. Their appearance was not so attractive as that of flaked maize or rice, and the flakes, being rather brittle, would probably produce a considerable amount of dust during transport which might interfere with mash-tun drainage. In the flaking process about 15% of meal was produced. This contained about two-thirds of the oil in the durra seed, leaving only about 1.3% in the flakes; it would constitute an excellent cattle food. The whole of the starch in the durra appeared to be rendered available by flaking. The character of the extract was quite satisfactory, closely resembling that from flaked rice: 336 lb. yielded 94.4 lb. of extract, as compared with 100–102 lb. from flaked maize. Replacement of half the flaked maize (15%) by flaked durra, in the preparation of bitter beer or pale ale, led to no considerable defect in the products, and total replacement would have been quite practicable. Flaked durra might prove excellent for direct human consumption. Good brewing sugar, for use in the copper or as priming of the malto-dextrin type was prepared from durra.—J. H. L.

*Hop extracts; Colour of — and the influence of the salts of water thereon.* H. Krumhaar. Woch. Brau., 1918, 35, 25–27.

BAWERY worts made with waters rich in carbonates sometimes assume a reddish tint after



boiling with hops and cooling, and the tint becomes less pronounced in the course of fermentation. From the author's experiments it is concluded that, at the same concentration, calcium and magnesium bicarbonates are equally active in producing this coloration, that the normal carbonates do not produce it, and that it is due to combination of the bicarbonates with certain hop constituents.

—J. H. L.

*Hop extracts; Coloration of* —. A. Bau. Woch. Brau., 1918, 35, 73–74, 79–83, 90–92. (See preceding abstract.)

EXPERIMENTS in which water containing magnesium bicarbonate was boiled with hop extract, indicated that the reaction to which the colour is due is not instantaneous but requires about an hour's boiling, that the presence of air is necessary for the colour to develop, and that addition of gypsum in amounts corresponding with brewing practice does not prevent the coloration. In equimolecular concentrations calcium bicarbonate produces a less pronounced colour than magnesium bicarbonate; but sodium carbonate is about as active as the latter. Boiling of calcium bicarbonate solutions before addition of hop extract does not entirely prevent the coloration, and in the case of magnesium bicarbonate previous boiling has no effect.—J. H. L.

*Beers; Attenuation of low gravity* —. E. Schlichting. U.S. Master Brewers' Assoc. Brewers' J., 1918, 54, 249.

DISCUSSING the problem of brewing palatable and stable beer containing not more than 2.75% of alcohol, the author recommends that a product of the following composition should be aimed at:—alcohol, 2.5–2.6% by weight; acidity, 0.12–0.14%; real unfermented extract, 4.3–4.4%; original gravity, about 9.5% Balling; ratio of alcohol to real extract, 1:1.66–1:1.75; apparent degree of attenuation 64–68%; real degree of attenuation 52–54%. It is stated that such a product may be obtained by using 75% of malt and 25% of raw grain, holding the mash for an hour at 38° R. (47.5° C.) and carrying out conversion at 58° R. (72.5° C.), hopping at the usual rate (for lager beer) in proportion to the extract, and fermenting as completely as possible. Great importance is attached to the ratio of alcohol to extract: too high a proportion of the latter renders the beer insipid.—J. H. L.

*Beer; Investigation of — for antineuritic and antiscorbutic potency.* A. Harden and S. S. Zilva. J. Inst. Brew., 1918, 24, 197–208.

THE history of the subject of deficiency diseases and their prevention and remedy is sketched, and experiments are described, comprising feeding tests with pigeons, guinea-pigs, and monkeys, which indicate that beer and malt are destitute both of the antineuritic and the antiscorbutic substances; i.e., they have neither preventive nor curative action on polyneuritis or scurvy produced by defective diet.—J. H. L.

*Oxalic acid in beer [urine, etc.]; Determination of* —. A. Bau. Woch. Brau., 1918, 35, 31–33, 40–42, 45–47, 51–53, 57–59, 63–65, 70–71.

THE minute quantities of oxalates present in beers (cp. Will, this J., 1916, 191) may be precipitated by a reagent prepared by mixing 500 c.c. of 50% acetic acid containing 25 grms. of crystallised calcium chloride, with 500 c.c. of saturated sodium acetate solution (cp. Kreis and Baragiola, this J., 1916, 483). 400 c.c. of filtered beer is treated with 80 c.c. of the clear reagent, and left for 38–44 hours

at a temperature below 7° C. The deposited calcium oxalate may be identified microscopically, or determined quantitatively by filtering off, washing thoroughly with cold water, igniting, and estimating the lime by dissolving in N/10 hydrochloric acid and titrating back with N/10 sodium hydroxide in presence of methyl orange. A correction may be made for the oxalate remaining in the mother liquor and the washings, on the basis that the solubility of calcium oxalate in the former is 4.86 mgrms., and in the latter 6.6 mgrms., per litre. The method might be applied to other liquids, e.g., urine, which, like beer, do not contain tartaric acid, this being the only other acid besides oxalic acid precipitated under the prescribed conditions. The results obtained with beers of different types ranged from 8 to 34 mgrms. of calcium oxalate per litre. The oxalates in beer appear to be derived chiefly from the hops and to some extent from the malt; during fermentation the oxalate-content of the wort diminishes.—J. H. L.

*Banana must; Alcoholic fermentation of* —. R. Perratti and V. Riviera. Staz. Sperim. Agrar. Ital., 1917, 50, 433–450. Bull. Agric. Intell., 1918, 9, 610–612.

EARLIER work on the chemistry and bacteriology of the *Musa sapientum* banana is summarised. To obtain a must which will retain after fermentation the flavour of the fruit, the skins as well as the pulp must be extracted. From such a must the authors isolated four organisms: a *Saccharomyces* predominant in the fermented liquid and designated *Sacch. musae*, a bacterial form abundant in the liquid, a variety of *Oospora lactis*, and a *Mycoderma* form. Particulars of these organisms are given. From an experiment on the fermentation of an extract of skins and pulp, with *Sacch. musae*, it appears possible to produce from the banana, by adding sugar to the must, and perhaps purifying after the primary fermentation, a fermented liquid of good colour, slightly alcoholic, and of attractive qualities amongst which the agreeable aroma of the fruit holds first place.—J. H. L.

*Alcohol; Production of — from algae.* Kayser. Comptes rend. Acad. d'Agric., 1918, 4, 450–451. Bull. Agric. Intell., 1918, 9, 612–613.

*Laminaria digitata*, dried to a moisture content of 10%, was heated for  $\frac{1}{2}$ –1 hour at 122° C., with water containing 3–6% of sulphuric acid. The cooled liquid, brought to an acidity of 0.1%, and sprinkled with brewers' yeast, fermented readily, especially after addition of nitrogenous material, and yielded 6 litres of alcohol per 100 lb. of dry algae. Higher yields might perhaps be obtained under industrial conditions, and the mineral matter in the vinasses might be utilised.—J. H. L.

*Cognac kept in wooden barrels; Alteration of the alcohol content of* —. A. Behre. Z. Unters. Nahr. Genussm., 1918, 35, 281–283.

ALTHOUGH considerable evaporation took place, the loss amounting to some 30%, when cognac was kept for 18 months in a wooden barrel, the alcoholic strength (40% by vol.) and the total solids remained almost constant; the alcohol-content tended to increase to the extent of about 1% per year.

—W. P. S.

*Enzymes; Chemistry of* —. H. von Euler. Z. Elektrochem., 1918, 24, 173–177.

THE inversion of sucrose by yeast is accompanied by an increase in the invertase to a maximum and an increase in the enzyme concentration according

to the equation  $dx/dt = k(a-x)$ , where  $a$  is the initial concentration of sucrose and  $x$  is the amount inverted after time  $t$ . In the hydrolysis of starch by the enzyme of *Mucor mucedo* the velocity of the enzyme formation is constant.—J. P. S.

*Sucrase [invertase]; Law of action of* —. H. Collin and A. Chaudun. *Comptes rend.*, 1918, 167, 208—210.

The authors find that only at concentrations of sucrose not exceeding 2% does the inversion of sucrose by invertase obey Wilhelm's law  $x = a(1 - e^{-kt})$ , where  $a$  is the initial concentration of sucrose and  $x$  is the quantity of sucrose hydrolysed after time  $t$ .—W. G.

#### PATENTS.

*Malt-drum; Rotary* —. O. Gaebel, Breslau, Germany. Eng. Pat. 116,966, Aug. 14, 1917. (Appl. No. 11,671 of 1917.)

A rotary malt-drum has within it two screws or helical blades of about the same graduation, one right-handed and the other left-handed. One is mounted on the outside of a central perforated pipe (which serves to admit air), and the other on the inside of a co-axial perforated cylinder or pipe of somewhat smaller diameter than the drum itself. When the drum rotates the contents are mixed owing to the fact that the two screws drive the central and peripheral parts of the charge in opposite directions.—J. H. L.

*Alcohol; Manufacture of — from grain in open vats with the employment of saccharifying Mucors.* Soc. d'Exploit. des Proc. H. Boulard, Paris. Eng. Pat. 102,945, Dec. 7, 1916. (Appl. No. 17,596 of 1916.) Under Int. Conv., Dec. 28, 1915.

COOKED grain mashies are saccharified and fermented in open vats by means of a robust *Mucor* such as *Mucor Boulard* No. 5 (this J., 1914, 497), and a rapidly acting yeast such as Yeast Boulard Nos. 21 to 30. A pure culture of the *Mucor* is first grown in a small cooked grain mash, about one-sixth as large as the main mash, in a closed vat, under conditions which exclude infection. When the mould has developed sufficiently this mash is added to the main mash in an open wooden or iron vat. The yeast is added a few hours later, and within 48 hours both saccharification and fermentation are complete. Foreign organisms which gain access to the vat are not able to develop to a serious extent within this period, the alcohol produced serving to hold them in check. It is stated that the process can be used in this country according to the present laws, whilst processes in which *Mucors* are employed in closed vats are not permitted.—J. H. L.

*Beverage; Non-alcoholic* —. A. L. Straus, Baltimore, Md. U.S. Pat. 1,265,274, May 7, 1918. Date of appl., Aug. 4, 1917.

A FERMENTED malt liquor is freed from alcohol and treated with some or all of the following:—water, salt, hops, gum arabic, sugar, quassia, a chill-proofing preparation, potassium metabisulphite, and carbon dioxide.—J. H. L.

*Beverage; Non-alcoholic — and process of producing the same.* A. L. Straus, Assignor to Baltimore Process Co., Baltimore, Md. U.S. Pat. 1,265,275, May 7, 1918. Date of appl., Oct. 15, 1917.

A FERMENTED liquor, prepared from glucose without malt, is freed from alcohol and treated with some or all of the following:—water, salt, sugar, quassia,

hops, a foam-producing material, a preserving material, e.g., potassium metabisulphite, a chill-proofing material, and carbon dioxide.—J. H. L.

*Alcoholic liquids [whisky]; Process for ageing* —. J. Von Glahn, Brooklyn, N.Y. U.S. Pat. 1,265,838, May 14, 1918. Date of appl., Oct. 9, 1914.

ALCOHOLIC liquids containing fusel oils, e.g., whisky, are matured by storage in contact with charcoal and with finely divided wood which has been previously impregnated with matured alcoholic liquid.

—J. H. L.

*Alcoholic liquors and the like; Apparatus for distilling in multiple effect* —. E. Barbet et Fils et Cie., Paris. Eng. Pat. 107,600, June 30, 1917. (Appl. No. 9466 of 1917.) Under Int. Conv., Oct. 19, 1914. Addition to Eng. Pat. 107,599 (see Fr. Pat. 478,885; this J., 1916, 1172).

SEE Addition of Oct. 19, 1914, to Fr. Pat. 478,885 of 1914; this J., 1916, 1173.

*Solid alcohol and process of producing same.* U.S. Pat. 1,266,080. See IIA.

#### XIXA.—FOODS.

*Bread; Use of lime water in the preparation of* —. Balland. *Comptes rend.*, 1918, 167, 198—201.

THE quantity of lime water necessary to produce a distinct yellow tinge with the flour is very variable, but is not sufficient completely to neutralise the acidity of the flour, so that panary fermentation is not inhibited. With regard to the quality of the bread, it is often difficult to distinguish between bread made with and without the addition of lime-water. The colour and taste of bread from very brown flour are apparently slightly improved by the use of lime-water.—W. G.

*Milk; Colorimetric determination of lactose in* —. A. J. P. Pacini and D. W. Russell. *J. Biol. Chem.*, 1918, 34, 505—507.

ONE c.c. of human milk is diluted with water to 100 c.c. About 0.5 gm. of solid picric acid is dissolved in about 10 c.c. of the diluted milk by stirring, and after 5 minutes, the solution is centrifuged for 10 minutes. A portion of the upper liquid is filtered to remove the fat, and 1 c.c. of the filtrate transferred to a long test-tube and heated with 1 c.c. of saturated sodium carbonate solution in boiling water for 20 minutes. After cooling and diluting to 10 c.c., the colour is compared with that obtained by similarly treating 1 c.c. of a standard 0.1% solution of pure lactose in saturated picric acid solution. When cow's milk is being tested, a slightly weaker standard solution of lactose should be employed.—H. W. B.

*Milk; Casein of human* —. A. W. Bosworth and L. A. Giblin. *J. Biol. Chem.*, 1918, 35, 115—117.

THE casein of human milk has all the properties associated with the casein of cow's and goat's milk. —H. W. B.

*Milk as a source of water-soluble vitamine.* T. B. Osborne, L. B. Mendel, E. L. Ferry, and A. J. Wakeman. *J. Biol. Chem.*, 1918, 34, 537—551.

MILK contains only a relatively small proportion of water-soluble vitamine. (See also *J. Chem. Soc.*, Sept., 1918.)—H. W. B.



*Sour milk; Free lactic acid in* —. L. L. Van Slyke and J. C. Baker. *J. Biol. Chem.*, 1918, 35, 147—178.

Sour milk contains lactates and free lactic acid, part of which is adsorbed by the caseinogen or casein. The amount of free lactic acid in milk that has soured under ordinary conditions may vary from 13 to 35 c.c. of N/10-acid per 100 c.c. of milk. About 20% of the free lactic acid is adsorbed by the casein. Methods are described for estimating the adsorbed and total free lactic acid in sour milk. (See further, *J. Chem. Soc.*, Sept., 1918.)

—H. W. B.

*Nitrogen in liquids poor in nitrogen [e.g., milk]; Micro-Dumas estimation of* —. G. Kraemer. *J. prakt. Chem.*, 1918, 97, 59—60.

The usual micro-Kjeldahl method for estimating nitrogen in small quantities of a liquid such as milk can be replaced satisfactorily by a micro-Dumas process. A small copper boat of approximately 0.25 c.c. capacity enclosed in a small thin-walled glass dish covered with a glass plate is weighed to the fifth decimal place, first empty and then filled with the milk. The boat is placed in a small vacuum desiccator and kept at 30°—35° C. until the water has evaporated; the boat and its contents are then introduced into the combustion tube and covered with fine copper oxide, preparatory to the combustion. The weighing, drying, and combustion occupy an hour.—D. F. T.

*Cream; Pasteurisation of* —. C. Larsen, J. M. Fuller, V. R. Jones, H. Gregory, and M. Tolstrup. *South Dakota State Coll. Agric., Bull. No. 171*, Nov. 1916, 529—548. *Bull. Agric. Intell.*, 1918, 9, 621—622.

In some European countries the whole of the butter, and in the United States about two-thirds, is made from pasteurised cream. The effect of pasteurising cream at different temperatures was studied, using a coil vat of 150 galls. capacity, operated by a 20 HP return tubular boiler and a 15 HP engine to run the coil. The cream was heated for 25 mins. at 140° F. (60° C.), or for 10 mins. at 160° F. (71° C.), or momentarily to 180° F. (82° C.) followed by immediate cooling. The middle temperature (160° F.) effected the greatest reduction in the total number of organisms; 160° and 180° F. were more effective than 140° F. in killing moulds and non-acid forming organisms. The only appreciable effects of pasteurisation on the composition of the cream were a decrease in moisture-content and a slight decrease in acidity. Microscopical comparison of the cream before and after heating showed a tendency for the higher temperatures (160° and 180° F.) to cause the fat globules to coalesce. Pasteurisation had no important influence on the chemical composition of the butter produced, nor did the high temperatures unfavourably affect the "body" of the product. The butter made from cream pasteurised at 180° F. showed the best keeping qualities.—J. H. L.

*Butter; Rancidity of* —. E. S. Guthrie. *Dairy Sci.*, Baltimore, 1917, 1, 218—233. *Bull. Agric. Intell.*, 1918, 9, 622—623.

APART from biological agencies (which were as far as possible excluded in the author's experiments) the purely chemical and enzymic transformations which occur in butter do not affect the iodine value considerably and do not cause rancidity; neither does exposure to high temperatures, light, and air. Truly rancid butter is rarely found, strong flavour

being often erroneously referred to as rancidity. The original paper contains a bibliography of the subject.—J. H. L.

*Casein; Preparation of pure* —. L. L. Van Slyke and J. C. Baker. *J. Biol. Chem.*, 1918, 35, 127—136.

CASEIN is precipitated in a pure form from undiluted milk by the addition of lactic or other acid, the milk being maintained in rapid motion by means of a rapidly revolving mechanical stirrer.—H. W. B.

*Casein; Action of pancreatic enzymes upon* —. H. C. Sherman and D. E. Neun. *J. Amer. Chem. Soc.*, 1918, 40, 1138—1145.

A COMPARATIVE study of the hydrolysis of casein by various preparations derived from the pancreas. Extraction of high-grade commercial pancreatin with 50% alcohol leaves a residue having about the same proteolytic activity as the original pancreatin. The sac precipitate which settles out of the amylase solution during the dialysis in 50% alcohol, preceding the final precipitation of the amylase preparation (Sherman and Schlesinger, *J. Amer. Chem. Soc.*, 1912, 34, 1110; 1915, 37, 1306; this J., 1915, 627), has 15 times the proteolytic activity of the original pancreatin and about 4 times that of the most active commercial trypsin. The final preparation of pancreatic amylase purified as described previously (*loc. cit.*) has proteolytic activity fully equal to that of high-grade trypsin. (See also *J. Chem. Soc.*, Sept., 1918.)—C. S.

*Barley; Dietary qualities of* —. H. Steenbock, H. E. Kent, and E. G. Gross. *J. Biol. Chem.*, 1918, 35, 61—74.

BARLEY, when tested by feeding it to rats, was found to contain an abundance of water-soluble vitamins, but was deficient in salts, protein, and fat-soluble vitamins.—H. W. B.

*Nutritive factors in plant tissues. I. The protein factor in the seeds of cereals.* T. B. Osborne, L. B. Mendel, E. L. Ferry, and A. J. Wakeman. *J. Biol. Chem.*, 1918, 34, 521—536.

PROTEIN concentrates were prepared from various cereals by treating them with diastase to remove the starch, and rats were fed on diets containing these various protein concentrates as sole sources of protein, with the exception of a small proportion of yeast added in each case to supply the necessary water-soluble vitamins. Normal growth was obtained with the concentrates from rice and barley, but not with those from maize or oats.—H. W. B.

*Carbohydrates and fats; Comparative study of the influence of — on the nutritive power of alimentary proteins.* F. Maignon. *Comptes rend.*, 1918, 167, 172—175.

FEEDING experiments with rats, using diets of egg-albumin and fat, or egg-albumin and starch, or a mixture of the three, indicate that the albumin is better utilised with fat than with starch, the minimum of albumin necessary being much lower with the former than with the latter.—W. G.

*Ovalbumin; Preparation of — and its refractive indices in solution.* A. R. C. Haas. *J. Biol. Chem.*, 1918, 35, 119—125.

THE crystallisation of ovalbumin from ammonium sulphate solutions only succeeds when the hydrogen ion concentration of the solution lies between  $10^{-5}$

and 10°. The refractive indices of ovalbumin in various solvents have been found to obey Robertson's law (J. Phys. Chem., 1909, 13, 472).—H. W. B.

*Vegetable gums in food products; Detection of* —. A. A. Cook and A. G. Woodman. J. Ind. Eng. Chem., 1918, 10, 530—533.

FAOM 50 to 200 grms. of the sample is diluted with water, 5 c.c. of dilute acetic acid and 25 c.c. of 10% tannin solution are added, the mixture is heated for 30 mins., centrifuged, and filtered; the filtrate is again treated with tannin solution (40 to 50 c.c.), heated, centrifuged, and filtered. Casein, proteins, gelatin, fat, etc., are thus removed. The clear filtrate is then mixed with twice its volume of acetone, centrifuged, and the precipitate collected, washed twice with acetone, dissolved in 50 c.c. of warm water, acidified slightly with acetic acid, 10 c.c. of ammonia (sp. gr. 0.9) is added, the mixture centrifuged, and filtered; calcium phosphate from the milk solids (if present) is thus removed. The gums are then precipitated from the filtrate by acidifying the latter with acetic acid and adding alcohol, one volume at a time. If, after the addition of five vols. of alcohol, a precipitate is not obtained gums are absent. The quantity of alcohol required for the precipitation and the character of the precipitate give an indication of the kind of gum present. For example, one volume of agar solution requires 3 to 4 vols. of alcohol, the precipitate is finely-divided and, when air-dried, remains soft and non-coherent; gum arabic requires 2 vols. of alcohol, the precipitate is flocculent and dries to a powder; Indian gum requires 2 to 3 vols. of alcohol and gives a stringy precipitate which dries to a dark-coloured tough film; gum tragacanth gives jelly-like clots with 2 vols. of alcohol, the clots drying to a semi-transparent layer; dextrin requires 3 vols. of alcohol, giving a fine, white sticky precipitate which tends to become hard when dried and kept. Microscopic examination of the tannin precipitate may reveal the characteristic diatoms found in agar if this gum is present, whilst the volatile acidity of Indian gum is of value as a confirmatory test. When pectins are present they may be removed from the gums by treating the ammoniacal solution of the latter (see above) with a few drops of iron-tannin solution before the acetic acid is added. Dextrins are separated from the gums by heating 0.5 gm. of the dried gum for 5 mins. in a water-bath with 50 c.c. of water and 25 c.c. of hydrochloric acid (sp. gr. 1.2). The method described above will detect the presence of 0.1 gm. of gum in 100 grms. of complex food substance.—W. P. S.

*Baking powder; Determination of carbon dioxide in* —. H. Schellbach and F. Bodinus. Z. Unters. Nahr. Genussm., 1918, 35, 236—240.

IN the method described the carbon dioxide evolved from the baking powder is absorbed in 33% potassium hydroxide solution and the quantity of potassium carbonate formed is determined volumetrically; 5% phosphoric acid solution is used to decompose the carbonate in the baking powder.

J. Tillmans and O. Heublein (*ibid.*, 257—266) find that the above method is untrustworthy.—W. P. S.

*Coffee substitutes; Microscopical examination of* —. C. Griebel. Z. Unters. Nahr. Genussm., 1918, 35, 223—235, 272—277.

DIAGRAMS are given showing the characteristic features of the seeds of the common corn spurrey (*Spergula arvensis*, L.) and the so-called aeneia (*Robinia pseudacacia*, L.), both of which are used

in Germany as coffee substitutes; the former seeds contain numerous conglomerations of starch cells, whilst the latter are free from starch but contain considerable quantities of fat and proteins. The microscopic appearance of the seeds of bird's foot (*Ornithopus sativus*, Brot.) is also described.

—W. P. S.

*Coffee substitutes containing lupine seeds. II.* Eckenroth. Z. Unters. Nahr. Genussm., 1918, 36, 240—242.

THE bitter taste of lupine seeds is due to the presence of alkaloids, saponins, etc., and although processes have been suggested for the removal of these substances, they are unreliable. Moreover, roasting does not destroy the bitter substances, and the author is of opinion that lupine seeds should not be used as a coffee substitute. (Compare Gonnerman, this J., 1918, 483 A.)—W. P. S.

*Cottonseed meal; Uniform nitrogen determination in* —. J. S. McHargue. J. Ind. Eng. Chem., 1918, 10, 533—535.

THE following points should be observed in the determination of nitrogen in cottonseed meal. The sample should be ground finely (40-mesh sieve) in order to obtain a homogeneous mixture of hulls and meal. When mercury is used as catalyst, loss of nitrogen may occur if the boiling is prolonged for more than 2 hrs.; the digestion period, using copper sulphate as catalyst, depends on the amount of sodium sulphate added, 12 grms. being sufficient to complete the decomposition in 2 hrs. Sodium sulphate is as efficient as potassium sulphate in the digestion. Precipitation of the copper as cuprous chloride facilitates the boiling and shortens the time of distillation. Suitable quantities of materials and conditions for the determination are:—cottonseed meal, 0.7 gm.; crystallised copper sulphate, 0.5 gm.; anhydrous sodium sulphate, 12 grms.; sulphuric acid, 25 c.c.; time of digestion, 2 hrs.—W. P. S.

*Alfalfa [lucerne] hay; Isolation and identification of stachydrine from* —. H. Steenbock. J. Biol. Chem., 1918, 35, 1—13.

STACHYDRINE is present in small amount in the phosphotungstic acid fraction of the aqueous extract of lucerne hay. The presence of stachydrine may vitiate values for histidine and lysine obtained by the Van Slyke method and for lysine obtained by the Kossel method when these values are obtained directly on the feeding material itself.—H. W. B.

*Leaf hay; Composition and feeding value of* —. F. Honecamp and E. Blanck. Landw. Versuchs.-Stat., 1918, 91, 291—308.

DAREN leaves of the beech, maple, lime, birch, alder, chestnut, etc., form a valuable feeding stuff; they contain from 10 to 23% of protein, 2 to 10% of fat, and 17 to 22% of crude fibre.—W. P. S.

*Heather and reindeer moss; Composition and di-* F. Honecamp and E. Blanck. Landw. Versuchs.-Landw. Versuchs.-Stat., 1918, 91, 223—251.

HEATHER meal contains about 7% of proteins, 9% of fat, 8% of ash, and 12 to 21% of crude fibre; when prepared from the leaves and flowers only it has a food value approximating that of poor quality hay. Reindeer moss is of little value as a fodder; it contains about 2% of fat and 4% of proteins.

—W. P. S.



*Pigweed, Amaranthus retroflexus L.; Proximate analysis of the seed of the common* —. E. P. Harding and W. A. Egge. J. Ind. Eng. Chem., 1918, 10, 529—530.

PIGWEEED grows abundantly in the State of Minnesota; analysis of the seeds yielded the following results:—Moisture, 11.28; ash, 4.33; ether extract, 7.03; proteins, 18.57; starch (by diastase) 32.40, (by acid inversion) 39.77; reducing sugars, trace; other sugars, 2.08; crude fibre, 10.59; tannin and undetermined, 6.35%. The seeds might be used as a poultry food and the meal as a cattle food.—W. P. S.

*Silver maple (Acer saccharinum); Analysis and composition of the seed of the* —. R. J. Anderson. J. Biol. Chem., 1918, 34, 509—513.

THE seed of the silver maple has a high food value. It contains 42% starch, 27.5% protein, 16% sugar, and 5% of ash, consisting chiefly of potassium phosphate. The chief protein is a globulin. The phosphorus in the seed is present chiefly in organic combination.—H. W. B.

*Sudan durra as a brewing material.* Briant and Harman. See XVIII.

*Determination of essential oils in non-alcoholic flavouring extracts.* Boyles. See XX.

*Abnormalities in the formal titration method.* Jodidi. See XXIII.

*Method for making electrometric titrations of solutions containing protein.* Baker and Van Slyke. See XXIII.

#### PATENTS.

*Grain; Method and apparatus for treating* — [with ozonised air]. P. J. H. Moore, Chicago, U.S.A. Eng. Pat. 116,952, Apr. 12, 1917. (Appl. No. 10,126 of 1917.)

IN a process of treating unbroken grain, such as wheat, preparatory to storage, the grain is fed continuously into the top of a tall chamber and discharged from the bottom, at such a rate that the chamber remains full, whilst ozonised air contained in an adjoining chamber is injected into the grain chamber through openings at different heights in the common wall. Each opening is situated below an inverted V-shaped trough extending across the grain chamber, and the ozonised air issuing from below these troughs percolates upwards through the descending column of grain and escapes from the top of the chamber. The air is ozonised by silent electric discharge in apparatus comprising readily accessible I-shaped glass plates wound with coils and separated by other insulating glass plates. —J. H. L.

*Wheat and other grains; Process of treating* —. A. C. Von Hagen, Kansas City, Mo. U.S. Pat. 1,265,700, May 7, 1918. Date of appl., Sept. 1, 1916.

THE grain is treated superficially in presence of moisture insufficient to saturate it, with a substance capable of yielding a halogen in the free state, *e.g.*, calcium hypochlorite, or simultaneously with an alkali, such as calcium hydroxide, to neutralise the free acidity of the grain, and a hypochlorite, to exert a germicidal action.—J. H. L.

*Egg food preparations.* M. Cutler, London, Eng. Pat. 117,022, Nov. 28, 1917. (Appl. No. 17,597 of 1917.)

WHOLE eggs or the yolks alone, in liquid or powdered form, are mixed with a liquid prepared by boiling

linseed with water; a preservative such as borax may be added.—J. H. L.

*Baking powder.* A. H. Peter, Assignor to Royal Baking Powder Co., New York. U.S. Pat. 1,265,369, May 7, 1918. Date of appl., Sept. 16, 1916.

CLAIM is made to baking powders containing lactide and acid lactates as acid constituents.—J. H. L.

*Curing meats.* G. F. Doran, Omaha, Nebr., U.S.A. Eng. Pat. 107,593, June 28, 1917. (Appl. No. 9298 of 1917.) Under Int. Conv., Apr. 29, 1916.

SEE U.S. Pat. 1,212,614 of 1917; this J., 1917, 401.

*Preserving fruits and other organic substances; Method of* —. H. C. M. Franks, Brooklyn, N.Y., Assignor to Franks & Co., Inc. Reissue 14,489, June 25, 1918, of U.S. Pat. 1,232,271, July 3, 1917. Date of appl., Mar. 18, 1918.

SEE this J., 1917, 938.

#### XIXB.—WATER PURIFICATION; SANITATION.

*Lime-sulphur solutions; Composition of* —. O. B. Winter. J. Ind. Eng. Chem., 1910, 10, 538—545.

THE presence of hydrogen sulphide, calcium hydro-sulphide, calcium hydroxyhydrosulphide, and the corresponding salts of other metals may be detected by titrating an aliquot portion of the sample with iodine until the yellow colour has disappeared and then repeating the titration using sodium nitroprusside to determine the end-point; after the polysulphide has been decomposed the blue colour of the nitroprusside remains as long as any sulphide sulphur is present. Ordinary lime-sulphur, which has stood for several days, does not contain any of the above-mentioned compounds, and cannot contain free lime, since any of the latter which may be present when the preparation is made gradually disappears. In freshly prepared lime-sulphur solution, the difference between the titrations with hydrochloric acid and ammoniacal zinc chloride solution is a measure of the free lime. When magnesium sulphate is added to a lime-sulphur solution there is a slight decrease in the content of monosulphide and of sulphide sulphur, the thiosulphate sulphur remains practically the same, magnesium replaces a part of the calcium forming magnesium polysulphide and calcium sulphate may separate, and a compound containing the SH radical is formed. The magnesium sulphate method for determining free lime in lime-sulphur solution is inaccurate. The sulphur which separates from lime-sulphur solution on standing is derived from the polysulphide and is not due to the presence of free sulphur. Orange-red crystals which separated from a concentrated lime-sulphur solution prepared with an excess of lime appear to consist of an additive compound of calcium oxide with calcium polysulphide,  $2\text{CaO} \cdot \text{CaS}_3 \cdot 11\text{H}_2\text{O}$ , but it is improbable that they exist in the solution form.—W. P. S.

*Disinfection; Adsorptive* — by metallic combination and disperse galvanic elements. H. Bechhold. Z. Elektrochem., 1918, 24, 147—150.

FINELY divided wood charcoal, fullers' earth, silicic acid, animal charcoal, iron oxide, alumina, calcium oxalate, and barium sulphate all act as disinfectants toward *Staphylococcus* due to their adsorptive properties. The bacteria are retained

living on the surface of the adsorption medium. The amount of adsorption varies from 99.97% with wood charcoal to 62.32% with alumina. The deposition of thin films of metals on the adsorption medium (gold, silver, copper, or mercury) has the effect of killing the adsorbed bacteria, the action of the metal being specific. A mixture of two metals is still more effective in certain cases. Thus in the case of typhus, mercury alone leaves 16,883 colonies, whereas mercury and copper together leave only one colony. Moreover, on injecting colloidal metals into mice, mixtures of metals were found to be more deadly than the single metals, thus: 4% silver killed in 4 days, 4% mercury had not killed in 11 days, but a mixture of 4% mercury and 4% silver killed in 6 hours.—J. F. S.

*Analysis of air from a mine fire.* Blakeley and Gelst. See IIA.

#### PATENTS.

*Furnaces; Incinerating* — J. W. Stokes, Dallas, Tex., U.S.A. Eng. Pat. 117,417, Feb. 26, 1918. (Appl. No. 3385 of 1918.)

An arched furnace chamber has a saucer-shaped floor and curved rear wall. The furnace has three grates: one on the side of the chamber furthest from the stack and separated from the main body of the chamber by a battie-wall is used for burning fuel; one in the centre of the front wall for completing the combustion of the garbage or other material to be incinerated; and an auxiliary grate on the side nearest the stack. Garbage is fed through a charging door in the top of the furnace on to an elevated grate supported on arches and having a forward inclination. The combustion gases pass above and below this grate and partly incinerate the material, the combustion being completed on, and the cinders discharged from, the centre grate.—W. H. C.

*Black rot and other fungoid and bacterial diseases in leaf-tobacco; Process for preventing the development of* — W. W. Garner and E. G. Reinhardt, Washington, D.C. U.S. Pat. 1,268,070, May 28, 1918. Date of appl., Jan. 10, 1918. (Dedicated to the public.)

The process consists in controlling the moisture content of tobacco by subjecting the material to the action of heated air under varying conditions of temperature and humidity, these being alternated, thereby preventing the growth of fungoid and other diseases in leaf tobacco.—W. G.

*Separation and settlement of solids and semi-solids from chemical and like manufacturing liquids.* Eng. Pat. 117,472. See I.

*Method of producing high-boiling phenols.* Eng. Pat. 116,905. See III.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Cinchona alkaloids; Identification of the* — by optical-crystallographic measurements. E. T. Wherry and E. Yanovsky. J. Amer. Chem. Soc., 1918, 40, 1063—1074.

PURIFIED samples of cinchonine, cinchonidine, quinine and quinidine were separately crystallised from alcohol and from benzene and the crystals examined under the microscope in ordinary light, parallel polarised light and convergent polarised

light; the refractive indices were determined by the immersion method in solutions of potassium mercuric iodide and glycerol of known indices. The results are given in tables which have been used successfully in the identification of the four alkaloids in the products obtained by fractional crystallisation of mixtures from alcohol or benzene. The general procedure in examining medicinal preparations for the presence of these alkaloids is illustrated in the case of a proprietary remedy presumably containing the alkaloids as sulphates. A few of the pills were ground with potassium carbonate, suspended in water, and shaken with chloroform. The gummy residue obtained from the chloroform extract after the evaporation of the solvent was shaken with dilute sulphuric acid, the filtered solution treated with dilute aqueous ammonia, and the resulting grey precipitate extracted with a few c.c. of benzene. The benzene layer was separated and allowed to evaporate. A drop of the concentrated solution was placed on a microscope slide and covered with a slip when evaporation was nearly complete. Crystals soon formed round the edge of the cover glass consisting of three distinct types, which were easily recognised as quinine, cinchonidine, and cinchonine respectively. The result was confirmed by immersing the crystals in liquids of known refractive indices. (See also J. Chem. Soc., Sept., 1918.)—C. S.

*Alkaloid from the common broom; A new volatile* — A. Valeur. Comptes rend., 1918, 167, 163—164.

In addition to the fixed alkaloid, sarothamnine, the author has isolated from the mother liquors in the manufacture of sparteine sulphate, a volatile alkaloid genisteine,  $C_{17}H_{23}N_3$ , m.pt., 60.5° C.; b.pt., 139.5°—140.5° C. at 5 mm. (corr.), 177°—178° C. at 22 mm. (corr.). It readily absorbs water vapour, forming a hydrate with 1 mol.  $H_2O$  ( $[\alpha]_D = -52.3$  in 4% alcoholic solution). (See further J. Chem. Soc., Sept., 1918.)—W. G.

*Coumarin; Detection and determination of* — in *fascitious vanilla extracts.* H. J. Wichmann. J. Ind. Eng. Chem., 10, 535—537.

TEN c.c. of the extract is rendered alkaline with 10% sodium hydroxide solution, 15 c.c. of water is added, and the mixture is extracted with 20 c.c. of ether. The ethereal solution is shaken with a few c.c. of concentrated alcoholic potassium hydroxide solution, washed with 10 c.c. of water, mixed in a test-tube with 1 c.c. of 50% potassium hydroxide solution, the ether and water evaporated, and the residue of potassium hydroxide fused. If coumarin is present, a greenish coloration develops as the water evaporates. This colour disappears as the heating is continued, the coumarin being converted into salicylic acid. The mass is cooled, dissolved in water, the solution acidified with sulphuric acid, extracted with benzene, and the benzene solution tested for salicylic acid by the ferric chloride reaction. The quantity of coumarin present is determined by treating 50 c.c. of the alcoholic extract with lead acetate, diluting to 100 c.c., filtering, and removing the excess of lead by the addition of dry potassium oxalate. Fifty c.c. of the filtrate is then extracted with ether, the ethereal extract treated with an excess of alcoholic potassium hydroxide solution and a few drops of phenolphthalein, and the vanillin salt removed by washing with several portions of 10 c.c. of water. The disappearance of the red coloration indicates that the washing is sufficient. The ether is then evaporated, and the residue of coumarin dried and weighed. The amount of the residue varies from 98 to 102% of the quantity of coumarin present.—W. P. S.



*Acetyltaminophenyl salicylate. Preparation of salophen.* R. Q. Brewster. J. Amer. Chem. Soc., 1918, 40, 1136—1138.

The investigation was undertaken in consequence of the difficulty of obtaining good yields of salophen. The reduction of crude *p*-nitrophenyl salicylate must be effected in a non-aqueous solvent in order to avoid hydrolysis of the ester and of its reduction product; with glacial acetic acid and zinc dust the yield of *p*-aminophenyl salicylate is 45% of the theoretical. Salophen was prepared without isolating the free base. Crude *p*-nitrophenyl salicylate (5 grms.) dissolved in 20 c.c. of glacial acetic acid (rendered anhydrous by adding sufficient acetic anhydride) was added slowly, with shaking, to 20 grms. of zinc dust and 15 c.c. of anhydrous acetic acid. After 20 minutes the liquid was decanted, the solid residue was washed with anhydrous acetic acid, and the combined liquors heated under a reflux condenser for 20 hours, cooled, and poured into 5 vols. of water. The crude salophen, m.p. 175°—180° C., was obtained in a yield of 55%, and a further 5% was obtained from the filtrate after evaporating the acid at 50° C. in a vacuum. After purification with charcoal and 50% alcohol the salophen had m.p. 187° C. *p*-Nitrophenyl salicylate crystallised once from alcohol and twice from petroleum spirit of high boiling point, has m.p. 145°—147° C. Using this material the yield of the free base is increased to 73%, and that of salophen to 80%, of the theoretical.—C. S.

*Esters; Anodic formation of — simultaneously with nitrobenzene and o-nitrophenol from mono-nitrobenzoic acids.* C. Schall. Z. Elektrochem., 1918, 24, 154—157.

MIXTURES of potassium *o*-nitrobenzoate and the free acid in acetic anhydride solution give on electrolysis as anode products, nitrobenzene, *o*-nitrophenol, and the *o*-nitrophenyl ester of *o*-nitrobenzoic acid. The yield varies very much with temperature. A concentrated aqueous solution of potassium acetate and *o*-nitrobenzoate on electrolysis yields nitrobenzene, *o*-nitrophenol, and methyl-*o*-nitrobenzoate. (See also J. Chem. Soc., 1918, i, 389.)—J. F. S.

*Malonic acid; Detection and identification of —.* Bouganlt. Ann. Chim. Analyt., 1918, 23, 154—155.

The method depends on the condensation of malonic acid with cinnamic aldehyde to form cinnamylidenemalonic acid which is almost insoluble in water: it is a yellow substance, m.p. 298° C. A quantity of 0.1 gm. of malonic acid, sodium malonate, or calcium malonate, is mixed with 15 drops of cinnamic aldehyde and 1 c.c. of acetic acid and heated in a sealed tube for 10 hrs. at 100° C. The mixture is then saturated with sodium carbonate, filtered, the cinnamylidenemalonic acid precipitated by the addition of alcohol, collected, dried at 100° C., and weighed. The yield is 0.110 gm. for each 0.10 gm. of malonic acid present.—W. P. S.

*Amyl acetate and similar solvents; Manufacture of — from petroleum pentane.* B. T. Brooks, D. F. Smith, and H. Essex. J. Ind. Eng. Chem., 1918, 10, 511—515

The chlorination of the pentane fraction (b.pt. 25° to 45° C.) is carried out by passing chlorine through a number of small orifices into a large quantity of cold pentane, the operation being stopped when the mixture contains about 20% of chlorine derivatives. The mixture should be kept at 10° C., or lower, during the operation, and the process is controlled by observing the sp. gr. of the mixture, a monochloride-content of 20% being indicated by a sp. gr. of 0.820. Fractional distillation of the mono-

chlorides affords an opportunity of rejecting material of unsuitable b.pt., and the monochloride product thus obtained (b.pt. 95°—140° C.) will contain about 90% of actual monochloride. The chloropentane is converted into amyl acetate by heating under pressure with sodium acetate and acetic acid in the presence of a solvent (alcohol, acetone, or amyl acetate); heating for 5 to 9 hrs. at 195° to 200° C. under a pressure of 180 to 230 lb. gives a yield of amyl acetate varying from 55 to 63% of the theoretical quantity. This synthetic amyl acetate cannot be distinguished from ordinary amyl acetate as regards its solvent power for cellulose esters, etc. It is doubtful if the amylenes, obtained as a by-product in the acetylation of chloropentane, can be converted profitably into alcohols or acetates. The estimated costs of producing 600 gallons of synthetic amyl acetate daily are given as:

	Dollars
Gasoline, 990 galls. at 40 cents ...	396.0
Chlorine, 5200 lb. at 2.5 cents ...	130.0
Sodium acetate, 4880 lb. at 15 cents ...	732.0
Soda ash, 1450 lb. at 3 cents ...	43.5
Interest and depreciation ...	30.3
Labour ...	48.0
Fuel and power ...	40.0
Total ...	1,419.8

—W. P. S.

*Acetone [in methyl alcohol]; Determination of —.* A. J. Field. J. Ind. Eng. Chem., 1918, 10, 552.

KEBLER'S modification of the Robineau and Rollin method (this J., 1897, 569) was found to yield the most accurate results; the presence of alcohol and the time of shaking did not influence the percentage of acetone. Messinger's method (this J., 1889, 138) yielded erratic results even when arsenious acid was used in place of thiosulphate for titrating the unused iodine (see Vaubel and Scheuer, this J., 1905, 252).—W. P. S.

*Essential oils; Determination of — in non-alcoholic flavouring extracts.* F. M. Boyles. J. Ind. Eng. Chem., 1918, 10, 537—539.

The so-called non-alcoholic flavouring extracts consist of an emulsion of the respective essential oils in mucilage of acacia, tragacanth, or other gums; glycerol is often present. To determine the essential oil in lemon, orange, almond, anise, and nutmeg extracts, 10 c.c. of the emulsion is measured in a graduated cylinder, transferred as completely as possible to a 50 c.c. flask, the cylinder is rinsed with 95% alcohol and, by the aid of a glass rod, all the precipitated gum, etc., is brought into the flask; the contents of the latter are then diluted with alcohol to 50 c.c., mixed, filtered after 30 mins., and the essential oil determined by the American official method in an aliquot portion of the filtrate. Direct steam distillation of the extract gives satisfactory results in the case of lemon, orange, and peppermint extracts provided that control distillations are made using extracts of known essential oil content to ascertain the quantity of essential oil recovered: this usually amounts to 90—95% of the amount present. In the case of cassia, cinnamon, and clove extracts the following method may be used:—10 c.c. of the extract is diluted to 50 c.c. with 95% alcohol, filtered, and 10 c.c. of the filtrate mixed with 50 c.c. of water and 1 c.c. of hydrochloric acid (1:1) and then extracted several times with ether. The ethereal solution is filtered through cotton wool, evaporated on a water-bath, the residue dried in a desiccator for 3 mins., and weighed.—W. P. S.

*Constitution of dicyanodiamide.* Hale and Vibrans. See XVI.

# PATENTS.

*Para-aminophenol; Manufacture of derivatives of —.* O. Imray, London. From Society of Chemical Industry in Basle, Switzerland. Eng. Pat. 116,920, June 25, 1917. (Appl. No. 9101 of 1917.)

SEE U.S. Pat. 1,263,238 of 1918; this J., 1918, 349A. *p*-Lactylaminophenol allyl ether and  $\alpha$ -bromoisovaleryl-*p*-aminophenol allyl ether form lustrous leaflets melting at 87° C. and 131° C. respectively. Isovaleryl-*p*-aminophenol allyl ether forms small lustrous needles, m.pt. 95° C.

*Local anesthetics; Preparation of substances for use as —.* H. Sefton-Jones, London. From N. Nagai, Tokyo-fu, Japan. Eng. Pat. 117,486, July 14, 1917. (Appl. No. 10,237 of 1917.)

The preparation of two new alkaloids, "allocain S" (O-benzoyldiethylmydriatin) and "allocain A" (O-benzoyl ethylmydriatin), both possessing local anæsthetic properties, is claimed. Mydriatin ( $\alpha$ -phenyl- $\beta$ -aminopropanol) in the form of its sulphate or hydrochloride is warmed with an ethyl halide in aqueous solution in the presence of sodium carbonate under a reflux condenser at 30°–40° C. for several hours. Sufficient ether is then added, after cooling, to dissolve the ethyl- and diethylmydriatin, and to the mixture a benzoyl halide or benzole anhydride is added and the mixture is vigorously shaken. The ethereal layer is separated and to it dilute hydrochloric acid is gradually added and the mixture shaken until the aqueous layer is acid. The ethereal and dilute acid solutions are separated and on evaporating the latter under reduced pressure "allocain S" crystallises out in the form of its hydrochloride. The ethereal solution, which contains N-benzoyl ethylmydriatin, is evaporated to remove the ether and the residue is heated in a water bath with 30% hydrochloric acid, and finally diluted with water until no further turbidity occurs. The turbid solution is filtered and the filtrate shaken with ether. The acid solution is separated from the ether, filtered if necessary, and evaporated on a water bath, during which process the N-benzoyl derivative is converted into an O-benzoyl derivative, and "allocain A" crystallises out as its hydrochloride, and may be purified by recrystallisation from water.—W. G.

*1,2,4-Methylhydroxyisopropylbenzene [carvacrol]; Process of producing —.* R. H. McKee, Ridgefield Park, N.J. U.S. Pat. 1,265,800, May 14, 1918. Date of appl., Apr. 7, 1917.

"SPRUCE turpentine" (a by-product from the manufacture of wood pulp; this J., 1918, 296A, 364A) is sulphonated, and an alkali salt of the sulphonic acid formed is fused with caustic alkali, and the dissolved melt treated with acid.—J. H. L.

*Dimethylaminotetraminoarsenobenzene; Process of preparing stable solutions of —.* L. Aeh, Mannheim, A. Rothmann, Heidelberg, and G. Giensa, Hamburg, Assignors to Farb. vorm. Meister, Lucius, und Brünning, Höchst, Germany. U.S. Pat. 1,265,864, May 14, 1918. Date of appl., Jan. 10, 1914.

A SALT of dimethylaminotetra-aminoarsenobenzene is treated with a soluble bicarbonate in presence of water. The solution formed, which probably contains the substance in the form of carbamate, has a yellowish colour which darkens on exposure to air, and yields dimethylaminotetra-aminoarsenobenzene on treatment with hydrochloric acid. (Cp. Ger. Pat. 269,660; this J., 1914, 374.)—J. H. L.

*Bismethylaminotetraminoarsenobenzene; Process of preparing derivatives of — which are soluble in water.* L. Aeh and H. Dieterich, Mannheim, Assignors to Farb. vorm. Meister, Lucius, und Brünning, Höchst, Germany. U.S. Pat. 1,265,865, May 14, 1918. Date of appl., June 15, 1915.

DERIVATIVES of 4,4'-bismethylamino-3,5,3',5'-tetra-aminoarsenobenzene substituted in the benzene nucleus, e.g., the 2,2'-dichloro-derivative, are dissolved in water by means of an alkali bicarbonate or by successive treatment with an alkali and carbon dioxide. (Cp. Eng. Pat. 8759 of 1915; this J., 1915, 1072.)—J. H. L.

*Surgical dressings; Process for manufacturing —.* K. J. Holliday, Glen Osborne Borough, Pa., Assignor to Holliday Chemical Co., Neville Island, Pa. U.S. Pat. 1,268,076, May 28, 1918. Date of appl., Mar. 29, 1918.

PARAFFIN wax and beeswax are mixed in the proportion of 95 to 65% of the former and 5 to 35% of the latter, and the mixture, with or without the addition of 0.1–5% of a gum of the elemi group, is emulsified with a base. The waxes and soaps of the emulsion are separated and then the soaps eliminated. A composition of paraffin and the esters and hydrocarbons of beeswax, having a m.pt. of 120°–140° F. (49°–60° C.), is thus obtained, which contains practically no free acids and is capable, when melted and applied to the flesh, of forming an elastic, non-cracking, and non-irritating covering.—W. G.

*Defibrinated-blood antitoxin; Process for refining —.* M. Dorset, Washington, D.C., and R. R. Henley, Tukoma Park, Md. U.S. Pat. 1,270,270, June 25, 1918. Date of appl., Feb. 27, 1918. (Dedicated to the public.)

DEFIBRINATED blood antitoxin is subjected to heat, and the coagulated hemoglobin, cell residues, and bacteria are removed by filtration without injury to the serum albumins, serum globulins, and antibodies. These three latter are then precipitated by acids, removed by filtration, redissolved, and reprecipitated, a refined, concentrated, and sterile antitoxin being thereby obtained. The process may be applied to phenolised hog cholera defibrinated blood antitoxin.—W. G.

*Blood-serum; Process for the separation of —.* M. Dorset, Washington, D.C., and R. R. Henley, Tukoma Park, Md. U.S. Pat. 1,270,271, June 25, 1918. Date of appl., Mar. 25, 1918. (Dedicated to the public.)

A NEUTRAL chemical salt is added to defibrinated blood, thereby causing a contraction of the cells and thus diminishing their volume and increasing the volume of the clear fluid without precipitation of the fibrinogen, serum globulins, or other soluble constituents.—W. G.

*Glycols; Manufacture of — [from oil-gas].* H. Ilbber, Pittsburgh, Pa. U.S. Pat. 1,270,759, June 25, 1918. Date of appl., Oct. 15, 1915.

OIL-GAS is cracked by subjecting it to a temperature of 600°–900° C., and the resulting gaseous mixture is chlorinated. The chlorine compounds are saponified by heating the mixture under pressure with aqueous alkali carbonate, the product being a mixture consisting essentially of ethylene-, propylene-, and butylene-glycols.—W. G.

*Process of hydrogenation.* U.S. Pat. 1,266,782. See XII.



## XXIII.—ANALYSIS.

*Phosphoric acid; Estimation of — as ammonium phosphomolybdate.* A. Villiers. *Bull. Soc. Chim.*, 1918, 23, 305—306.

THE author prefers his method (this J., 1893, 787) in which the phosphoric acid is precipitated and weighed as ammonium phosphomolybdate under definite conditions as more accurate than that of Clarens (this J., 1918, 352 A) in which the nitrogen in the ammonium phosphomolybdate precipitate is estimated and from that result the percentage of phosphoric acid calculated.—W. G.

*Radium; The bisulphate method of determining —.* H. H. Barker. *J. Ind. Eng. Chem.*, 1918, 10, 525—527.

THE mineral containing radium is mixed with sodium or potassium bisulphate and fused in a hard glass tube, the heating being continued long enough to expel all traces of radium emanation; the sides of the tube are washed down by introducing a small quantity of bisulphate and allowing it to fuse and run down the sides of the tube. The latter is closed with a stopper carrying two tubes, one extending to within an inch of the melt and the other just through the stopper. After the emanation has been allowed to accumulate for a given definite period, the tube is connected with the evacuated ionisation chamber of an electro-scope, bulbs containing sulphuric acid and concentrated sodium hydroxide solution, respectively, being inserted between the tube and the chamber; the air admitted to the tube passes through a sulphuric acid bulb. The tips of the tubes in the stopper are not broken until all the connections have been made. If the material under examination contains thorium, the gas must not be transferred directly to the electro-scope during fusion, but must be collected in a gas-burette to allow the decay of the thorium emanation.—W. P. S.

*Osmium; A new reaction of —.* L. Tschugaeff. *Comptes rend.*, 1918, 167, 235.

WHEN a solution containing osmium in the form of its tetroxide or as an osmichloride is warmed for a few minutes with an excess of thiourea and a few drops of hydrochloric acid, the liquid acquires a deep red or a rose colour according to the concentration of the osmium. By this means the metal may be detected at a dilution of 1 in 100,000.—W. G.

*Organic matter; New method of rapid destruction of —.* P. Duret. *Comptes rend.*, 1918, 167, 129—130.

THE material is immersed in 10% sulphuric acid in a large flask, treated with 10—20 grms. of ammonium persulphate, and the mixture heated to boiling. When effervescence ceases a further 10—20 grms. of ammonium persulphate is added and the heating continued, this being repeated until all brown colour has disappeared and white fumes appear. After cooling, the mass is treated with water. The solution can be used for the detection of metals or metalloids. The method is simple and rapid and is applicable to urines, hairs, and wool, and to such substances as sugars, fats, glycerol, and cecodyl compounds.—W. G.

*Formol titration method; Abnormalities in the —.* S. L. Jodidi. *J. Amer. Chem. Soc.*, 1918, 40, 1031—1035.

AN explanation is given of the abnormalities which sometimes render untrustworthy Sørensen's formol titration method of estimating amino-acids, poly-

peptides, etc. The function of the formaldehyde is to convert the basic nitrogen in the molecule into a neutral group so that the carboxyl group can be accurately titrated. Irregularities occur when this function is incompletely performed or when the molecule contains other groups such as the phenol or guanidine group. (See also *J. Chem. Soc.*, Sept., 1918.)—C. S.

*Electrometric titrations of solutions containing protein; Method for making —.* J. C. Baker and L. L. Van Slyke. *J. Biol. Chem.*, 1918, 35, 137—145.

APPARATUS is described which permits of the electrometric titration of solutions containing protein by a reagent, the local chemical action of which is prevented by means of a rapidly-revolving mechanical stirrer. (See further, *J. Chem. Soc.*, Sept., 1918.)—H. W. B.

*Ammonia; Determination of — in urine, serum, etc.* H. Wiessmann. *Landw. Versuchs.-Stat.*, 1918, 91, 347—352.

TEN c.c. of the sample is mixed with 40 c.c. of water and distilled at 50°C. under 5—10 mm. pressure with the addition of 10 c.c. of 10% sodium carbonate solution; the distillate is collected in a definite quantity of standardised sulphuric acid. The distillation of the ammonia is complete in about 45 mins. Urea does not yield any ammonia under these conditions. (See also *J. Chem. Soc.*, Sept., 1918.)—W. P. S.

*Dubrisay's method of testing [viscosity of] lubricating oils.* Nicolardot and Masson. *See* IIa.

*Determining specific gravity of viscous tar, etc., at different temperatures.* Myhill. *See* III.

*Estimation of mixtures of thiosulphuric, sulphurous, trithionic, and sulphuric acids.* Billeter and Wavre. *See* VII.

*Determination of acetic acid by distillation with phosphoric acid.* Munn. *See* VII.

*Estimation of ammonia and hydrochloric acid by weighing as ammonium chloride.* Villiers. *See* VII.

*Analysis of ammoniacal liquors.* *See* VII.

*Method of analysing mixtures of bicarbonates and carbonates, or alkali carbonates and hydroxides.* Clarens. *See* VII.

*Measurement of the time of set of calcined gypsum.* Emley. *See* IX.

*Instrument for measuring plasticity.* Emley. *See* IX.

*Standard apparatus for the determination of sulphur in iron and steel.* Pulsifer. *See* X.

*Rapid determination of carbon in steel by the barium carbonate titration method.* Cain and Maxwell. *See* X.

*Determination of copper minerals in partly oxidised ores.* Cremer. *See* X.

*Sulphide precipitation of group 2a metals.* Shibko. See X.

*Nephelometric values of cholesterol and the higher fatty acids.* Csonka. See XII.

*Determination of free sulphur in soft rubber compounds.* Upton. See XIV.

*Method for counting certain protozoa in the soil.* Itano and Ray. See XVI.

*Rate of production of colour in alkaline solutions of dextrose and picrate.* Addis and Shevky. See XVII.

*Institute of Brewing method for determination of the moisture content of barley.* See XVIII.

*Determinations of acidity in barley by titration in stages.* Relchard. See XVIII.

*Determination of oxalic acid in beer [urine, etc.].* Bau. See XVIII.

*Colorimetric determination of lactose in milk.* Pacul and Russell. See XIXa.

*Micro-Dumas estimation of nitrogen in liquids poor in nitrogen [c.g., milk].* Kraemer. See XIXa.

*Detection of vegetable gums in food products.* Cook and Woodman. See XIXa.

*Determination of carbon dioxide in baking powder.* Schellbach and Bodlnus. See XIXa.

*Uniform nitrogen determination in cottonseed meal.* McLargue. See XIXa.

*Composition of lime-sulphur solutions.* Winter. See XIXb.

*Identification of the cinchona alkaloids by optical crystallographic measurements.* Wherry and Yanovsky. See XX.

*Detection and determination of coumarin in factitious vanilla extracts.* Wichmann. See XX.

*Detection and identification of malonic acid.* Bougault. See XX.

*Determination of acetone [in methyl alcohol].* Field. See XX.

*Determination of essential oils in non-alcoholic flavouring extracts.* Boyles. See XX.

#### PATENTS.

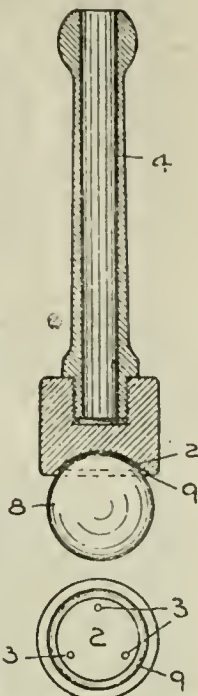
*Viscosity of fluids; Method of determining the — and means for use in connection with the same.* A. G. M. Michell, Melbourne. Eng. Pat. 117,234, Feb. 21, 1918. (Appl. No. 3124 of 1918.) Under Int. Conv., July 12, 1917.

The viscosity of a fluid, e.g., oil, is measured by the time taken to detach, by a definite force, two

contiguous surfaces separated by a film of fluid.

A hollow stem, 4, is provided with a shallow cup, 2, at one end, the spherical surface of the cup having three slight projections, 3, symmetrically placed and about 1—2 mm. diameter and 0.01 mm. in height. The rim of the cup is recessed with a groove, 9, of slightly larger diameter. The cup is placed over a corresponding steel ball, 8, with a film of oil to be tested between them, and a thermometer may be placed in the hollow stem. The cup is lifted and the time taken for the ball to become detached is observed. The recessed groove being filled with oil, maintains the film between the surfaces during their separation. In a modification, the cup is inverted and the ball attached to one arm of a balance and counter-weighted. A definite lifting force is then applied by adding a known weight to the other arm of the balance, and the time determined as before. The viscosity is directly proportional to the time. The viscosity of a gas may be determined by supplying it to the capillary space between the ball and the cup through a passage in the base of the cup.

—W. F. F.



*Hardness testing machines.* W. and T. Avery, Ltd., and J. Dobson, Birmingham. Eng. Pat. 117,526, Aug. 30, 1917. (Appl. No. 12,453 of 1917.)

The specimen to be tested is mounted on a plate supported by the short arm of a lever, the long arm of which is connected through a system of levers to the short arm of a steelyard. The movement of the steelyard is limited by stops. The short arm is also connected through levers to an indicating mechanism, e.g., a pendulum or spring balance. The application of the test weight on the long arm of the steelyard presses a steel ball between the specimen below and a plate carried by a vertical screw above. The indicating pointer is deflected by the indentation of the specimen and is brought back to zero by screwing down the upper plate, the load on the steel ball due to the test weight being then a maximum.—W. F. F.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Baines. Composition for insulation of steam and hot-water pipes. 13,546. Aug. 21.

Becker and Olson. Composition of matter. 14,120. Aug. 30.

British Westinghouse Electric and Manufacturing Co. Condensers of the jet type. 13,990. Aug. 28. (U.S., Sep. 29, 1917.)

British Westinghouse Electric and Manufacturing Co. Steam-actuated ejectors. 13,991. Aug. 28. (U.S., Sep. 21, 1917.)



British Westinghouse Electric and Manufacturing Co. Condensers for steam, etc. 13,992. Aug. 28. (U.S., Oct. 1, 1917.)

Brown (Bangkok Dock Co.). Devices for removal of ash, dust, etc., from gases, etc. 13,741. Aug. 23.

Cowburn, and Cowburn and Cowpar. Carboy hampers or crates. 13,890. Aug. 27.

Crosfield and Sons, and Hilditch. Manufacture of catalysts. 14,096. Aug. 14.

Cruyt, and Heenan and Froude. Apparatus for treating and cooling air or gas. 13,905. Aug. 27.

Davis. Pulverisers. 13,964. Aug. 28.

Erith, and Erith's Engineering Co. 14,057. *See* II.

Fawcett, Preston, & Co., and Harris. Apparatus for separation of vapours or gaseous fluids from liquids. 13,962. Aug. 28.

Goldreich. Ovens. 13,771. Aug. 23.

Graemiger. Device for measuring or controlling velocity of flowing fluid. 13,609. Aug. 21. (Switz., Aug. 31, 1917.)

Hind. Mixing and kneading appliances. 13,494. Aug. 20.

Hoofnagle. 13,607. *See* XI.

Kent and Mulliner. 13,678. *See* II.

Kent-Johnston. Apparatus for drying vegetable, animal, or mineral substances. 13,955. Aug. 28.

Mason. Filter-presses. 13,557. Aug. 21.

Pradaïrol. Apparatus for purifying and drying steam. 14,098. Aug. 30. (Fr., July 7, 1917.)

Robertson. Compressed-air liquid-spraying machine. 13,857. Aug. 26.

Stein et Cie. Regenerative furnaces. 14,177. Aug. 31. (Fr., Sep. 8, 1917.)

Thunholm. Evaporating-apparatus. 13,760. Aug. 23. (Sweden, Aug. 23, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,258 (1917). Lea. Registering-apparatus chiefly for use in registering the amount of feed of granular and like substances. (118,333.) Sep. 4.

12,281 (1917). Burns. Process and apparatus for separation of suspended particles from gases. (118,334.) Sep. 4.

15,525 (1917). Barbet et Fils et Cie. Cooling-plates for continuous rectification apparatus. (110,910.) Aug. 28.

17,609 (1917). Bruce and Baynes. Apparatus for the transmission or exchange of heat. (118,375.) Sep. 4.

9444 (1918). Matousek. Centrifugal separators. (118,262.) Aug. 28.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING

#### APPLICATIONS.

Bates. Gas-producers. 13,673, 13,674. Aug. 22.

Bowler and others. 13,941. *See* XII.

Brown (Bangkok Dock Co.). 13,741. *See* I.

Brown and Galloway. Fuel briquettes, etc. 13,899. Aug. 27.

Burney. Apparatus for distillation of solid carbonaceous material. 14,176. Aug. 31.

Cruyt, and Heenan and Froude. 13,905. *See* I.

Corbushley. 14,022. *See* XIII.

Davidson and Liversedge. Gas-cleaning apparatus. 13,938. Aug. 27.

Docking, and Sharp and Preston. 13,715. *See* XVIII.

Docking, and Sharp and Preston. Gas and air mixing apparatus for producer or suction gas generators, etc. 13,777. Aug. 24.

Edwards. 13,531. *See* V.

Erith, and Erith's Engineering Co. Apparatus for drying and carbonising materials. 14,057. Aug. 29

Evans. Gas producers, etc. 13,957. Aug. 28.

Greensill. Oil fuel. 13,631. Aug. 22.

Hamon. Fuel. 13,533. Aug. 20.

Kent and Mulliner. Settings for retorts. 13,678. Aug. 22.

Levi. Refractory blocks, fuels, radiators, etc. 13,710. Aug. 23.

Maclaurin. Manufacture of lubricating greases. 13,831. Aug. 24.

McLeod. Apparatus for extracting products from peat, carbonaceous substances, etc. 13,638. Aug. 22.

Maxfield. Incandescent gas lighting. 13,684. Aug. 22.

Morris. 13,859. *See* III.

Oakbank Oil Co., and Wishart. 13,842. *See* VII.

Parkes and Pearson. Manufacture of mixtures of mineral oils and coal tar. 13,610. Aug. 21.

Piette, and Soc. Franco-Belge de Fours à Coke. Coking-ovens. 13,670. Aug. 22.

Pryce-Jones. Manufacture of briquettes. 13,782. Aug. 24.

Silberrad. 13,699. *See* XX.

Stein et Cie. 14,177. *See* I.

Swinburne. 13,951. *See* XII.

Ten Bosch. Removing water from peat, etc. 13,463. Aug. 19. (Holland, July 3.)

Wellington. Apparatus for quenching coke. 13,452. Aug. 19.

Wellington. Apparatus for mixing gaseous fuels. 13,737. Aug. 23.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,764 (1916). Maxim. Conversion of heavy hydrocarbons into lighter hydrocarbons. (118,122.) Aug. 28.

11,891 and 16,806 (1917). Ionides. Gaseous combustion. (118,313.) Sep. 4.

12,151 and 12,722 (1917). Ottini. Apparatus for storing coal gas and the like for use in motor vehicles. (118,327.) Sep. 4.

13,784 (1917). Marks (Swan Process Oil Co.). *See* XII.

4812 (1918). Yates, Brayshaw, and Brayshaw. Burner for gas-fired furnaces. (118,239.) Aug. 28.

## III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Morris. Process by which coal tar and/or dehydrated coal tar may be utilised in conjunction with mineral and/or shale oils. 13,859. Aug. 26.

Parkes and Pearson. 13,610. *See* II.

## IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

Higgins. Solid and soluble dyes. 14,009. Aug. 29.

Soc. Chimique des Usines du Rhône, anc.

Gilliard, P. Monnet, et Cartier. Process for obtaining halogen derivatives of indigo and of its homologues. 13,618. Aug. 21. (Fr., Oct. 10, 1917.)

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Brunner. Manufacture of spinning and weaving material. 13,612. Aug. 21. (Holland, Oct. 20, 1917.)

Cater. Process of water-proofing and/or air-proofing, and material therefor. 13,719. Aug. 23.

Clark (Jespersen Paper Co.). Method of removing printers' ink from paper stock. 14,170. Aug. 31.

Edwards. Manufacture of an insulating material and a fuel from paper, etc. 13,531. Aug. 20.

Finch and Whiteside. Absorbent writing-paper. 13,901. Aug. 27.

Macdonald. Manufacture of vulcanised fibre. 14,125. Aug. 30.

Reid. Coating or impregnating thread, yarn, ropes, fabrics, etc. 14,192. Aug. 31.

Rinman. Process of retting fibrous substances. 14,104. Aug. 30.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3651 (1917). Moore. Manufacture of pulp for making paper, paper boards, and the like. (118,291.) Sep. 4.

12,229 (1917). Jarmain. Process for the separation of grease from wool-scouring or like effluents or liquors. (118,332.) Sep. 4.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATIONS.

Calico Printers' Association, and Roberts. Bleaching, dyeing, etc., fabrics. 14,147. Aug. 31.  
Poulson. Sizing textile fabrics, etc. 13,785. Aug. 24.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

Bergman and Larson. Manufacture of pure aluminium hydroxide and ammonium sulphate. 13,873. Aug. 26. (Sweden, Oct. 9, 1917.)

Bloch and Dreyfus. Process of manufacturing alkaline manganates. 13,792. Aug. 24.

Bloch and Dreyfus. Process of manufacturing potassium permanganate. 13,793. Aug. 24.

Carborundum Co. (Tone). Purified crystalline alumina, and method of making same. 13,998. Aug. 28.

Clarke, and Rare Metals, Ltd. Process for production of ammonia, ammonium hydrate, and ammonium salts. 13,471. Aug. 19.

Clarke, and Rare Metals, Ltd. Process for production of nitric and nitrous acids and manufacture of nitrates. 13,754. Aug. 23.

Cowper-Coles. Method of extraction of nitrogen from the air, etc. 13,886. Aug. 27.

Curphery. Production of chemical compounds. 13,596. Aug. 21.

Dunningham and Hargreaves. Manufacture of chlorides of sulphur. 13,812. Aug. 24.

Fellowes and Hyatt. Process for obtaining products from seaweed. 13,524. Aug. 20.

Ingham. 13,837. *See* XVII.

Lytle. Manufacture of lead compounds. 13,436. Aug. 19.

Oakbank Oil Co., and Wishart. Manufacture of sulphate of ammonia. 13,842. Aug. 26.

#### COMPLETE SPECIFICATIONS ACCEPTED.

11,887 (1917). Dutt and Dutt. Process for the manufacture and production of potassium aluminate, alumina, and potassium carbonate. (118,155.) Aug. 28.

11,888 (1917). Dutt and Dutt. Process for the manufacture and production of sodium aluminate, alumina, and sodium carbonate. (118,156.) Aug. 28.

13,884 (1917). Statham. *See* XI.

#### VIII.—GLASS; CERAMICS.

##### APPLICATIONS.

Carborundum Co. (Tone). 13,998. *See* VII.

Darrah. Glazed bricks. 13,838. Aug. 26.

Ferneyhough. Utilisation of heat from potters' ovens, kilns, etc. 13,883. Aug. 27.

Levl. 13,710. *See* II.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,117 (1917). Kiefer. *See* IX.

6398 and 6399 (1918). Haddan (Corning Glass Works). Manufacture of glass for absorbing ultra-violet radiations. (118,397 and 118,398.) Sep. 4.

#### IX.—BUILDING MATERIALS.

##### APPLICATIONS.

Darrah. 13,838. *See* VIII.

Hines and Scott-Moncrieff. Cement. 13,567. Aug. 21.

#### COMPLETE SPECIFICATION ACCEPTED.

12,117 (1917). Kiefer. Bricks or building blocks. (114,137.) Sep. 4.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

##### APPLICATIONS.

Alexander, Imbery, and Vint. Electrical device for automatically indicating correct temperature of electrically-heated steel before quenching to obtain correct hardening thereof. 13,698. Aug. 22.

Bailey and Hall. Furnaces for melting steel, etc. 14,139. Aug. 31.

Bodman. Metallic alloy. 14,149. Aug. 31.

Cowper-Coles. Process for Sherardising. 13,885. Aug. 27.

Henderson. Process for removing coatings from metals. 13,932. Aug. 27.

Jascowitz and Rosenbaum. Cleaning metals by electrolysis. 13,642. Aug. 22.

Levin. Means for recovery of solder from tin cans, etc. 14,000. Aug. 28.

Lord. Furnaces for heat treatment of metals. 14,113. Aug. 30.

Marino. Process for removing paint, varnish, and grease from iron, steel, and copper, rust from iron or steel, and oxide or sulphide from copper. 13,665. Aug. 22.

Morley. Preservative preparations for protection of metal surfaces. 13,663. Aug. 22.

Whibley. Welding metals. 14,131. Aug. 30.

Williams. Manufacture of armour plating. 13,429. Aug. 19.

#### COMPLETE SPECIFICATIONS ACCEPTED.

6846 (1917). Cooper Co. Alloys. (113,259.) Aug. 28.

7329 (1917). Pescatore. *See* XI.

11,511 (1917). Cary. Electric welding. (118,138.) Aug. 28.

11,865 (1917). Welford. Production of aluminium. (118,312.) Sep. 4.

12,912 (1917). Harvey, and Morgan Crucible Co. Crucible melting-furnaces. (118,346.) Sep. 4.

13,635 (1917). Avery, Ltd., and Allcut. *See* XXIII.

14,336 (1917). Burnett. Chemically depositing chromium upon metals, particularly iron. (118,182.) Aug. 28.

17,368 (1917) and 8586 (1918). Wrightson, Rinquist, and Head, Wrightson, and Co. Blast-furnace stoves. (118,203.) Aug. 28.

18,053 (1917). Godfrey and Way. Manufacture of tin-plate. (118,377.) Sep. 4.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

Alexander and others. 13,698. *See* X.

Edwards. 13,531. *See* V.

Hoofnagle. Treating gases and vapours electrically. 13,607. Aug. 21.

Jascowitz and Rosenbaum. 13,642. *See* X.

Jungner. Primary galvanic batteries with unalterable electrolyte. 14,106. Aug. 30. (Sweden, Sep. 4, 1917.)

Keene. Electric furnaces. 14,079. Aug. 30. (U.S., Sep. 5, 1917.)

Ridoni, and Soc. Talco e Grafite Val Chisone. Manufacture of natural graphite electrodes. 13,984. Aug. 28. (Italy, Mar. 6.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

7329 (1917). Pescatore. Electrodes for arc soldering. (118,296.) Sep. 4.

11,511 (1917). Cary. *See* X.

13,884 (1917). Statham. Electrolytic cells, adapted for the manufacture of caustic alkali from sodium chloride. (118,355.) Sep. 4.



13,956 (1917). Wade (Booth-Hall Co.). Electric furnaces. (118,357.) Sep. 4.  
 17,942 (1917). Whitney. Electric secondary or storage batteries. (118,208.) Aug. 28.  
 1907 (1918). Johnson (Commercial Truck Co.). Secondary or storage cells or batteries. (118,227.) Aug. 28.

3460 (1918). Travis, and Watson and Co. Supply of electrical energy to furnaces or other appliances. (118,233.) Aug. 28.

## XII.—FATS; OILS; WAXES.

### APPLICATIONS.

Bowler, Paterson, and Paterson's Productions, Ltd. Treatment of oils, vegetable fermented liquors, etc. 13,941. Aug. 27.

Dicker (Devik and Lehne). Process of recovering fat or oil from fatty substances. 14,001. Aug. 28.  
 Maclaurin. 13,831. *See* II.  
 Swinburne. Treatment of oils. 13,951. Aug. 28.

### COMPLETE SPECIFICATIONS ACCEPTED.

12,032 (1917). Kimura. Preparing catalytic agents for hydrogenating oils. (118,323.) Sep. 4.

12,220 (1917). Jarman. *See* V.

13,784 (1917). Marks (Swan Process Oil Co.). Process and apparatus for purifying oils. (118,353.) Sep. 4.

18,546 (1917). Talbot. *See* XIX.

2057 (1918). Barry, Ostlere, and Shepherd, and Barry. Treatment of linseed-oil fatty acids. (118,228.) Aug. 28.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

### APPLICATIONS.

Curbishley. Manufacture of carbon black. 14,022. Aug. 29.

Denniss. Colours for artists. 13,821. Aug. 24.

Henderson. 13,932. *See* X.

Marino. 13,665. *See* X.

Morley. 13,663. *See* X.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

### COMPLETE SPECIFICATIONS ACCEPTED.

11,473 (1917). Mellersh-Jackson (Morgan and Wright). Treatment of india-rubber and the like. (118,305.) Sep. 4.

4801 (1918). Moore. Device or apparatus for the drying or heat treatment of rubber or other material or articles. (118,394.) Sep. 4.

## XVII.—SUGARS; STARCHES; GUMS.

### APPLICATIONS.

Ingham. Manufacture of algin. 13,837. Aug. 26.  
 Jacobson and Wrathall. Washing and purifying sugar. 13,778. Aug. 24.

Pictet. Manufacture of levoglucosan. 13,530. Aug. 20. (Switz., Dec. 27, 1917.)

## XVIII.—FERMENTATION INDUSTRIES.

### APPLICATIONS.

Bayer and Orla-Jensen. Process for production of alcohol and yeast from seaweed. 14,946. Aug. 29. (Denmark, Aug. 16, 1917.)

Bowler and others. 13,941. *See* XII.

Docking, and Sharp and Preston. Gas-heated

cylinders for drying or roasting malt, etc. 13,715. Aug. 23.

### COMPLETE SPECIFICATION ACCEPTED.

15,525 (1917). Barbet et Fils et Cie. *See* I.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

### APPLICATIONS.

Buckley. Preserving meat, etc. 13,427. Aug. 19.  
 Hegney and Hegney. Sterilising milk. 13,656. Aug. 22.

Kent-Johnston. 13,955. *See* I.

Marshall. Process for sterilising and drying animal and vegetable matter. 13,774. Aug. 23.

West. Method of treating grain for preservation thereof. 13,811. Aug. 24.

### COMPLETE SPECIFICATION ACCEPTED.

18,546 (1917). Talbot. Butter substitutes and the like, and the preparation of same. (118,215.) Aug. 28.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

### APPLICATIONS.

Confectionery Ingredients, Ltd., Kane, King, and Matthews. Manufacture of vanillin. 13,504. Aug. 20.

Confectionery Ingredients, Ltd., Kane, King, and Matthews. Manufacture of protocatechuic aldehyde. 13,505. Aug. 20.

Ingham. 13,837. *See* XVII.

Silberrad. Manufacture of ethylene. 13,699. Aug. 22.

Stevenson. Manufacture of acetic anhydride. 13,640. Aug. 22.

### COMPLETE SPECIFICATION ACCEPTED.

10,183 (1917). Marks (Nagai). Manufacture of synthetic drugs. (118,298.) Sep. 4.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

### APPLICATIONS.

Barron and Barron. Method of coating photographic sensitised glass plates. 13,488. Aug. 20.

Barron and Barrou. Method of stripping films off old negatives. 13,489. Aug. 20.

Camiller and Lyell. Multi-colour screens for natural-colour cinematography, and manufacture thereof. 13,461. Aug. 19.

Camiller and Lyell. Multi-colour screens for natural-colour photography, and manufacture thereof. 13,462. Aug. 19.

Stevenson. Manufacture of non-inflammable films or filaments for cinematography, etc. 13,569. Aug. 21.

## XXII.—EXPLOSIVES; MATCHES.

### COMPLETE SPECIFICATION ACCEPTED.

12,009 (1917). Aktiebolaget Carlit. Ammonium perchlorate explosives. (112,417.) Sep. 4.

## XXIII.—ANALYSIS.

### COMPLETE SPECIFICATION ACCEPTED.

13,635 (1917). Avery, Ltd., and Allcut. Impact-testing machines. (118,175.) Aug. 28.

ERRATUM.—This Journal, Aug. 15, 1918, page 452 A, col. 1, line 12 from top, after "Gregory" insert "Morrison."

## I.—GENERAL; PLANT; MACHINERY.

## PATENTS.

*Evaporating and distilling apparatus [for water].* A. Duoda, Genoa, Italy. Eng. Pat. 111,480, Nov. 9, 1917. (Appl. No. 16,423 of 1917.) Under Int. Conv., Nov. 16, 1916.

The apparatus consists of an evaporating box of trapeziform section with rounded corners, containing a nest of heating coils. The coils communicate with one another through headers, and are arranged to conform with the shape of the box. The coils extend above the level of the water in the evaporating box to superheat the steam.—J. H. P.

*Rectifying and concentrating liquids; Apparatus for —.* D. S. Monteith, Assignor to Chemical Construction Co., Charlotte, N.C. U.S. Pat. 1,271,349, July 2, 1918. Date of appl., Feb. 14, 1918.

The acid or other liquid is fed down a tower into a concentrating chamber heated electrically. The concentrated liquid is run off from the chamber through a cooler and the hot gases and vapours given off pass away up the tower.—W. H. C.

*Pulp-filter.* T. L. Crane, Tyrone, N. Mex. U.S. Pat. 1,268,935, June 11, 1918. Date of appl., Apr. 5, 1917.

A rotary suction drum is supported on horizontal bearings in a casing, and a pair of circular members, supported by arms journaled in the same bearings, carry a number of angle-iron members having their apices adjacent to the drum to remove pulp from it. The circular members are oscillated by a crank and connecting rod.—W. F. F.

*Filtering process.* A. L. Genter, Assignor to The General Engineering Co., Salt Lake City, Utah. U.S. Pat. 1,269,700, June 18, 1918. Date of appl., Jan. 23, 1917.

The mixture is continuously filtered and thickened and the filter medium cleansed *in situ* by utilising the current of the mixture in one chamber to collect the solids removed from the filter medium therein and convey them to the succeeding filter chamber.—W. H. C.

*Centrifugal filtering and clarifying apparatus.* O. E. Enell, Jersey City, N.J. U.S. Pat. 1,269,067, June 11, 1918. Date of appl., Oct. 12, 1916.

The liquid to be filtered is introduced into a rotating bowl within which is a sleeve having openings communicating with the interior of the bowl. Filter frames are mounted on the sleeve and each frame has an inner receiving chamber. The base of the bowl has a number of removable partitions or baffles to retard the flow of liquid.—J. H. P.

*Centrifugal dryer.* F. B. Anderson, Assignor to C. O. and A. D. Anderson, Cleveland, Ohio. U.S. Pat. 1,271,112, July 2, 1918. Date of appl., Mar. 6, 1917.

A CONICAL screen mounted on a rotating horizontal shaft is surrounded by a casing. The material to be dried is fed into the smaller end of the screen and the dried material is arrested at the opposite end, whence it is continuously removed and discharged into a discharge spout in the casing. An off-take for water is also provided in the casing.—L. A. C.

*Separator or clarifier; Centrifugal —.* H. A. Riggs, Indianapolis, Ind. U.S. Pat. 1,271,367, July 2, 1918. Date of appl., May 3, 1916.

The bowl of the separator is mounted on a hollow central tube which tapers upwards and is provided with a conical head-piece which is enclosed by a flanged and slotted feed-tube.—W. H. C.

*Dryer.* O. C. McCormick, Cleveland, Ohio, Assignor to J. E. Moyer, Akron, Ohio, S. K. Hine, Girard, Ohio, J. W. Wright, Youngstown, Ohio, and W. P. Canfield, Pittsburgh, Pa. U.S. Pat. 1,268,983, June 11, 1918. Date of appl., Sep. 9, 1916.

The articles to be dried are carried on racks which are mounted on sprocket chains, and within the drying chamber is a series of sprocket wheels for receiving the chains. The chains pass alternately up and down from one end of the chamber to the other and also out through openings in the opposite end walls to allow of the racks being loaded and unloaded.—J. H. P.

*Furnace.* J. S. Tierney, Brooklyn, N.Y., Assignor to American Shop Equipment Co., Chicago, Ill. U.S. Pat. 1,269,132, June 11, 1918. Date of appl., Feb. 6, 1917.

Two jets of air, one or both containing fuel, are injected at one end of a mixing chamber leading to a horizontal furnace chamber and are inclined so as to impinge on one another. An opening in the mixing chamber behind the jets is traversed by a bridge, the side walls of which converge towards the rear, so that fluid under pressure supplied through the opening is directed over the upper and lower surfaces of the mixing chamber.—W. F. F.

*Furnace.* W. S. Ralya, Assignor to A. H. Bohnstengel, Cincinnati, Ohio. U.S. Pat. 1,271,361, July 2, 1918. Date of appl., Dec. 9, 1915.

A FURNACE having an arched top is heated by the combustion of a regulated mixture of fluid fuel and air, which is introduced into the furnace at one side, and in such a manner that the products of combustion follow the curve of the arched top. The waste gas passes through passages below the furnace floor to the stack.—W. H. C.

*Muffle.* J. B. Farquhar, Vancouver, B.C. U.S. Pat. 1,269,270, June 11, 1918. Date of appl., Dec. 18, 1916.

A MUFFLE is constructed in two parts, one being an arched wall and roof member with an end wall formed integrally therewith, and the other comprising the floor and being provided with an "edge bead" to receive the bottom edge of the wall. The inner edge of the bead is bevelled outwards to provide a space at the outside of the wall, and this space is filled with a sealing material. The floor is recessed to receive the wall, the inner side of the recess having a vertical wall surface which bears against the lower end of the arched wall.—J. H. P.

*Mixing dry lime with liquids; Process and apparatus for —.* J. W. Adams and O. H. Egge, Spreckels, Cal. U.S. Pat. 1,269,994, June 18, 1918. Date of appl., Feb. 24, 1917.

The liquid is contained in a vertical vessel provided with tubes, tube-plates, and a central well, similar to a vacuum evaporator. The liquid is caused to flow up the tubes and to return down the central well and the lime or other powdered material is distributed over the surface of the liquid as it flows towards the central well.—W. H. C.



*Fire-extinguishing and fireproof composition.* G. E. Ferguson, New York. Assignor to Pyrene Manufacturing Co. U.S. Pat. 1,271,506, July 2, 1918. Date of appl., Nov. 18, 1913.

THE composition consists of a mixture of substances which when exposed to heat gives off ammonia, and also produces a fireproof coating which serves to hold the ammonia about the burning material. A mixture of sodium silicate, borax, microcosmic salt, and monosodium phosphate is specified.—J. H. P.

*Drying materials; Art of —.* A. F. Wright, Newark, N.J., U.S.A. Eng. Pat. 117,704, Aug. 10, 1917. (Appl. No. 11,512 of 1917.)

SEE U.S. Pat. 1,251,317 of 1917; this J., 1918, 142A.

*Utilisation and combustion of fuel.* Eng. Pat. 117,637. See IIA.

*Apparatus for treating gases with liquids.* Eng. Pat. 117,843. See IIA.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Water-gas formation; Theory of —.* Gwosdz. Z. angew. Chem., 1918, 31, 137–140.

EXPERIMENTS were made to ascertain the parts played in water-gas formation by the supposed primary reactions (1)  $C + H_2O = CO + H_2$ , and (2)  $C + 2H_2O = CO_2 + 2H_2$ , and the secondary reactions (3)  $C + CO_2 = 2CO$ , and (4)  $CO + H_2O = CO_2 + H_2$ . Superheated steam was passed over various forms of heated carbon and the composition of the issuing gases determined. The temperatures varied from 855° down to 560° C., and the speed of the current of steam was also varied. Natural forms of carbon containing ash in appreciable quantities behaved in a markedly different manner from purified carbon almost free from ash. With gas coke (8.5% ash) and wood charcoal (1.4% ash) a considerable proportion of carbon dioxide was always formed (up to 29%). On the other hand lamp carbon (0.1% ash) at temperatures down to 600° C. gave mixtures closely resembling water-gas as manufactured. Even at 560° C. the mixture:— $CO_2$  8.6%,  $CO$  39.5%,  $H_2$  40.0%, residue 11.9% was obtained. It is concluded that reaction (2) cannot be a primary reaction, because reaction (3) is known to be too sluggish to account for the formation of the high percentage of carbon monoxide found at these low temperatures. It is also concluded that reaction (1) is the primary reaction of water-gas formation, and that the carbon monoxide formed reacts with the excess of steam tending to establish the water-gas equilibrium, and that this reaction is catalytically influenced by the finely divided ash in the carbon.

—H. J. H.

*Use of pulverised coal [in lead smelting].* Rice. See X.

*Behaviour and detection of the smallest quantities of carbon monoxide in the chloride pipette.* Hofmann and Schibsted. See XXI11.

### PATENTS.

*Fuel; Utilisation and combustion of —.* G. Helps, Nuneaton. Eng. Pat. 117,637, Nov. 22, 1916. (Appl. No. 16,709 of 1916.)

A FURNACE heated by gas or vapour, e.g., a boiler

furnace, is provided with a series of parallel pipes arranged longitudinally round the cylindrical surface to be heated, and a combustible mixture is supplied to them. A number of burner heads are mounted along the pipes, adapted to give a coneless or nearly coneless flame as described in Eng. Pat. 111,495 (this J., 1918, 48 A) and thus cover the walls with a close film of flame. Additional longitudinal pipes with radial branches extending nearly to the cylindrical surface are provided for the supply of a regulated quantity of additional air. The system of pipes is mounted on a carriage moving on rails to facilitate removal. In a modification the furnace is provided with a firebrick filling, leaving a central longitudinal passage and a number of small longitudinal troughs in contact with the furnace wall. Burners are arranged at the inlets of the troughs to project a flame along the furnace wall, and additional air is fed through small radial passages leading from the central longitudinal passage to which the air is supplied. Alternatively, the firebrick device may be in the form of an arch supported on a wheeled carriage. The flame passages may be provided with spiral baffles or perforated discs to retard the hot gases. The combustion products may subsequently be passed through a feed-water heater.—W. F. F.

*Coke-ovens; Apparatus for converting beehive-ovens into by-product —.* B. Zwillinger, New York. U.S. Pat. 1,269,895, June 18, 1918. Date of appl., July 12, 1917.

EACH oven of a row of beehive coke-ovens is provided with a transverse combustion flue, divided laterally, and situated beneath but not communicating with the oven. The air for combustion is preheated by passing it through a heating passage in the setting below the floor in front of the ovens, and then through transverse conduits centrally below the oven to the combustion flue. Combustible gas is also supplied to the combustion flue adjacent to the hot air exit. The hot gases pass from the combustion flue through return flues beneath and towards the sides of the oven, and thence through a passage adjacent to the air heating passage. The by-products are withdrawn at the top of the oven.

—W. F. F.

*Gas producer plant [for wood, etc.].* W. De C. Turner, Perth, Australia. Eng. Pat. 117,721, Sept. 11, 1917. (Appl. No. 13,041 of 1917.)

WOOD or other inferior fuel is fed into the producer by an endless conveyor operated by a hand wheel from a feeding chamber at the side, which is provided with an outer door and a communicating door so as to act as an air lock. The producer is lined with firebricks, those in one layer being hollow and connected by openings with the producer so as to act as a discharge conduit for the gas. The gas passes through a cooling and scrubbing chamber provided with baffles, and with a rotating wheel having a perforated periphery which breaks up the water supplied to the chamber. The gas passes through the wheel and then upwards through a vertical scrubber, where it meets a falling shower of water produced by a number of horizontal fans on a central vertical shaft, driven by water jets. The fan blades are constructed with upturned edges to divert the gas radially inwards to facilitate scrubbing, and with downturned flanges to receive the water jets. Each fan also carries a horizontal meshed disc through which the gas passes. The gas leaving the top of the scrubber passes upwards through a chamber containing filtering material, and thence to a storage chamber.—W. F. F.

*Gas and phosphatic fertiliser material; Process of producing* — A. F. Delacourt, Assignor to Soc. Anon. Ital. G. Ansaldo & Co., Genoa, Italy. U.S. Pat. 1,269,795, June 18, 1918. Date of appl. Apr. 11, 1918.

FUEL containing relatively large amounts of sulphides, e.g., lignite, is gasified in the presence of sufficient calcium phosphate to slag the ash and to unite with the bulk of the oxides of sulphur produced. The slag formed is suitable for use as a fertiliser.—L. A. C.

*Gas-producers; Feeding mechanism for* — C. W. Lummis, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pats. (A) 1,270,962 and (B) 1,270,963, July 2, 1918. Dates of appl., Mar. 10, 1916, and Oct. 12, 1915.

(A) The fuel is fed from a hopper into a horizontal rotary cylinder having an opening on one side which connects alternately with the hopper and the producer as the cylinder is rotated. The cylinder works in a lower fixed cylindrical seating enclosing not more than half its circumference, so that it is freely movable away from the seating. The remaining surface of the rotating cylinder is partly covered by a yielding cylindrical section which is pressed against it by springs. The seating is gas-tight in all positions of the rotating cylinder. (B) In a feeding mechanism of the type described in (A), the yielding section is replaced by several longitudinal gulding plates which prevent the escape of fuel past the opening in the rotary cylinder as it passes through the upper part of its movement towards the fixed seating.—W. F. F.

*Gases; Method of and means for storing* — F. C. Short and F. W. Moore, Walsall. Eng. Pat. 117,842. (Appl. Nos. 11,001, July 31, and 12,444, Aug. 30, 1917.)

BROKEN or powdered charcoal, preferably that obtained from coconut shells, is impregnated with a metal such as platinum, gold, palladium, iron, nickel, or cobalt, by impregnating with a suitable salt and then reducing the salt to deposit the metal in finely divided form. The product is used in a storage container as an absorbent for gases such as coal gas.—W. F. F.

*Gases; Apparatus for treating — with liquids*. N. Wilton, and The Chemical Engineering and Wilton's Patent Furnace Co., Ltd., Hendon, Middlesex. Eng. Pat. 117,843, July 31, 1917. (Appl. No. 11,039 of 1917.)

GAS, e.g., coal gas which is to be treated with water to extract ammonia, or with oil to extract benzol, is passed through a chamber divided by parallel vertical partitions into a number of compartments connected by openings near the top of the partitions. Supplementary partitions extend midway through each compartment from the top downwards for two-thirds of the height, so that the gas takes a zigzag path. That section of each compartment through which the gas flows downwards is provided with a number of contact surfaces, such as bundles of wooden boards, which are kept coated with liquid distributed by a perforated plate above. The washed gas passes under the supplementary partition and upwards through the adjoining passage to the next compartment, so that it always flows over the contact surfaces in the same direction as the liquid. The liquid passes through the casing in a general direction opposite to that of the gas, the bottom of all the compartments being connected by pipes to maintain a predetermined level. The liquid in each compartment is withdrawn by an "air-lift" pump below, and raised to a perforated

spraying pipe at the top, so that the proportion of absorbed gas in the liquid increases as the liquid passes towards the gas inlet end of the casing. The pumps are preferably operated by a portion of the gas which has passed through the apparatus and has been compressed to the necessary pressure.

—W. F. F.

*Fuel for use in explosion engines*. L. Solari, Turin, Italy. Eng. Pat. 109,802, Sep. 3, 1917. (Appl. No. 12,625 of 1917.) Under Int. Conv., Feb. 26, 1916.

The fuel consists of a mixture of alcohol, free or nearly free from water, and petrol. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 2515 of 1914 and 18,468 of 1907; this J., 1915, 948.)

—W. F. F.

*Liquid fuel*. A. A. Backhaus, Baltimore, Md., Assignor to U.S. Industrial Alcohol Co. U.S. Pats. (A) 1,271,114 and (B) 1,271,115, July 2, 1918. Date of appl., Oct. 12, 1917.

A liquid fuel is composed of a petroleum distillate (gasoline, kerosene), an alcohol (ethyl alcohol), and (A) an aromatic nitro-compound (nitrobenzene) or (B) a phenol (cresol).—L. A. C.

*Crude petroleum and natural gas separator*. M. J. Trumble, Assignor to F. M. Townsend and A. J. Gutzler, Los Angeles, Cal. U.S. Pat. 1,269,134, June 11, 1918. Date of appl., Nov. 14, 1914.

Oil and gas are passed through a pipe, provided with a heating jacket and containing a device for reducing velocity, to the top of a vertical cylindrical expansion chamber. The heating fluid passes also through a coil in the bottom of the expansion chamber. The oil is distributed over the surface of the chamber by a spreading cone carried by the lower end of a pipe passing upwards concentrically through the oil inlet aperture and provided with a valve for withdrawing gas at a regulated pressure. The oil collects at the bottom of the chamber and is withdrawn through a pipe, the inlet of which is kept submerged in the oil by a float valve, which controls the liquid level by regulating the discharge.

—W. F. F.

*Oil-shale; Method of and apparatus for treating — [in situ]*. L. H. Rogers, New York. U.S. Pat. 1,269,747, June 18, 1918. Date of appl., Apr. 6, 1918.

PORTIONS of an oil-shale bed are removed, leaving columns of the shale undisturbed. An impervious wall is built around each column, being spaced therefrom and having a metal lining. Means are provided for supplying heat for distillation of the shale in these columns and for the removal of the products.—L. A. C.

*Petroleum product and process of making the same*. R. de M. Taveau, Assignor to The Atlantic Refining Co., Philadelphia, Pa. U.S. Pat. 1,271,387, July 2, 1918. Date of appl., July 6, 1914.

The acid sludge resulting from the treatment of petroleum or petroleum products with concentrated sulphuric acid is agitated with water in the presence of sufficient alkali to neutralise the free sulphuric acid and all or a part of the sulphonic acid content. The water-soluble sulphonates form an emulsifying agent and the resulting emulsion is heated below the emulsion-breaking temperature. The emulsion is then "broken" and allowed to separate into layers containing respectively asphaltic material and sulphonates in solution.—L. A. C.



*Process of making high-percentage calcium carbide.*  
U.S. Pat. 1,271,229. See VII.

*Viscosity-measuring instrument.* U.S. Pat. 1,270,800.  
See XXIII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Destructive distillation; Processes of —.* C. Turner, Manchester. Eng. Pat. 117,645. (Appl. Nos. 8743, June 18, and 17,298, Nov. 23, 1917.)

CARBONACEOUS material, such as peat, lignite, oil shale, oil-impregnated material, or coal, is subjected to destructive distillation in a vessel in which the pressure is raised until it has attained a desired maximum and then reduced rapidly, the operations being repeated until distillation is complete. The retort may be heated externally, but, preferably, superheated steam or heated inactive gas is passed into it. The products pass out of the retort during the reduction of pressure and are condensed. For example, a cylindrical iron container 2½ ft. in diameter and 7 ft. high is connected at the bottom through a stop valve with a boiler generating steam at 20 lb. pressure, and at the top through a similar valve with a condensing plant. The container is charged with air-dried peat and steam blown through to expel the air. The outlet valve is then closed, and steam, superheated to about 500° C., passed in until the pressure is 5 lb. per sq. in.; the inlet valve is then closed and the outlet valve opened until the pressure falls to 2 lb. per sq. in. The operations are repeated until there is no further distillation of products.—L. A. C.

*Wood-gas regulating valve; Automatic — for destructive wood distillation plants.* R. A. and W. Cooper, East Smethport, Pa. U.S. Pat. 1,269,392, June 11, 1918. Date of appl., June 10, 1916. Renewed Jan. 12, 1918.

BRANCH pipes fitted with fluid-controlled dampers connect a main gas pipe, which is connected with a boiler, to each condenser of a battery of retorts and condensers for wood distillation. The main gas pipe is kept under suction.—L. A. C.

*Destructively distilling wood; Method of —.* R. C. Palmer, Pensacola, Fla., Assignor to the Government and people of the United States. U.S. Pat. 1,271,071, July 2, 1918. Date of appl., Mar. 23, 1917. (Dedicated to the public.)

A SUITABLE amount of water is added to wood tar, wood tar pitch, or any of the fractions of crude wood tar, and the mixture distilled under pressure or in the presence of a chemical catalyst, e.g., phosphoric acid. Methyl alcohol, acetic acid, acid phenols, and tar oils are recovered from the condensed vapours. Mixtures of wood with wood tar are distilled at a temperature necessary to carbonise the wood, the vapours confined in the retort under pressure, then released and condensed.

—L. A. C.

*Cathode device; Incandescent —.* G. S. Meikle, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,267,888, May 28, 1918. Date of appl., Aug. 15, 1916.

A SEALED container is provided with a filling of neutral gas, together with a quantity of iodine, and with co-operating electrodes, at least one of which is adapted to operate at incandescence.

—B. N.

## III.—TAR AND TAR PRODUCTS.

*Hydrogenation of aromatic compounds with the help of platinum.* III. Hydrogenation with platinum containing oxygen. Willstätter and Jaquet. See XX.

### PATENTS.

*Benzol and toluol; Purification of commercial — from thiophene and other similar compounds.* P. K. Dutt and J. D. Hamer, Leeds. Eng. Pat. 117,683, July 27, 1917. (Appl. No. 10,786 of 1917.)

COMMERCIAL benzol or toluol is agitated cold with the addition of sufficient chlorine to convert the thiophene or thiotolene into their chloro derivatives, which, having high boiling points, are readily separated by fractional distillation. The requisite quantity of chlorine is determined by analysis, about 1.5% of chlorine being sufficient to remove 0.5% of thiophene or thiotolene.—L. A. C.

*Process of making high-percentage calcium carbide.*  
U.S. Pat. 1,271,229. See VII.

*Coumarone resin and process of making same.*  
Eng. Pat. 117,016. See XIII.

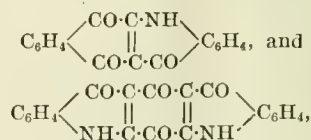
## IV.—COLOURING MATTERS AND DYES.

*Colouring matters; Isolation of — by means of picric and dichloropicric acids.* R. Willstätter and G. Schudel. Ber., 1918, 51, 782—788.

IN his work on anthocyanins, Willstätter has already achieved a certain measure of success in the isolation of the colouring matters by a process which depends on their partition between water and amyl alcohol (compare this J., 1916, 300). By this method, however, only non-glucosidic colouring matters are efficiently extracted, diglucosides often being left entirely in the aqueous layer. A wider range of colouring matters can be extracted as picrates if picric acid is added to the solution, especially if a mixture of amyl alcohol and acetophenone (2 : 1) is used as the medium. Unfortunately the picrates of these colouring matters are usually not very soluble in organic media. Dichloropicric acid is much superior in this respect, and, by the use of this acid, rosaniline, pararosaniline, and Methylene Blue can be completely extracted from aqueous solutions by ether, Safranine by diethylketone, and mono- and diglucosidic colouring matters can be removed completely by one extraction with the above amyl alcohol-acetophenone mixture. The preparation of dichloropicric acid is described. (See further J. Chem. Soc., 1918, i., 399.)—J. C. W.

*Phenonaphthacridonequinone and quinacridonequinone; The linear —.* W. St. Leśniński. Ber., 1918, 51, 695—706.

THESE quinones, represented by the formulæ,



respectively, have been synthesised in order to determine whether quinones of the acridone series have the properties of vat dyes. Phenonaphthacridonequinone sublimes in orange needles, m.pt. 384° C., and forms a blue alkaline-hydrosulphite vat from which cotton is dyed orange-yellow, and

quinacridonequinone, a refractory yellow powder, is also reducible, but the bluish-violet product is insoluble, and therefore not applicable as a dye. (See further J. Chem. Soc., 1918, I., 405.)

—J. C. W.

*Dyestuffs derived from m-phenetidine.* F. Reverdin, A. Rilliet, and C. Vernet. Arch. Sci. phys. nat., 1918, 46, 74—80.

*m*-PHENETIDINE when coupled with diazobenzene chloride yielded benzeneazo-*m*-phenetidine, m.pt. 84°—85° C. This base when further diazotised and coupled with  $\beta$ -naphthol gave the insoluble disazo compound,  $C_6H_5N : N \cdot C_6H_4(OC_2H_5)_2 \cdot N : N \cdot C_{10}H_7 \cdot OH$ . Benzeneazo-*m*-phenetidine was diazotised and coupled with the following compounds giving dyestuffs, the dyeing properties of which are given in brackets:—with R salt (reddish-violet on silk, duller on wool, not fast to washing); with naphthionic acid (reddish-brown on silk, not fast to light); with  $\gamma$ -acid, 2,8,6-aminonaphtholsulphonic acid (violet-brown on wool); with chromotrope acid, 1,8-dihydroxynaphthalene-3,6-disulphonic acid (bluish-violet on ordinary wool, greyish-blue on chrome-mordanted wool); with R-acid, 2,3,6-naphtholdisulphonic acid (brownish-red on ordinary wool, giving a chrome lake); with  $\beta$ -naphthol-7-sulphonic acid (violet-red, moderately sharp, on wool); with  $\alpha$ -naphthol-1-sulphonic acid; with II-acid, 1-amino-8-naphthol-3,6-disulphonic acid (brownish-violet on wool). All of these colours are devoid of special qualities which might give them a technical interest, although some of them, from the point of view of shade and tinctorial properties and leaving cost out of count, could rival some of the colours used industrially. *m*-Sulphanilic acid when diazotised and coupled with *m*-phenetidine yielded *m*-sulphobenzeneazo-*m*-phenetidine, reddish brown needles, m.pt. 214°—215° C., which when further diazotised gave a diazo derivative, only slightly soluble and very stable, which was coupled with *p*-nitrobenzeneazo-8-amino- $\alpha$ -naphthol-3,6-disulphonic acid, giving the trisazo compound,  $SO_2 \cdot H \cdot C_6H_4 \cdot N : N \cdot C_6H_4(OC_2H_5)_2 \cdot N : N \cdot C_{10}H_7 \cdot OH$  ( $NH_2$ ), ( $SO_3$ )<sub>2</sub>  $N : N \cdot C_6H_4 \cdot NO_2$ , a blackish-green powder, dyeing wool bluish-green at 1%, deep bluish-green at 3%, bluish-green almost black at 5%, and black at 8.5%. These tints are moderately fast to potting and fulling, and the shade is scarcely modified by chrome-mordanting. When *p*-nitraniline was diazotised and coupled with *m*-phenetidine it gave *p*-nitrobenzeneazo-*m*-phenetidine, which without being isolated was further diazotised and coupled in alkaline solution with chromotrope acid giving the compound,  $NO_2 \cdot C_6H_4 \cdot N : N \cdot C_6H_4(OC_2H_5)_2 \cdot N : N \cdot C_{10}H_7 \cdot OH$ , a brownish-black powder, dyeing wool a dull violet. If the nitro group of the *p*-nitraniline nucleus in this compound is reduced by sodium sulphide (Meister, Lucius, u. Brünning, 1892, Ger. Pat. 91,283; Friedländer, 4, 725) a brown compound is obtained, which dyes wool blue.—W. G.

*Fisetol: Attempt to synthesise* —. J. Tambor and E. M. Du Bois. Ber., 1918, 51, 748—751.

$\omega$ -BROMO-2-HYDROXY-4-METHOXYACETOPHENONE, from *m*-dimethoxybenzene and bromoacetyl bromide, is heated with sodium acetate and acetic anhydride, when the diacetate of  $\omega$ -2-dihydroxy-4-methoxyacetophenone,  $HIO \cdot C_6H_3(OCH_3)_2 \cdot CO \cdot CH_2 \cdot OH$ , is formed. The latter is the methyl ether of fisetol, but has not yet been demethylated. (See further J. Chem. Soc., 1918, I., 395.)—J. C. W.

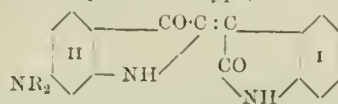
## PATENTS.

*Lignonsulphonic acid or its salts; Method of preparing azo colouring matters from* —. E. Oman, Stockholm, Sweden. Eng. Pat. 103,480, Jan. 4, 1917. (Appl. No. 200 of 1917.) Under Int. Conv., Jan. 20, 1916.

ISOLATED lignonsulphonic acid or the solution of its salts prepared by precipitating the ligninsulphonates from sulphite-cellulose waste liquor by sodium chloride and concentrating the filtrate to one-fifth of its bulk, is coupled with diazotised aromatic amines, e.g., aniline or 4-nitro-2-toluidine, and the azo compound, varying in colour from light red to dark brown, is precipitated by the addition of alcohol or a salt.—J. F. B.

[Indigoid] vat dyes; Yellowish-brown —. W. Bauer and A. Herre, Opladen, Germany, Assignors to Synthetic Patents Co., New York. U.S. Pat. 1,266,092, May 14, 1918. Date of appl., Mar. 23, 1915.

A CLAIM for vat-dyes of the type,



in which the benzene nucleus I does, and nucleus II may, contain halogen, and R may be oxygen or hydrogen. The compound obtained by combining 5,7-dichloroisatin with 6-aminoindoxyl is specially mentioned; it is a dark brown, lustrous, crystalline powder, soluble in concentrated sulphuric acid with reddish-violet colour, which dyes cotton from the hydrosulphite vat in yellowish-brown shades, fast to light, washing, and chlorine.—J. C. W.

*Process for the manufacture of inks for printing on paper.* Eng. Pat. 117,023. See XIII.

[Indelible] writing inks. Eng. Pat. 117,117. See XIII.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Woollen fabric; Determination of acid in* —. A. Woodmansey. J. Soc. Dyers and Col., 1918, 34, 172—175.

The determination of small quantities of acid in wool is complicated by the strong affinity between the fibre and the acid, which makes it extremely difficult to extract the latter by simple washing. A method has been devised based on the principle of distribution of the acid between two immiscible solvents, the wool being one and the aqueous portion the other. The indicator employed is a solution of Methyl Red in dilute alcohol (0.5 gm. per litre). This indicator, when spotted on the cloth, shows a distinct indication only when the cloth has been treated and dried with 0.2% of sulphuric acid, proving that wool itself is essentially alkaline. For the determination of acid in a sample of woollen cloth, standard curves must first be established for a similar cloth which is neutral and has been treated with known quantities of acid. Pieces of the neutral cloth, 10 grms. each, are steeped in 300 c.c. each of solutions made up with increasing quantities of standard sulphuric acid. After standing overnight to attain equilibrium, 250 c.c. is withdrawn and titrated. The curves are plotted with total acid as ordinates and acidity of 250 c.c. of aqueous solution as abscissæ. Similar determinations are carried out with the sample to be tested, three pieces being taken, one with water and two with known quantities of the same acid. By superposing the curve of the sample on the curve of the



standard, the equivalent of acid already present in the former is found by difference. If the "net" acidity be desired, it is necessary to determine the amount of acid required to neutralise the wool and deduct this from the total value found. If the quantity of sample available is only small, the data for the curve may be obtained from one piece with successive additions of acid.—J. F. B.

*Cellulose and its derivatives; The fluorescence of* — S. J. Lewis. J. Soc. Dyers and Col., 1918, 34, 167–172.

It had previously been recorded by Hartley (Chem. Soc. Trans., 1893, 245) that cellulose in the form of white blotting paper is fluorescent and capable of rendering visible the whole of the ultraviolet spectrum as far as wave-length 2000. This phenomenon has been studied by the author, and the effects have been recorded photographically to show the relative intensity of the degradation of ultraviolet light at various wave lengths to visible rays capable of passing through glass and affecting the photographic plate. Graphs have been constructed in which the intensities are plotted as ordinates and the wave-lengths as abscissae. The general results show that the power and distribution of the fluorescent properties are definite functions of the chemical constitution, and their variations conform to what is known of the influence of substituent groups on the properties of the original substance. Normal cellulose, from whatever source it is derived, gives a fairly uniform spectrum, but the intensity varies with the specimen under observation. The cellulose from rhubarb stalk and cuticle falls in the same group. Modified celluloses, such as viscose fabric and parchmentised paper, show a considerable divergence from the normal; well beaten "bank" paper falls in the same class, which is characterised by a strong effect at a wave-length of 2750. Ground wood paper (lignocellulose) is devoid of fluorescent properties, and the cellulose nitrates are nearly, if not quite, inactive. On the other hand, the acetylcelluloses exhibit a fluorescence which is generally much stronger than that of the normal cellulose, and which is much stronger towards the visible region than towards the extreme ultraviolet. For media of the same chemical constitution the resulting degraded spectrum is much the same for the transparent film through which the ultraviolet light is transmitted as for the opaque network in which it is reflected at the surface of the fibres.—J. F. B.

*Artificial silk; The formation of the thread in the spinning of* — H. Ost. Z. angew. Chem., 1918, 31, 141–144.

CUPRAMMONIUM solutions are spun from groups of single glass capillary jets, 0.2–0.16 mm. wide and 1–4 mm. long in the capillary. Viscose is spun from platinum discs pierced with 15–25 holes of 0.10 mm. diam. The spinning pressure is 1.5–2.5 atmos. in both cases. At the Elberfeld works the cuprammonium is prepared by blowing air through aqueous ammonia in presence of metallic copper at a low temperature, the cotton being subsequently introduced. The spinning solution contains 7–8% cellulose, 2.5–3% Cu, and 7–8%  $\text{NH}_3$ , together with some sucrose and other additions; the solution is strained several times through the finest steel wire cloth. Viscosity is determined in a tube, 32 mm. wide, 280 mm. long, with marks indicating a volume of 150 c.c.; the tube terminates in a short orifice, 5 mm. wide. A suitable solution runs out in 5–6 mins., and falls in a continuous thread 2 m. long before breaking up into drops. A coagulating bath containing 33 grms. of caustic soda and 8 grms. of sugar per 100 c.c. is generally used, at 48°–50° C.; sulphuric acid containing 32–50 grms. per 100 c.c.

behaves similarly, but the threads are not so good. In practical spinning finer jets than those mentioned are not satisfactory, owing to clogging, and the fineness of the thread is controlled by "drawing out." Fine threads can be spun from coarse jets and coarse threads from fine jets, depending on the relation between the speed of drawing and the supply of cellulose solution; this latter depends on the pressure of delivery and the internal resistance of the capillary, i.e. its length and fineness. There is a limit to the extent to which the diameter may be reduced by drawing out, owing to the breaking of the thread by the increased frictional resistance. The maximum effect obtained in practice represents an extension of the filament to 5–6 times its original length. The diameter of the threads spun from 0.2 mm. capillaries under practical conditions is about 0.1 mm. wet and 0.03 mm. after drying. Much finer threads are spun by the Thiele-Bemberg system, with diameters down to 10–15  $\mu$ , approximating to that of natural silk. In this system capillaries are not used, the fluid being delivered from orifices of 0.8 mm. into a long column (500 mm.) of a very weak coagulating bath, e.g. a 2% solution of caustic soda for cuprammonium. Owing to the slow action of the coagulating bath the thread remains plastic for a considerable time, and is drawn out to a very fine diameter before it is finally solidified. With the capillary process and concentrated coagulating baths the thread is immediately solidified, and its final diameter is fixed as soon as it comes in contact with the liquid of the bath. Experiments are described showing the nature of the formation of the thread in spinning cuprammonium solutions from glass capillaries. With a capillary 0.2 mm. wide and 3.5 mm. long, a pressure equivalent to 1000 mm. of mercury and a drawing speed of 6.5 m. per min. give a thread of 0.2 mm. diam., i.e. equal to the diameter of the jet; complete coagulation takes place in this case immediately at the orifice of the jet. When the drawing speed is increased to 44 m. per min., which is normal practice for these conditions, the thread is drawn out to a diameter of 0.10 mm., but this reduction takes place some distance up the capillary where the solution is perfectly fluid, and the coagulating liquid enters the mouth of the capillary. With a capillary 6.5 mm. long, the point of reduction is shifted still higher up owing to the reduced supply caused by increased frictional resistance, and if the delivery pressure is not increased the speed of drawing must be decreased to 34 m. per min. to avoid breaking the thread; the diameter of the thread is then 0.12 mm. With a shorter capillary, 1.5 mm. long, under the same pressure, the supply is considerably greater, and the speed of drawing can be increased to 80 m. per min., giving a thread 0.09 mm. in diameter; in this case the capillary is practically filled and the point of reduction is situated at the orifice. A jet 1.0 mm. long, but 0.16 mm. wide, permits a drawing speed of 85 m. per min., but the point of reduction is visibly inside the capillary; the thread has a diameter of 0.10 mm. When a thread breaks, the drawing tension is nil and the thread issuing under pressure has a diameter larger than that of the capillary. A thread of 0.22 mm. diameter can be spun from a capillary of 0.20 mm. if the jet be very short (1.0 mm.) and the drawing speed low (9 m. per min.). With a very short jet, a very high drawing speed (88 m. per min.) may exert a pull right up the capillary, so that the thread is drawn from the wide portion of the jet and occupies only about half the bore of the capillary.—J. F. B.

*Waterproofed canvas; Manufacture of* — E. R. Clark. India Rubber J., 1918, 56, 175–176.

WATERPROOFED canvas for military purposes must retain its waterproof qualities under extremes of

temperature and in spite of rough usage. It is important that it should be resistant to mildew. The samples examined by the author are classified as manufactured by processes involving the use of the following:—(1) aluminium soaps; (2) asphaltum, paraffin, pitch, etc.; (3) cellulose dissolved in cuprammonium solution and other forms of dissolved cellulose; (4) drying oils; (5) two layers of fabric with an intermediate adhesive layer. (1) Basic aluminium acetate and saponified linseed oil yield a specially durable product. The fabrics have a good water-repellent surface, but do not stand rough handling well. (2) Asphaltum is a good material for the purpose. Disadvantages of paraffin are that it tends to become brittle, and to favour the growth of mildew. Rosin and wool grease are unsatisfactory admixtures. A solution of rubber in melted paraffin gives good results. (3) Objections to this process are that it is expensive, and that the fabrics are harsh, and are liable to give off an irritant dust when handled. They strongly resist the growth of mildew. (4) These fabrics are liable to spontaneous combustion, and almost invariably crack as the result of repeated creasing. (5) The middle layer of double fabrics must be possessed of good waterproofing and lasting properties. Poor rubber, for example, is of no value. In examining samples the following points should be taken into consideration:—The sample when held to the light should show no pin-holes, reed-marks, or badly-coated patches. It should not be greasy or very stiff, nor have an objectionable odour. The coating should not rub off nor crack on sharp creasing. The colour should be satisfactory. For testing the waterproof quality, the sample is stretched over the mouth of a bottle and a spray of water (*e.g.*, from a Gooch crucible) allowed to fall on it from a height. The time which elapses before the first drop passes through, and the amount passing through in a given time are noted; or, the material is folded into a pocket which is then filled with heavy articles and immersed in water. A good fabric should not wet through in 24 hours. Samples should not develop mildew growths in 5 days when suspended in a desiccator containing a variety of such growths. The effect of temperatures of 0° F. (–18° C.) and 120° F. (49° C.) for eight hours upon the waterproof quality of samples should be determined. A chemical examination should also be made.—E. W. L.

#### PATENTS.

*Drying apparatus [for fibrous materials].* A. Hirth, Stuttgart-Cannstadt, Germany. U.S. Pat. 1,266,946, May 21, 1918. Date of appl., June 24, 1915.

IN an apparatus for suspending and drying strands of material, a number of arms are provided with means for feeding strands to the arms, means for presenting the feeder to alternate sides of each arm and displacing it lengthwise of the arms.

—J. F. B.

*Coated fabrics; Apparatus for treating* —. W. P. Egan and J. J. Sheehan, Lynn, Mass. U.S. Pat. 1,268,941, June 11, 1918. Date of appl., May 7, 1917.

A STEAM chamber is provided with a perforated wall over which a coated fabric moves while moist steam passes through it. The steam is supplied by a number of perforated pipes extending the length of the chamber, and heat is also radiated into the chamber from a steam pipe inclined upwards into the chamber, but substantially closed at its inner end.—W. F. F.

*Silk, silk waste, and the like; Process of degumming* —. K. Tanaka, Nakashima Gun, Japan. U.S. Pat. 1,268,513, June 4, 1918. Date of appl., Dec. 8, 1916.

SILK is treated with a degumming fluid, *e.g.* a solution of soap and sodium bicarbonate, the liquid being heated by steam under a pressure of 15–20 lb.

—J. F. B.

*Wood pulp; Manufacture of* —. O. Kress and S. D. Wells, Madison, Wis. U.S. Pat. 1,266,957, May 21, 1918. Date of appl., Dec. 10, 1915. (Dedicated to the public.)

WOOD is impregnated with an alkaline digestion liquor at about 185° F. (85° C.) under pressure; the pressure is reduced before digestion takes place, and the excess of the alkaline liquor is removed. This is cooled and the rosin soap is skimmed off. The wood impregnated with liquor is digested with steam, and the foaming rosin soap, distilled turpentine and other volatile oils produced in the digestion of the wood are collected.—J. F. B.

*Paper pulp; Manufacture of* —. S. D. Wells, Madison, Wis. Dedicated to the Government and people of the U.S.A. U.S. Pat. 1,268,193, June 4, 1918. Date of appl., April 2, 1918. (Dedicated to the public.)

WOOD or other fibrous material for the production of paper pulp is digested by a liquor in which the concentration of active chemicals during the digestion is reduced to the desired degree by the introduction of spent lye while the digestion is proceeding, whereby the cellulose is protected to a marked degree from the action of the digestion liquors, while their action on the incrusting matters is not diminished.—J. F. B.

*Paper-making machine.* J. Galipeau, Assignor to J. H. Garth, Montreal, Canada. U.S. Pat. 1,266,673, May 21, 1918. Date of appl., May 12, 1917.

THE travelling screen of the paper machine is provided with a suction-box which acts upon the screen and has its suction connection at one end, and means, combined with the suction connection, whereby the box is reciprocated across the direction of travel of the screen.—J. F. B.

*Paper; Waterproof composition for coating — and process of making same.* B. Kniffler, Winchester, Mass., Assignor to Vera Chemical Co., Stoneham, Mass. U.S. Pats. 1,266,955 and 1,266,956, May 21, 1918. Date of appl., Nov. 22, 1913.

THE coating emulsion consists of a mixture of paraffin wax, ammonium stearate, and sufficient water to form a viscous mass with the quantity of ammonium soap employed. It is prepared by adding ammonia to a fatty acid to form an ammonium soap in presence of paraffin wax, heating the mixture and adding sufficient water to form a viscous emulsion, which is subsequently diluted with water.—J. F. B.

*Sulphite-cellulose manufacture; Process of dissolving waste lye from* —. I. Söraas, Hardanger, Norway. U.S. Pat. 1,268,774, June 4, 1918. Date of appl., Mar. 27, 1918.

SULPHITE-CELLULOSE waste lye is heated to a temperature not materially below 80° C. while confined in an atmosphere of a non-oxidising gas; it is then heated in a second operation to about 100° C. in the presence of an oxidising gas under pressure



until the lignin is precipitated. This second heating is performed while introducing compressed air into the liquid, whereby the oxygen in the air is considerably reduced, and the exhaust gas of the second operation is conveyed to the containers in which the first heating is carried out, serving as the non-oxidising atmosphere.—J. F. B.

*Beating machines for making pulp for paper manufacture.* S. Heys, Birmingham. Eng. Pat. 117,880, Dec. 22, 1917. (Appl. No. 18,997 of 1917.)

*Method of preparing azo colouring matters from lignonsulphonic acid or its salts.* Eng. Pat. 103,480. See IV.

*Case-hardening material.* U.S. Pat. 1,270,874. See X.

*Process for the manufacture of inks for printing on paper.* Eng. Pat. 117,023. See XIII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENTS.

*Dyeing; Composition for use in* — J. W. Fries, Winston-Salem, N.C. U.S. Pat. 1,266,577, May 21, 1918. Date of appl., June 16, 1915.

A dry powdered composition for use in dyeing cotton goods is composed of a mixture of a vegetable dye extract, such as logwood, and an ammoniacal hydrated metallic oxide, such as copper oxide. A mixture of a non-alkaline metallic salt, e.g., a copper salt, and an alkaline salt is treated with sufficient ammonia solution to prevent a precipitate being formed, and this liquid is mixed with the vegetable dye extract to form a thick paste, which is finally dried.—J. F. B.

*Dyeing wool and other fibrous substances; Apparatus for* — F. Geissler, Cottbus, Germany. U.S. Pat. 1,267,209, May 21, 1918. Date of appl., Aug. 14, 1916.

The apparatus consists of a box adapted to the shape of yarn dyeing machines, both the lid and one side of the box being removable and guided in grooves in the box, and the lid, when in position, overlapping the upper edge of the removable side.

—J. F. B.

*Skein-dyeing machine.* H. Dudley, Philadelphia, Pa. U.S. Pat. 1,268,441, June 4, 1918. Date of appl., June 28, 1917.

The apparatus consists of a dyeing chamber, a skein-carrying frame within the dyeing chamber, foraminous plates below and above the skein-carrying frame, a series of tubular members abutting on the outer face of each of the foraminous plates, whereby liquid may be passed in either direction through the foraminous plates and the dyeing chamber. A second dyeing chamber is provided adjacent to the first, also containing a fibre-carrying frame, and the top and bottom portions of the second chamber are connected respectively with the top and bottom portions of the first.—J. F. B.

*Dyeing process.* F. J. Oakes and S. O. Dillon, New York. U.S. Pat. 1,271,067, July 2, 1918. Date of appl., July 2, 1915.

For dyeing vegetable fibres (e.g., cotton) a lake is produced by adding the colouring matter to a bath

containing a salt of a heavy metal (copper sulphate) and an alkali (sodium carbonate). The lake is then dissolved in a hot acid solution (acetic acid) in the presence of a neutral salt (sodium sulphate), and the material to be dyed immersed in the solution.—L. A. C.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Potash salts in Hungary.* F. von Konek-Norwall. Chem.-Zeit., 1918, 42, 365—367.

LARGE quantities of alunite occur in Northern Hungary. The potassium sulphate may be recovered by roasting at a red heat until all the aluminium sulphate has been decomposed and sulphuric acid fumes are no longer evolved. The residual mass consists of potassium sulphate (14 to 15%), alumina, and frequently a considerable quantity of silica; it forms a white powder and may be used directly on the land, but when it has to be transported for a distance it is more economical to extract it with water and separate the potassium sulphate by crystallisation from the solution. The mixture of alumina and silica may be used in the manufacture of porcelain, or the alumina converted into alum for use in the textile trades, the sulphuric acid for making the alum being obtained by condensing the vapours which are given off when the original mineral is roasted.—W. P. S.

*Alumina; Production of* — in the aluminium industry, with special reference to the rotary kiln. W. von Escher. Chem.-Zeit., 1918, 42, 361—362. (See also this J., 1918, 504 A.)

A type of rotary kiln specially adapted to drying and dehydrating aluminium hydroxide containing 45—50% total H<sub>2</sub>O is described. Owing to the high temperature required, 1500°—1550° C., the kiln differs considerably from the type used in the cement industry. It consists essentially of five parts, a feeding device, the kiln proper, a cooler for the hot alumina, an automatic device for weighing and delivering the product into sacks, and a dust-collecting equipment for recovering dust from the escaping gases. The kiln is fired with producer or natural gas, the air being preheated and supplied under pressure. The losses are very small, under 1%. The dehydrated alumina should have a loss on ignition of at most 0.15%.—E. H. R.

*Hydroxides; Solubility of some metallic* — in water. G. Almkvist. Z. anorg. Chem., 1918, 103, 240—242.

The following solubilities have been determined, using ordinary distilled water at 20° C.:—Ni(OH)<sub>2</sub>, 12.7 mgrms. per litre; Co(OH)<sub>2</sub>, 3.18; BiO(OH), 1.44; Fe(OH)<sub>3</sub>, 0.151; MnO(OH)<sub>2</sub>, 0.43 mgrms. per litre. (See also J. Chem. Soc., 1918, ii., 320.)

—E. H. R.

*Vanadic acid; Application of rapidly rotating metallic reductors in the determination of* — F. A. Gooch and W. Scott. Amer. J. Sci., 1918, 48, 427—436.

The reduction of vanadic acid may be effected rapidly in the presence of sulphuric acid by means of rapidly rotating electrodes of copper or silver, or by a rotating cylinder of zinc without the use of the electric current. The following procedure is recommended, as the volume and temperature of the solution and the relative size and speed of rotation of the zinc cylinder affect the rapidity of the reduction:—Volume of

solution at outset, heated to boiling, 80 c.c.; 5 c.c. of concentrated sulphuric acid; diam. of cylinder, 2 cm.; surface of cylinder exposed to action, 25 sq. cm.; speed of rotation, 850 r.p.m. At the end of the reduction, the solution is diluted to 250 c.c. and 10 c.c. of a saturated solution of silver sulphate added to ensure all the vanadium being in the form of tetroxide; the solution is then boiled to coagulate the precipitated silver, filtered, again heated to boiling, and titrated with standard potassium permanganate. A correction, as determined in a blank test, must be applied. (See also J. Chem. Soc., Oct., 1918.)—A. B. S.

*Temperature viscosity relations in the ternary system lime-alumina-silica.* Field and Royster. See X.

*Baghouses for zinc oxide.* Cregan. See X.

*Determination of turpentine.* Tausz. See XIII.

*Nitrate and nitrite assimilation [by plants]. Iron and oxygen as necessary agents for the reduction of alkali nitrites by auto-oxidisable compounds.* Baudisch. See XVI.

*Influence of foreign substances on the activity of catalysts. IV. Experiments with palladium hydrosol in presence of mercury and mercuric oxide.* Paul and Hartmann. See XX.

*Volumetric determination of barium and solubility of barium chromate in various aqueous solutions.* Waddell. See XXIII.

*Behaviour and detection of the smallest quantities of carbon monoxide in the "chlorate" pipette.* Hofmann and Schibsted. See XXIII.

*Melting points of cristobalite and tridymite.* Ferguson and Merwin. See VIII.

#### PATENTS.

*Sodium carbonate and ammonium sulphate; Manufacture of — from bisulphate of soda.* Soc. Industrielle de Produits Chimiques, Paris. Eng. Pat. 109,814, Sep. 19, 1917. (Appl. No. 13,487 of 1917.) Under Int. Conv., Sep. 20, 1916.

NITRE cake is treated with water and sufficient ammonia to neutralise the free acid and an excess according to the quantity of sodium sulphate present. The mixture is heated and treated with carbon dioxide under slight pressure with formation of ammonium bicarbonate, which by double decomposition with a portion of the sodium sulphate forms ammonium sulphate and sodium bicarbonate, the latter precipitating out. The mother liquors after neutralisation with nitre cake are fractionally crystallised to separate sodium and ammonium sulphates and the sodium sulphate is used in a fresh charge.—C. A. K.

*Reduction; Process of [producing barium sulphide by] electrical —.* C. H. Fulton, St. Louis, Mo., Assignor to Metallurgical Laboratories, Inc., Chicago, Ill. U.S. Pat. 1,267,347, May 21, 1918. Date of appl., July 25, 1917.

Barium sulphide is produced by mixing approximately 100 parts of finely-divided barium sulphate (barytes) with 70 to 85 parts of finely crushed coke and 20 parts of a carbonaceous binding material, the coke acting as a conductor of electricity as well as a reducing agent. The mixture is formed into

briquettes, retaining approximately their original form after being subjected to a temperature high enough to bring about the desired reduction. A current of electricity is passed through one or more of the briquettes, whilst they are confined in an electric reduction furnace and in engagement with the electrodes of the furnace. The briquettes are subsequently treated with water to dissolve out the barium sulphide.—B. N.

*Alumina; Producing crystalline —.* R. H. White, Niagara Falls, N.Y., Assignor to Norton Co., Worcester, Mass. U.S. Pat. 1,269,141, June 11, 1918. Date of appl., Aug. 18, 1917.

The method consists of electrically fusing in a carbon hearth furnace a non-uniform charge of which the lower portion contains an oxidising addition yielding volatile reduction products, whereby reduction of alumina by the carbon hearth is prevented.—J. H. P.

*Aluminous materials; Purifying —.* L. E. Saunders, Niagara Falls, N.Y., Assignor to Norton Co., Worcester, Mass. U.S. Pats. (A) 1,269,223 and (B) 1,269,224, June 11, 1918. Date of appl., Aug. 18, 1917.

(A) The aluminous material is mixed with sufficient carbon to effect a substantial reduction of the alumina, and is then fused. The reduced impurities are separated and the residue is mixed with an oxidising agent such as bauxite and again fused in an electric furnace. (B) The crude aluminous material is fused with a proportion of carbon sufficient theoretically for the complete reduction of the non-aluminous oxides and with sufficient metallic iron to prevent the reduction of the alumina.

—A. B. S.

*[Aluminium chloride;] Process of purification [of —].* V. M. Weaver, Harrisburg, Pa., Assignor to Weaver Co. U.S. Pat. 1,269,236, June 11, 1918. Date of appl., Aug. 9, 1915.

ALUMINIUM chloride is purified by passing it through a bath of molten aluminium and collecting the purified aluminium chloride vapour which is evolved. The process may be applied to other compounds, the molten bath used being composed of the metal forming the base of the compound to be purified.—J. H. P.

*Sodium hydrosulphite; Method of manufacturing —.* A. K. Gyzander, Westbrook, Me. U.S. Pat. 1,270,150, June 18, 1918. Date of appl., Apr. 14, 1917.

SALTS of sulphurous acid are converted into hydrosulphites by the action of hydrogen gas under pressure.—J. H. P.

*Sodium aluminate; Process of making —.* W. L. Melick, Columbus, Ohio. U.S. Pat. 1,271,192, July 2, 1918. Date of appl., May 7, 1917.

A SATURATED aqueous solution of sodium hydroxide is heated to boiling with bauxite ground to the fineness of at least 70-mesh. The mixture is in the form of a thick paste, the consistency of which is maintained during the boiling, by the addition of water. The sodium aluminate formed is subsequently separated.—J. H. P.

*Calcium carbide; Process of making high percentage —.* D. R. Russell, Chicago, Ill. U.S. Pat. 1,271,220, July 2, 1918. Date of appl., Mar. 25, 1918.

ELECTRODES are inserted in a conglomerate of lime and carbon which is then smelted to high percentage



calcium carbide. The carbon used is pitch coke made from the pitch of completely distilled by-product coke-oven coal tar.—J. H. P.

*Cyanides and ammonia; Process of manufacture of* —. L. P. Devaucelle, Courbevoile, France, and F. Bensa, Genoa, Italy. Eng. Pat. 12,946, Sep. 9, 1915. Under Int. Conv., Sep. 9, 1914.

SEE Fr. Pat. 478,919 of 1914; this J., 1916, 1109. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 433 of 1882.)

*Alkaline earth fluorides and soluble potassium salts; Process for obtaining* —. F. B. Dehn, London. From W. G. Henshaw, San Francisco, U.S.A. Eng. Pat. 117,901, July 24, 1917. (Appl. No. 9672 of 1918.)

SEE U.S. Pat. 1,194,344 of 1916; this J., 1916, 966. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 21,673 of 1897, and in pursuance of Sect. 8, Sub-sect. 2, to Eng. Pat. 110,540.)

*Alumina; Purification of* —. Mineral Products Corporation, New York, Assignees of J. W. Hornsey, Summit, N.J., U.S.A. Eng. Pat. 118,063, Feb. 27, 1918. (Appl. No. 3528 of 1918.) Under Int. Conv., Aug. 11, 1917.

SEE U.S. Pat. 1,255,749 of 1918; this J., 1918, 206 A.

*Potassium compounds; Manufacture of* — from glauconite and like minerals. F. Tschirner, Newark, N.J., U.S.A. Eng. Pat. 117,870, Nov. 2, 1917. (Appl. No. 16,013 of 1917.)

SEE U.S. Pats. 1,254,450 and 1,254,451 of 1918; this J., 1918, 181 A.

*Carbonic acid; Apparatus for cleaning* —. L. Nathan, Zürich, Switzerland. U.S. Pat. 1,268,872, June 11, 1918. Date of appl., Jan. 11, 1916.

SEE Eng. Pat. 12,165 of 1914; this J., 1915, 1026.

*Silicic acid or hydrated silica; Process for making* —. W. C. Arsem and J. G. E. Wright, Schenectady, N.Y., Assignors to General Electric Co. U.S. Pat. 1,270,093, June 18, 1918. Date of appl., Aug. 23, 1917.

SEE Eng. Pat. 113,769 of 1917; this J., 1918, 241 A.

*Apparatus for rectifying and concentrating liquids.* U.S. Pat. 1,271,349. See I.

*Method of recovering graphite from slag.* U.S. Pat. 1,271,146. See X.

*Apparatus for the electrolysis of water.* Eng. Pat. 117,533. See XI.

## VIII.—GLASS; CERAMICS.

*Glass; Notes on annealing* —. S. English and W. E. S. Turner. J. Soc. Glass Technol., 1918, 2, 90—102.

BADLY annealed or strained glass readily breaks or shatters when exposed to shock or sudden change of temperature. To anneal a glass properly, it must be heated to a temperature at which all strain rapidly disappears without the glass undergoing deformation, and the subsequent cooling must be as rapid as possible without any recurrence of

strain. Specimens of strained glass were heated until free from strain when examined between crossed nicols, the temperature and time of heating being noted. If the temperature is sufficiently high the disappearance of the strain is very rapid. The authors conclude that the time-temperature curve representing ideal conditions of annealing is a double one, indicating that a glass may be cooled rapidly to just below the temperature at which strain disappears, but between this critical point and that at which the glass becomes rigid the cooling must be very slow. When the glass is rigid it may again be cooled rapidly. Thus, a glass with an upper critical temperature of 585°—590° C. may be cooled at the rate of 25° per hr. from 590° to 500° C., but even when the rate of cooling is only 9° per hour between 509° and 400° C. a slight strain is produced. The temperature at which the more rapid cooling of the rigid glass may be commenced varies with different glasses; it may be found by cooling glasses rapidly from different temperatures and then examining them to see which are strained, or by watching the movement of the rings when the glass is cooled between crossed nicols. In the latter case, the highest temperature which will not cause the rings to move in 12 hrs. may be taken as the temperature below which the cooling may be accelerated. In manufacturing practice, a lehr with three distinct cooling stages is recommended. Electric lamp bulbs and wine glasses made of lead glass may be annealed by placing them in a hot box lined with asbestos and allowing them to cool freely. Alkali-lime glasses may be cooled from the annealing temperature (about 550° C.) to 320° C. in 3½ hours, and below that temperature in a further 3 hrs. Boric oxide heat-resisting lamp glasses are rather difficult to anneal, very careful cooling between 500° and 360° C. at a rate not exceeding 9° C. per hour being necessary; below 360° C. a more rapid cooling is satisfactory. Glasses used for chemical work are the most difficult to anneal; one examined by the authors cannot be cooled between 500° C. and 400° C. without showing some signs of stress, whilst others may be annealed rapidly.—A. B. S.

*Cristobalite and tridymite; Melting points of* — [and new high-temperature electric furnace]. J. B. Ferguson and H. E. Merwin. Amer. J. Sci., 1918, 46, 417—426.

CLEAR quartz crystals were heated for 144 hrs. at temperatures ranging from 1300° to 1400° C., when the greater part of the material was inverted to cristobalite with a few clear nuclei of tridymite. These were separated and the melting points determined. The cristobalite melted at  $1710^{\circ} \pm 10^{\circ}$  C. The tridymite grains inverted into cristobalite at  $1667^{\circ}$ — $1677^{\circ}$  C. without melting. Natural tridymites, from Nevada and Mexico respectively, melted sharply at  $1670^{\circ} \pm 10^{\circ}$  C. These results confirm the earlier observations of Fenner (this J., 1913, 22), but are in opposition to those of Le Chatelier (this J., 1917, 964) and show that the region of stability of cristobalite is above that of tridymite. The electric furnace used was constructed on the cascade principle, the inner coil being made of an alloy of platinum with 20% of rhodium and the outer coil of pure platinum. The inner coil is wound on a helically grooved magnesia tube and the two coils are insulated from one another by well burned magnesia powder. With this furnace the charge can be maintained above 1700° C. for periods of several hours in an oxidising atmosphere. (See also J. Chem. Soc., Oct., 1918.)—A. B. S.

*Temperature-viscosity relations in the ternary system lime-alumina-silica.* Feild and Royster. See X.

## PATENTS.

*Refractory material and process of making same.* J. O. Handy and R. M. Isham, Assignors to Allen S. Davidson Co., Pittsburgh, Pa. U.S. Pats. (A) 1,270,818 and (a) 1,270,819, July 2, 1918. Dates of appl., Jan. 22 and 30, 1917.

(A) A CARBONATED refractory material, such as magnesium carbonate, is mixed with a liquid containing a small amount of a soluble chloride, dried, and calcined at a temperature high enough to expel most of the chlorine and to produce a refractory material free from carbon dioxide and resistant to air, steam, and hot water. (B) Dolomite is calcined, extracted with cold water until the lime present in the residue is reduced to 3–10%, and then re-heated with a small amount of a blinding material.

—A. B. S.

*Lining for furnaces.* O. J. T. G. R. Martin, Vancouver. U.S. Pat. 1,271,052, July 2, 1918. Date of appl., Sep. 21, 1917.

A MIXTURE composed of 15 parts of brick clay, 8 parts of infusorial earth, 4 parts of soapstone, and 6 parts of asbestos, rendered plastic by water containing  $\frac{1}{4}$  oz. of salt per gallon.—F. C. Th.

*Aluminous abrasives; Preparing* —. L. E. Saunders and R. H. White, Niagara Falls, N.Y., Assignors to Norton Co., Worcester, Mass. U.S. Pat. 1,269,222, June 11, 1918. Date of appl., Aug. 18, 1917.

A SMALL amount of an oxygen compound of zinc is added to the charge of material used for making aluminous abrasives in order to prevent the reduction of the alumina by the carbon in the charge.

—A. B. S.

*Producing crystalline alumina.* U.S. Pat. 1,269,141. See VII.

*Purifying aluminous materials.* U.S. Pats. 1,269,223 and 1,269,224. See VII.

## IX.—BUILDING MATERIALS.

## PATENTS.

*Fibrous materials [wood]; Process of treating [waterproofing]* —. G. E. Ferguson, Assignor to W. E. Silverthorne, New York. U.S. Pat. 1,268,446, June 4, 1918. Date of appl., Apr. 9, 1913. Renewed Oct. 27, 1917.

THE fibrous material to be rendered waterproof is immersed in a molten waterproofing substance and the air removed by suction from the pores. The temperature is then raised so as partly to char the fibres, and pressure is applied to force the liquid into the interstices of the material. The material is then withdrawn and allowed to cool.—F. C. Th.

*Fibrous [roofing] material and method of making the same [from scrap leather].* A. L. Clapp, Braintree, Mass., Assignor to R. W. Bird, Framingham, Mass. U.S. Pat. 1,269,905, June 18, 1918. Date of appl., July 29, 1915.

FELT for roofing is made by disintegrating scrap leather into a fine, fibrous, pulpy mass, adding a softening agent, and then forming the mass into soft, porous sheets.—A. B. S.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Basic steel process; Influence of badly burnt lime in the —.* M. Backheuer. Stahl u. Eisen, 1918, 38, 748–750.

LIME for slag making in the basic process should give a loss on ignition of not more than 2–4%, or at the highest 6%. Dead-burnt lime, however, is dissolved with difficulty in the slag. Badly burnt lime exercises a harmful influence both on the quality of the steel, resulting in non-metallic inclusions, and also on the working of the heats as a result of the chilling effect. In the basic Bessemer process poor lime results in an excessive loss of manganese, the steel being highly oxidised. The blow is abnormal and the elimination of phosphorus in the after-blow uncertain. With badly burnt lime 1–3% manganese is the minimum necessary in the mixer-iron used. Excessive loss during the blow and considerable production of scrap also result from the addition of incompletely burnt lime.

—F. C. Th.

*Cyanide solutions; Estimation of oxygen in working* —. H. A. White. J. Chem., Met., and Min. Soc., S. Afr., 1918, 18, 292–297.

A COLORIMETRIC method is based on the oxidation of alkaline photographic developers (e.g., pyrogallol, amidol) by dissolved oxygen, with the formation of quinones. A series of standard tints is made by saturating water at a known temperature with air, developing the colour by the addition of 0.1 gm. of pyrogallol and 1 c.c. of 2N sodium hydroxide, and suitably diluting. The sample of liquor is then treated similarly, care being taken to exclude air in all cases, and matched with one of the standard solutions. A more permanent series of tints can be produced by matching suitable solutions of dyes against the standards prepared from water of known oxygen content.—C. A. K.

[Copper.] *Experiments in fettling reverberatory furnaces.* F. Rutherford. Eng. and Min. J., 1918, 106, 109–110.

SINCE 1912, reverberatory copper-smelting furnaces have been provided with holes along the sides of the arch through which the charge is fed instead of being dropped in the centre of the bed. This method of fettling protects the side walls. The former practice of fettling with siliceous ore and feeding of hot calcines at the centre has given place to feeding hot calcines at the sides of the furnace. In the former method a siliceous scum or blanket is formed which covers the slag and causes loss of metal. If no calcines are available, the ore used for side-feeding should be as nearly self-fluxing as possible and contain enough sulphur to combine with the copper.—W. R. S.

*Zinc oxide; Baghouses for* —. J. F. Cregan. Eng. and Min. J., 1918, 106, 126–132.

Two types of baghouse, the Eastern and the Western, have gradually been evolved. The principal difference consists in the disposition of the bags: in the Eastern type, they are suspended from the distributing pipes situated just under the roof of the baghouse; the bags have a length of 44 ft. In the Western type the bags, which average 30 ft. in length, are fastened at their upper ends by means of ropes to pulleys suspended from the roof; the distributing pipes are laid along the floor of the baghouse. In the Eastern States the ores smelted are clean silicates free from lead and sulphur; the fuel is anthracite. In the West there is no constant ore or fuel supply. Silicates, carbonates, and



sulphides are smelted; practically all the ores contain lead, while the fuel used is coal high in sulphur and volatile matter. Western zinc oxide is sold according to chemical analysis as well as according to colour test.—W. R. S.

*Zinc spraying process; An electrical* — W. Kasperowicz. *Z. angew. Chem.*, 1918, 31, 144.

AN electrically heated apparatus for spraying molten zinc according to Schoop's "metallising" process has been introduced. The zinc is contained in the "pistol" in the form of two wires connected with any existing electric system and the points of these two zinc wires are caused to touch so that they short-circuit the current and form a small arc. The molten metal is immediately sprayed out by a jet of compressed air and the wires are fed forward by a mechanical arrangement, so that the points are always in the correct position to give a quietly burning arc for uninterrupted work. The best results are obtained with continuous current. The electrical process is more economical than the gas heating process, because the heat is generated only at the working points; moreover the apparatus melts and sprays twice as much metal in the same time as compared with the gas-heated "pistol." (See also this J., 1918, 472 A.)—J. F. B.

[*Lead.*] *Mechanical Scotch-hearth smelting.* W. E. Newnam. *Eng. and Min. J.*, 1918, 106, 101–104.

MECHANICALLY operated Scotch hearths are now in general use in the Middle Western States, as well as in Canada and Spain, at places where the supply of galena is insufficient for blast-furnace work. Some plants have a daily capacity of 100 tons of concentrate. Two types of installation are in vogue. In the first, the hearths are placed in a straight line facing in the same direction; the flue system is placed outside the building. In the second, the hearths are placed in two lines, facing in opposite directions, with the flue system between the lines. This arrangement is employed where the space is limited, or where a large number of hearths are installed. The quantity of grey slag formed averages 2% of the ore. Drawings are given for a 100-ton plant comprising five mechanically operated hearths.—W. R. S.

*Coal; Use of pulverised* — [in lead smelting]. C. T. Rice. *Eng. and Min. J.*, 1918, 106, 91–94.

THE furnaces of the refining plant at the Bunker Hill lead smelter (Kellogg, Idaho) are fired with pulverised coal, which is far cheaper than oil. The Holbeck return-circuit system is used, in which the coal is crushed in a closed circuit and under partial vacuum. The powdered coal is also circulated by a current of air in a closed circuit throughout the plant, the excess returning to the bins of the system after the desired amount of coal has been drawn off by means of deflectors to the burners. 85% of the coal dust is smaller than 220-mesh. The pulveriser crushes 20 tons (24 hours' supply) of slack in 8 hours, two men being required.—W. R. S.

*Lead; Determination of* — in ores, etc. W. Stahl. *Chem.-Zeit.*, 1918, 42, 317.

A WEIGHED quantity of the finely-divided sample is mixed with about 500 c.c. of water, acidified with nitric acid, and heated at 40° C. for 2 hrs. The mixture is then diluted with water, treated with hydrogen sulphide, filtered, and the insoluble portion washed with warm water containing a small quantity of potassium sulphide. The insoluble portion is then heated with nitric acid until all

sulphur has been oxidised, the solution evaporated with the addition of sulphuric acid, and the lead sulphate, etc., collected and washed with dilute sulphuric acid. The precipitate is then extracted with warm ammoniacal ammonium tartrate solution, filtered, and the insoluble portion washed with warm water. The filtrate now contains the lead sulphate together with antimony; the antimony remains in solution when the filtrate is acidified with sulphuric acid and mixed with alcohol, whilst the lead sulphate is re-precipitated; and is collected, dried, and weighed.—W. P. S.

*Antimony; Pure* — E. Groschuff. *Z. anorg. Chem.*, 1918, 103, 164–188.

TECHNICALLY refined antimony is purer than electrolytic antimony prepared by the electrolysis of antimony sulphide solution. The latter, by further refining, gives a highly pure product containing less than 0.02 impurity. To obtain chemically pure antimony, the tri- or pentachloride is distilled and converted into chloroantimonic acid,  $\text{SbCl}_4\text{H}_4\text{O}$ , which can be freed from all impurities by recrystallisation. The purified product is hydrolysed to antimonic acid, which is reduced by fusion with potassium cyanide. Antimony so prepared contains no recognisable impurities. Its melting point, which is an important fixed point in thermometry, is 630.3° C. A method has been devised for detecting and estimating the impurities in nominally pure antimony. The principle of the method consists in converting the antimony into chloroantimonic acid, which is then fractionally crystallised. The impurities are concentrated in the mother liquors, which are subsequently analysed by the usual methods. (See also *J. Chem. Soc.*, 1918, ii., 322.)—E. H. R.

*Mercury; Extraction of* — from its ores by sodium sulphide. C. H. Holland. *New Zealand J. Sci. Tech.*, 1918, 1, 153–154.

ONE part of an ore containing cinnabar, ferrie oxide, and a hydrocarbon is crushed, and agitated with 4.5 parts of an aqueous solution containing 4% sodium sulphide and 1% sodium hydroxide. The solution is filtered and mercury is precipitated from the double sulphide of mercury and sodium by aluminium shavings. The mercury is obtained partly in metallic form, and partly as a powder mixed with foreign matter, which is heated in a retort with manganese dioxide and lime. The total yield of mercury is 84.5%.—W. F. F.

*Lime-alumina-silica; Temperature-viscosity relations in the ternary system* — A. I. Feild and P. H. Royster. *U.S. Bureau of Mines. Tech. Paper* 189. 1918. 33 pages.

THE investigation was confined to that range of composition encountered in iron-blast-furnace slags. Synthetic melts were made and their viscosities measured at temperatures ranging from the melting point to an upper limit of 1600° C., an electric furnace being used and the temperatures measured by a Holborn-Kurlbaum optical pyrometer. The viscosity was estimated by rotating the graphite crucible containing the molten material and determining the torque on a steel ribbon used to suspend a graphite rod in the axis of the crucible. In the binary system calcium metasilicate-gehlenite ( $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ), a maximum viscosity for temperatures between 1350° and 1600° C. is shown by a mixture of about 30% gehlenite and 70% calcium metasilicate, and a minimum viscosity by the eutectic mixture. At the saturation temperature and at 50° and 100° C. superheat respectively, the binary eutectic exhibits maximum viscosity when

compared with contiguous mixtures. Viscosity-temperature measurements were made of mixtures representing seven quintuple points of the ternary system. Two of these mixtures ( $\text{CaO}$  47.2,  $\text{Al}_2\text{O}_3$  11.8,  $\text{SiO}_2$  41%, and  $\text{CaO}$  49.5,  $\text{Al}_2\text{O}_3$  43.7,  $\text{SiO}_2$  6.8% respectively) do not obey the usual rule of possessing a viscosity-temperature curve continuous at the melting point, so that crystallisation of these mixtures should proceed without great difficulty. Viscosity measurements were made of compositions lying on the boundary curves between the fields of calcium metasilicate and anorthite, calcium metasilicate and gehlenite, gehlenite and anorthite, and calcium orthosilicate and gehlenite at temperatures of  $1400^\circ$ ,  $1500^\circ$ , and  $1600^\circ$  C. Each of the viscosity-composition curves terminates at a quintuple point representing a eutectic and includes between these two ternary eutectics the binary eutectic between those two compounds whose stability fields it limits. In the boundary curve between calcium orthosilicate and gehlenite there are also two quintuple points which are not eutectics, *viz.*, the points where gehlenite,  $\alpha$ - and  $\beta$ -calcium orthosilicate are in equilibrium with their melt. The viscosities of the mixtures corresponding to these two points are much higher than for any composition lying in this part of the system; this appears to be due to the existence of the molecular aggregate identified with  $\beta$ -calcium orthosilicate. In each of these boundary curves the viscosity increases from a minimum at a binary eutectic to a maximum at a quintuple point. Some idea of the general nature of the isothermal viscosity surfaces can be gained from the profiles of these surfaces at  $1450^\circ$  C. shown for mixtures containing 7, 10, 15, 18, and 21%  $\text{Al}_2\text{O}_3$  respectively. In Fig. 1 for 7%  $\text{Al}_2\text{O}_3$  the boundary curve between the fields of calcium metasilicate and akermannite occurs at  $a$ ;  $f$  lies on the boundary curve of the binary system calcium metasilicate-gehlenite, and  $b$  on that of the

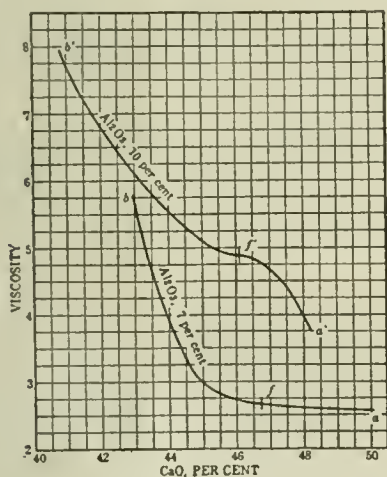


FIG. 1.

Profiles of the isothermal viscosity surfaces at  $1450^\circ$  C. for mixtures containing 7 and 10%  $\text{Al}_2\text{O}_3$ .

boundary system calcium metasilicate-anorthite. For the curve representing 10%  $\text{Al}_2\text{O}_3$  the letters  $a'$ ,  $f'$ , and  $b'$  show the intersection of the same curves, this part of the calcium metasilicate field having a low viscosity. The curve for 15%  $\text{Al}_2\text{O}_3$  (Fig. 2) starts on the left of the binary system metasilicate-anorthite and passes to the gehlenite field, the viscosity decreasing to a minimum at 45%  $\text{CaO}$  and then increasing rapidly with increas-

ing lime. The curve for 18%  $\text{Al}_2\text{O}_3$  (Fig. 2) starts at a composition corresponding to the eutectic between the metasilicate and anorthite and increases to a maximum at the point representing the intersection of the metasilicate-gehlenite binary system (44.5%  $\text{CaO}$ ) and then decreases. At 37.5%  $\text{CaO}$  the curve for 21%  $\text{Al}_2\text{O}_3$  (Fig. 2) begins with a high viscosity because of its proximity to a quintuple

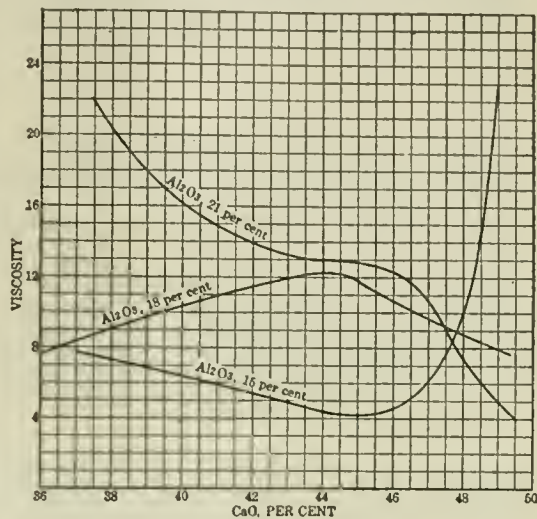


FIG. 2.

Profiles of the isothermal viscosity surfaces at  $1450^\circ$  C. for mixtures containing 15, 18, and 21%  $\text{Al}_2\text{O}_3$ .

point and then decreases with increasing lime. The viscosity surfaces are not of a simple nature at any given temperature and their form depends greatly on the temperature. In the present field it is obviously futile to search for a general relation between viscosity and percentage composition, as the melted mass is not a mixture of oxides but of more complex compounds. Hence, the desulphurising power of a slag cannot be expressed simply in terms of the percentage of lime present. The investigation of what occurs in manufacturing processes involving molten silicates is still further complicated by the fact that even an approach to equilibrium is seldom obtained.—A. B. S.

*Vanadium in sedimentary rocks; A possible source of —.* A. H. Phillips. Amer. J. Sci., 1918, 46, 473—475.

A brown, spotted holothurian, *Sticopus mobii*, found at the Tortugas contains 0.123% V after being dried at  $110^\circ$  C. The author suggests that the occurrence of vanadium in sedimentary rocks and coals may be due to its withdrawal from solution in water by this or similar organisms.—A. B. S.

#### PATENTS.

*Open-hearth steel process.* C. H. Elliott and E. T. McCleary, Assignors to The Youngstown Sheet and Tube Co., Youngstown, Ohio. U.S. Pat. 1,266,928, May 21, 1918. Date of appl., July 20, 1917.

NEAR the end of the heat, the composition of the bath of metal is tested, silicon being added to the bath to retain the carbon during the testing period. The metal may be recarburised after addition of the silicon.—C. A. K.



*Electrolytic deposition of metals.* N. H. M. Dekker, Paris. Eng. Pat. 117,638, Apr. 21, 1917. (Appl. No. 5632 of 1917.)

A METALLIC salt is melted by means of a steam jet so as not to cause the loss of water of crystallisation as would happen if the salt were melted by external heat. Around the anode the aqueous solution of the acid formed by the electrolysis is neutralised by the addition of an oxide, hydroxide, or basic salt of the metal to be produced. The resulting salt is, when present in sufficient quantity, removed and added to the bath and the metal recovered.—F. C. Th.

*Electrodeposition; Process and apparatus for* —. N. Huggins, New York, Assignor to Copper Products Co. U.S. Pat. 1,268,465, June 4, 1918. Date of appl., Apr. 18, 1916.

A ROTARY cathode is formed with end journals which fit into slots in the walls of the electrolytic tank, so that the cathode may be easily removed. Conducting mould-blanks are mounted on and in contact with the cathode, with walls of insulating material adjacent to and between the mould-blanks. The insulating walls extend beyond the mould-blanks, thus forming cavities for the deposited metal. A source of electric current is connected to the cathode and to a suitable anode.—B. N.

*Electrodeposition under compression; Process and apparatus for* —. E. G. Cook, Long Island City, N.Y., Assignor to Copper Products Co. U.S. Pat. 1,271,136, July 2, 1918. Date of appl., May 13, 1916. Renewed May 11, 1918.

A CONDUCTING mould is mounted on a cylindrical conducting cathode and has a series of insulating rings outstanding so as to leave a series of annular cavities in which the metal is electrolytically deposited. During the deposition the deposit is mechanically burnished.—F. C. Th.

*Separation of mineral values from ores by the flotation process.* K. Sundberg, Stockholm. Eng. Pat. 117,781, Feb. 19, 1918. (Appl. No. 2964 of 1918.)

A MIXTURE of minerals is treated by the flotation process in an apparatus with several compartments, and the foams from the first and last compartments respectively are taken separately to two other flotation apparatus and placed in the first or last chamber as the case may be and treated afresh. Three pieces of apparatus, each with five chambers, are used. The foams from the intermediate chambers are passed through again.—F. C. Th.

*[Copper] ores; Process for concentrating* —. B. H. Dosenbach, Butte, Mont. U.S. Pat. 1,268,940, June 11, 1918. Date of appl., Mar. 7, 1918.

ORE containing copper partly as sulphide and partly in a form which is convertible into copper sulphate by the action of sulphuric acid, is treated with acid and metallic copper then precipitated from the solution. The precipitated copper and unaltered sulphide are separated from the gangue by flotation.—W. F. F.

*Ores; Process of and apparatus for concentrating* —. C. J. B. Armstrong, Cobalt, Ontario, Assignor to Metals Recovery Co., Augusta, Me. U.S. Pat. 1,269,150, June 11, 1918. Date of appl., Mar. 15, 1917.

THE froth generated in a flotation tank by treating a pulp containing powdered ore and water and a frothing agent, with a gas, is accumulated in substantial depth and extended area above the pulp

level, and extended in the direction of its travel. The discharge through which the froth escapes is constricted horizontally, so as to prolong the retention period of the froth in the tank.—T. H. B.

*Ores; Process of concentrating* —. J. M. Callow, Salt Lake City, Utah, Assignor to Metals Recovery Co., Augusta, Me. U.S. Pat. 1,269,157, June 11, 1918. Date of appl., Nov. 2, 1916.

THE ore is reduced, in presence of a frothing agent, to a fineness sufficient to liberate the metallic constituents, and sodium bisulphate is added to the fine ore in sufficient quantity to immunise certain mineral components against flotation. On subsequently subjecting the material to flotation treatment, with or without addition of an acid, a selective action on one metallic constituent is obtained.—T. H. B.

*Metalliferous materials; Process and apparatus for utilising sulphurous gases with treatment of* —. *Process and apparatus for sulphatising metalliferous ores.* G. C. Westby, Ludwig, Nev., Assignor to Western Process Co. U.S. Pats. (A) 1,266,731 and (B) 1,266,732, May 21, 1918. Date of appl., Apr. 10, 1917.

(A) METALLIFEROUS ores, not containing fine particles, are charged into a closed tower, and subjected to the action of waste sulphurous gases, the top of the ore column being, at the same time, sprayed with a non-alkaline liquor. The soluble values are leached from the ore and are recovered from the base of the tower. (B) The process is applied to metalliferous fines by arranging them in low heaps on spaced transverse supports in a tower, the heaps in adjacent rows not being in alinement. Steam may be injected with the sulphurous gases.

—C. A. K.

*Nickel; Method of separating* — *from nickel-copper alloys.* J. Dhavernas, Assignor to United States Nickel Co., New Brunswick, N.J. U.S. Pat. 1,266,775, May 21, 1918. Date of appl., July 13, 1916.

COPPER-NICKEL matte is roasted, the oxidised material reduced to metal, and treated with an acid so as to dissolve the nickel. Small quantities of dissolved copper are precipitated by the addition of metallic nickel, and the solution of nickel salts is drawn off and treated to obtain the metal.

—C. A. K.

*Blast-furnace, and blast-furnace operation.* E. E. Slick, Westmont borough, Pa. U.S. Pats. 1,267,004 and 1,267,005, May 21, 1918. Dates of appl., June 29, 1914, and Apr. 16, 1915.

UNDER a charging bell and hopper a stationary distributor, surrounding the bell when lowered, has a number of fingers arranged to deflect portions of the charge towards the centre of the furnace whilst allowing other portions to pass towards the furnace wall. There are thus formed an inner and an outer series of heaps with intermediate portions of coarser materials, and the charge smelts and settles uniformly.—C. A. K.

*Smelting furnace.* T. A. M. Stevenson, Winston-Salem, N.C. U.S. Pat. 1,269,974, June 18, 1918. Date of appl., Mar. 29, 1917.

A WAGON on rails acts as a crucible to hold the product of a smelting furnace, a second one acting as a forehearth. These are run into a compartment provided with outlets for the slag and metal runners and also with a shaft admitting cold air.

—F. C. Th.

*Smelting and refining furnace.* O. J. T. G. R. Martin, Vancouver. U.S. Pat. 1,271,051, July 2, 1918. Date of appl., July 28, 1917.

THE furnace has two superposed compartments heated by electric current from carbon electrodes built in the walls. The top compartment has a charging opening above and a discharge aperture at the lower end, whence material passes into the lower compartment. Means are provided for opening and closing the doors and for indicating the height of the charge in each compartment.

—F. C. Th.

*Electrodes for electric welding; Treating* — J. H. Gravell, Brooklyn, N.Y., Assignor to Thomson Spot Welder Co., Boston, Mass. U.S. Pat. 1,267,400, May 28, 1918. Date of appl., Oct. 20, 1917.

A THIN layer of fusible metal, of melting point lower than that of the electrode, is applied between the electrode and the surface of the work engaged by it, and pressure is applied.—B. N.

*Copper alloy; Process for producing a hard* — O. C. M. Knudsen, Brooklyn, N.Y. U.S. Pat. 1,267,669, May 28, 1918. Date of appl., Mar. 22, 1917.

A HARD alloy containing only a small amount of aluminum is produced by melting aluminum and adding copper broken into small particles. The temperature is then raised to melt the copper and the whole well mixed.—F. C. Th.

*Tungsten ingots; Process for welding* — C. A. Pfanstiehl, Waukegan, Ill., Assignor to Pfanstiehl Co., Inc., North Chicago, Ill. U.S. Pat. 1,267,801, May 28, 1918. Date of appl., Oct. 15, 1915.

AN ingot consisting of compressed tungsten particles is compressed longitudinally and at the same time heated to the sintering temperature by an electric current passed through the ingot from a pair of carbonised tungsten electrodes pressed against its ends.—F. C. Th.

*Tungsten powder; Production of metallic* — P. G. Keyes, East Orange, N.J., Assignor to Cooper Hewitt Electric Co., Hoboken, N.J. U.S. Pat. 1,270,812, July 2, 1918. Date of appl., July 6, 1914.

TUNGSTEN oxide is reduced by intimately mixing it with an alkali metal dissolved in liquid ammonia, the latter being distilled off and the residue heated slowly to 500°–600° C.—T. H. B.

*Electrical reduction of ores; Method and apparatus for* — B. G. Cobb, Dawson City, Canada, Assignor to J. F. Skerrett, H. Giffin, H. M. Kidder, R. I. Barr, and E. Inescourt, New York, and Andes Exploration and Smelting Corporation. U.S. Pat. 1,267,976, May 28, 1918. Date of appl., May 23, 1913. Renewed Oct. 11, 1917.

AN arc is produced between electrodes in the charge of crushed ore. A supplementary current is passed between a pair of electrodes of an independent circuit placed on the surface of the ore mass at a definite distance from the positive electrode of the main circuit, and the polarity of the supplementary electrodes is frequently reversed.—F. C. Th.

*Coating process [for metals].* J. P. A. McCoy, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,268,030, May 28, 1918. Date of appl., July 3, 1914.

A FINELY divided metallic oxide is fed into a reducing flame which melts it and reduces the oxide

to metal, which is sprayed in the liquid state on to the surface by the flame.—F. C. Th.

*Ores; Process of treating* — H. E. Cleaves and F. W. Horton, Washington, D.C. U.S. Pat. 1,268,323, June 4, 1918. Date of appl., Mar. 14, 1917.

ONE containing one or more of the metals lead, zinc, and copper, is treated at a temperature of 800°–1100° C. with gaseous hydrogen chloride. The lead and zinc chlorides are volatile at that temperature and distil off to be subsequently condensed.

—F. C. Th.

*Fume-arrester for smelting plants.* A. Ferrari, Collinsville, Ill. U.S. Pat. 1,268,447, June 4, 1918. Date of appl., Sep. 1, 1917.

THE fumes pass from the furnace into a horizontal condensing passage and thence to a chimney. The interior of the passage and part of the stack are sprayed with water from perforated tubes.

—F. C. Th.

*Metal composition [alloy].* H. K. Sandell, Assignor to H. S. Mills, Chicago, Ill. U.S. Pat. 1,268,495, June 4, 1918. Date of appl., Mar. 23, 1917.

A NICKEL-TUNGSTEN alloy consisting essentially of more than 50% Ni, preferably 4 parts Ni and 1 part W, and more than 3% of graphitic carbon.

—F. C. Th.

*Metals; Process for chemically extracting pure* — from their ores. F. G. Clark and A. T. Stuart, Assignors to The Toronto Power Co., Ltd., Toronto, Canada. U.S. Pat. 1,269,054, June 11, 1918. Date of appl., July 5, 1917.

THE ore and a gaseous reagent in regulated quantities are passed through a reaction zone at a temperature sufficient to cause the reaction but below the fusing point of the metals. The gaseous reagent combines with oxygen, sulphur, phosphorus, and manganese, and the metals are separated in a pure state.—T. H. B.

*Lead or zinc or both metals; Continuous process for recovering* — from ores or slag. F. Ohlmer, Griesheim, Germany. U.S. Pat. 1,269,110, June 11, 1918. Date of appl., Sep. 26, 1914.

A wet mixture of the metalliferous material with carbonaceous fuel is fed into a revolving furnace in which it is heated at a temperature below the reduction temperature of zinc oxide, to granulate the charge, which is then fed into a second revolving furnace, and heated sufficiently in a reducing atmosphere to reduce the oxides, without smelting, the metallic fumes being afterwards oxidised in an oxidising zone.—T. H. B.

*Metals; Method of recovering* — from solution. R. Gahl, Miami, Ariz. U.S. Pat. 1,269,418, June 11, 1918. Date of appl., Aug. 15, 1917.

THE metals, e.g., copper or iron, are recovered from dilute solutions of, for instance, the sulphates by adding a large excess of a suspension of finely divided limestone. The limestone precipitate is divided into richer and poorer fractions, the poorer parts being added to a fresh quantity of solution.

—F. C. Th.

*Rust prevention.* C. I. Gesell, Mamaroneck, N.Y. U.S. Pat. 1,269,926, June 18, 1918. Date of appl., Jan. 7, 1918.

THE metal is provided with a coating of non-metallic paint, having holes or fissures in it, and is made the negative electrode of a voltaic cell, e.g., by holding it in contact with an unpainted body of zinc in sea water.—T. H. B.



*Metallic articles; Box for treating — by heat. Means for heat-treating metallic articles.* J. C. Henderson, Washington, D.C., Assignor to Driver-Harris Co., Harrison, N.J. U.S. Pats. (A) 1,270,519 and (B) 1,270,520, June 25, 1918. Dates of appl., July 8, 1916, and Feb. 1, 1918.

(A) A CLOSED container for use in the heat treatment of metals is made from a non-warping and heat-resisting alloy containing from 5 to 30% of Cr and from 50 to 90% of a metal of similar atomic weight to iron and classified with it in Mendelejeff's table. (B) A carrier for articles to be heat treated at high temperatures consists of a cast perforated basket of an alloy of nickel and chromium of low heat conductivity.—C. A. K.

*Case-hardening material.* H. Rodman, Edgewood, Pa., Assignor to Rodman Chemical Co., Verona, Pa. U.S. Pat. 1,270,874, July 2, 1918. Date of appl., Aug. 29, 1914.

THE metal to be carburised is packed in a mixture of charred resins and lignose, e.g., the by-product of wood fibre manufacture known as black ash, and the whole heated.—T. H. B.

*Graphite; Method of recovering — from slag.* E. C. Ewen, Saginaw, Mich. U.S. Pat. 1,271,146, July 2, 1918. Date of appl., Dec. 17, 1917.

MOLTEN metal from a furnace is poured into a ladle, the metal is then removed from the ladle whilst holding back the slag and another charge of metal is poured upon the latter. The metal is then again removed, whilst holding back the slag, which is afterwards screened and finally subjected to the action of an air separator to recover the graphite.

—T. H. B.

*Cadmium-evolving process.* W. McA. Johnson, Hartford, Conn. U.S. Pat. 1,271,172, July 2, 1918. Date of appl., July 30, 1914.

A SULPHUR-BEARING ore of cadmium and zinc is incompletely roasted so as to reduce the sulphur to about 3%. The material is then reduced in a current of gas at a temperature sufficiently high to evolve cadmium vapour but too low to evolve zinc. The residue is treated at a higher temperature to produce zinc containing only a trace of cadmium.

—F. C. Th.

*Tin; Method of and apparatus for smelting —.* E. N. Morrill, Warren, N.H., Assignor to Andes Exploration and Smelting Corporation. U.S. Pat. 1,271,200, July 2, 1918. Date of appl., Nov. 27, 1917.

A CRUCIBLE furnace lined with magnesite and cooled by water-cooled plates is heated to a temperature slightly below the boiling point of tin by passing an electric current through a bath of molten slag. From time to time tin ore, slag, and reducing material are added, each charge being smaller in quantity than the molten material already present. The approximate proportions of the constituents of the charge are 60% tin concentrates, 1000 lb.; slag, 100 lb.; lime 35–50 lb.; silica, 50–70 lb.; and coal, 125–150 lb.—F. C. Th.

*Blast-furnaces, kilns, or the like; Elevators for —.* Soc. J. Munier et Cie., Frouard, France. Eng. Pat. 111,673, Nov. 23, 1917. (Appl. No. 17,299 of 1917.) Under Int. Conv., Nov. 28, 1916.

*Blast-furnace skips.* Soc. J. Munier et Cie., Frouard, France. Eng. Pat. 111,674, Nov. 24, 1917. (Appl. No. 17,370 of 1917.) Under Int. Conv., Nov. 25, 1916.

*Electroplating articles or pieces having holes; Apparatus for use in —.* Platt Bros. and Co., Ltd., Oldham, and G. L. A. R. B. Colin, Uccle, Belgium. Eng. Pat. 117,658, July 23, 1917. (Appl. No. 10,535 of 1917.)

*Concentration of crushed ores; Apparatus for —.* W. McDermott, London. Eng. Pat. 118,074, May 14, 1918. (Appl. No. 8076 of 1918.)

*Zinc; Electrothermic extraction of —.* E. S. Berglund, Trollhättan, Sweden. U.S. Pat. 1,271,267, July 2, 1918. Date of appl., July 25, 1917.

SEE Eng. Pat. 109,435 of 1917; this J., 1918, 185 A.

## XI.—ELECTRO-CHEMISTRY.

*Melting points of cristobalite and tridymite [and new high-temperature electric furnace].* Ferguson and Merwin. See VIII.

*An electrical zinc spraying process.* Kasperowicz. See X.

*Electro-analysis of bismuth without platinum electrodes.* Poch. See XXIII.

## PATENTS.

*Electrolysis of water; Apparatus for the —.* K. Kimura, Kobe, Japan. Eng. Pat. 117,533, Sep. 19, 1917. (Appl. No. 13,441 of 1917.)

SEVERAL anodes and cathodes are suspended from a detachable tank cover, the latter being provided with a series of pole-chamber heads which are separately connected to and in independent communication with box-like screens surrounding the suspended anodes. The lower edges of the anode screens and the suspended pole plates are kept in position by distance pieces, and the interior of the tank is thus divided by the screens into a series of anode chambers and a single cathode chamber.

—B. N.

*Electrolyte; Film-forming —.* J. Coulson, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,266,557, May 21, 1918. Date of appl., May 3, 1916.

THE claim is for an electrolyte containing ammonium citrate the concentration of which is substantially expressed by the equation  $P = 3310 \times 2.72^{0.0181v}$ , where P equals the percentage concentration of the electrolyte and v equals the voltage which is to be used. The percentage concentration should not be materially over 2% and must be less than 10%.—B. N.

*Electrode; Totally-submerged —.* H. H. Stout, Assignor to Nichols Copper Co., New York. U.S. Pat. 1,267,141, May 21, 1918. Date of appl., July 26, 1916.

THE electrode is suspended by means of lateral lugs resting on ledges carried by the side walls of the electrolytic tank, and the side walls are provided with portions extending above the ledges, so that the top edge of the electrode is below the top of the tank. The electrode is thus totally submerged in the electrolyte.—B. N.

*Anode connector.* J. H. Gillis, Assignor to British America Nickel Corporation, Ltd., Toronto, Canada. U.S. Pat. 1,267,653, May 28, 1918. Date of appl., Oct. 15, 1917.

A SOLUBLE anode, used in conjunction with a suitable electrolyte, is provided with an anode connector of aluminium having one or more contact pieces of copper secured to it.—B. N.

*Electric furnace; Device for closing the orifices in the roof and walls of an — through which the electrodes pass.* T. H. Watson and Co. (of Sheffield), Ltd., and W. Travis, Sheffield. Eng. Pat. 118,023, Nov. 8, 1917. (Appl. No. 16,321 of 1917.)

*Melting refractory substances by electric arcs; Methods for —.* J. G. Webb, Richmond, Va., U.S.A. Eng. Pat. 115,017, Sep. 25, 1917. (Appl. No. 13,780 of 1917.) Under Int. Conv., Mar. 16, 1917.

SEE U.S. Pat. 1,234,836 of 1917; this J., 1917, 1017.

*Process of [producing barium sulphide by] electrical reduction.* U.S. Pat. 1,267,347. See VII.

*Producing crystalline alumina.* U.S. Pat. 1,269,141. See VII.

*Treating electrodes for electric welding.* U.S. Pat. 1,267,400. See X.

## XII. FATS; OILS; WAXES.

*Plant products from Colombia.* A. L. Bacharach, Analyst, 1918, 43, 289—291.

*Oil of Jessenia polycarpa.*—This oil is obtained from the nuts of the "sejen" or "unamo" palm and is used medicinally and for cooking. It is a pale yellow slightly fluorescent oil, closely resembling olive oil in its behaviour in the elaidin test and in chemical and physical characters. Samples from three separate consignments gave the following results:— $[n]_D^{20} = 1.4682$ ; free fatty acids (as oleic acid) 1.9 to 2.6%; saponif. value 188.5 to 190.5; iodine value (Wijs) 73.5 to 74.8; one of the samples had sp. gr. (15°/4° C.) 0.9161; Hehner value 93.8; iodine value of fatty acids 79.5; molec. equiv. of insoluble fatty acids 273.

*Seeds of Caryodendron Orinocense.*—These seeds, known locally as "tacy," are roasted and used for food. They have a greyish-brown brittle husk (about 15.5% of the seed), and average about 3.1 grms. in weight. A sample of the kernels had the following composition:—water, 4.43; ash, 2.95; crude fibre, 2.40; oil, 53.3; proteins, 12.90; and non-nitrogenous substances, 24.00. Extracted "tacy" oil had the following characters:—sp. gr. (15°/4° C.) 0.9220;  $[n]_D^{20} = 1.4744$ ; free fatty acids (as oleic acid), 15.8%; saponif. value, 188.1; Hehner value, 94.4; and iodine value (Wijs), 168.5.

—C. A. M.

*The glycerides of butter fat.* Amberger See XIXA.

*Influence of foreign substances on activity of catalysts. IV. Experiments with palladium hydrosol in presence of mercury and mercuric oxide.* Paal and Harlmann. See XX.

*The copper method for estimating glycerol.* Weiss. See XXIII.

## PATENTS.

*Fatty acids and their glycerides; Method for saturation by means of hydrogen of unsaturated — and apparatus therefor.* Soc. de Stéarinerie et Savonnerie de Lyon, and P. Berthon, Lyons. Eng. Pat. 107,969, June 25, 1917. (Appl. No. 9120 of 1917.) Under Int. Conv., June 27, 1916.

HYDROGENATION of oils or fatty acids at a constant speed of reaction is effected by the progressive raising of the temperature and a methodical introduction of the catalyst, hydrolysis of the oil under treatment being avoided by removal of the water of reaction formed. In hydrogenating oils for edible purposes the apparatus described ensures the automatic distillation of the free fatty acids, thus avoiding contamination of oil by traces of metallic catalyst or the necessity for a subsequent neutralisation. The apparatus consists of a compressor for hydrogen, two reaction chambers in series or parallel, a tubular heat-exchanger where the temperature of the gas is automatically regulated and the greater part of the free fatty acid distilled over is condensed, two water-cooled tubular condensers for further condensation of the same, a chamber containing caustic soda where certain volatile impurities of the oils are absorbed, a water separator, a heat-exchanger for preliminary cooling, and a refrigerator for freeing the gas from the last traces of moisture prior to re-entering the cycle where fresh gas is added previous to entering the compressor. Means are provided at the base of the digester for ensuring perfect contact of the reagents and progressive and methodical distribution of the catalyst. The catalytic mixture is prepared in a steam-heated vessel fitted with an energetic agitating device formed by baffles and counter-baffles.—A. de W.

*Oil; Apparatus for extracting —.* W. J. Garnett, Eastport, Me. U.S. Pat. 1,271,028, July 2, 1918. Date of appl., Oct. 3, 1917.

A "COOKER VAT" and a reservoir are so arranged that the upper end of the latter is below the plane of the bottom of the former. A steam supply pipe extends into the "cooker vat," which is fitted on the bottom with a drain pipe leading to the reservoir. At the point where the drain pipe enters the reservoir it is fitted with an elbow, a bushing carried by the lower end of the elbow, and a reduced valved discharge pipe carried by the bushing.

—A. de W.

*Fats; Method of super-refining —.* S. R. Barnett, Belleville, Wis. U.S. Pat. 1,271,118, July 2, 1918. Date of appl., June 4, 1917.

IN order to remove free fatty acids, oils and fats are agitated with hot lime water in the presence of a catalyst (sodium bicarbonate). Carbon dioxide is then passed into the mixture to precipitate excess of lime and to prevent oxidation. The sludge, containing the calcium soaps, is separated.

—L. A. C.

*Soap containing sulphur; Manufacture of —.* T. Tanaka, Tokyo, Japan. Eng. Pat. 117,060, Apr. 30, 1918. (Appl. No. 7249 of 1918.) Under Int. Conv., Aug. 1, 1917.

NEUTRAL soap stock is mixed with melted rosin soap which has been incorporated with sulphur, and a mixture of sodium perborate, or similar oxygen-yielding salt, with a small amount of vaseline is added, and the whole kneaded into a homogeneous mass, and moulded into tablets. This soap does not emit hydrogen sulphide when used.—C. A. M.



*Treatment of edible oils and fats and production of butter substitutes.* Eng. Pat. 117,014. See XIXa.

*Viscosity measuring instrument.* U.S. Pat. 1,270,800. See XXIII.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Turpentine; Determination of — and the detection of adulterants.* J. Tausz. Chem.-Zeit., 1918, 42, 349—351.

TURPENTINE and other unsaturated hydrocarbons combine with mercuric acetate, or are oxidised by it; one mol. of acetic acid is set free for each mol. of terpene, etc., present and a determination of this acidity gives a measure of the turpentine present. Ten c.c. of pure turpentine and 10 c.c. of the sample under examination are each diluted with methyl alcohol to 100 c.c. (the reaction proceeds more quickly in the presence of methyl alcohol) and 10 c.c. of each of these solutions is mixed with 50 c.c. of saturated mercuric chloride solution (in methyl alcohol) and 50 c.c. of N/10 potassium hydroxide solution (also prepared with methyl alcohol). The mixtures are diluted with 100 c.c. of water, shaken, kept in the dark for 1 hr., phenolphthalein is added, and sodium chloride solution is run in until a red coloration develops; the mixtures are then titrated with N/10 sulphuric acid. A control test, without turpentine, is made at the same time. Usually, turpentine substitutes do not react with mercuric acetate; rosin spirit yields a small quantity of free acetic acid, and if this adulterant is present, turpentine containing approximately the same quantity as the sample should be used for the comparison titration. When pure turpentine is heated under a reflux condenser for 3 hrs. with mercuric acetate solution and methyl alcohol, and then steam-distilled, no trace of oil is found in the distillate. Hydrocarbons, however, such as mesitylene, cumene, eymene, petroleum spirit, etc., pass almost quantitatively into the distillate and their quantity may be measured. The loss is about 0.2 c.c. for each 10 c.c. of hydrocarbon, but it varies with each apparatus and should be determined separately. This method permits further examination of the distilled adulterant. The reaction between turpentine and mercuric acetate may also be used gravimetrically. Five c.c. of the sample is diluted to 100 c.c. with methyl alcohol and 5 c.c. of this solution is mixed with 50 c.c. of saturated aqueous mercuric acetate solution and heated for 4 hrs. in a closed flask in a boiling water bath; after cooling, the mercurous acetate formed is collected, washed with alcohol and ether, dried, dissolved in nitric acid (1:1), the solution treated with sodium chloride, and the mercurous chloride collected and weighed. A control test is made at the same time, using pure turpentine; the quantity of turpentine in the sample is proportional to the amount of mercurous chloride found.—W. P. S.

*Baghouses for zinc oxide.* Cregan. See X.

#### PATENTS.

*Ink for printing on paper; Process for the manufacture of —.* J. Hope, Rouen. Eng. Pat. 117,023, Dec. 7, 1917. (Appl. No. 18,156 of 1917.)

SULPHUR colours produced by the action of sulphur or sulphides on organic substances (e.g. *Cachou de Lava*), are rendered soluble by means of sodium sulphide or converted into precipitates or lakes, and incorporated with oils, resins, varnishes, or other thickening agents to form printing inks.

Paper to which such inks have been applied may be completely bleached with chloride of lime and made into new paper.—C. A. M.

*Writing inks [; Indelible —].* M. Shinozaki, Tokyo, Japan. Eng. Pat. 117,117, July 3, 1917. (Appl. No. 9534 of 1917.)

SOLUBLE Induline or other alkali-soluble dyestuff (90 grms.) is dissolved in caustic alkali solution (5 grms. in 2 galls.), and the solution incorporated with a mixture of lampblack or other finely-divided carbon (135 grms.) and caustic alkali solution (1 grm. in 0.4 gall.), with or without the addition of soft soap (15 grms.), and the whole heated and stirred until perfectly mixed.—C. A. M.

*Paint [; Emulsion —].* R. Illeemann and J. A. Montgomerie, Glasgow. Eng. Pat. 117,785, Mar. 6, 1918. (Appl. No. 3900 of 1918.)

A PAINT miscible with water and yielding a water-proof coating is obtained by adding to a mixture of one part of pitch (or bitumen) and one part of tar, rendered fluid by the aid of heat, a liquid suspension of one part of clay and one part of water and heating the mixture, with agitation, for only a sufficient length of time to ensure incorporation without driving off the water. Agitation is then continued until cold. Other colouring matters may be added during the course of manufacture to obtain different coloured paints or the proportions given may be varied.—A. de W.

*Coumarone resin and process of making same.* C. Ellis, N. L. Foster, and Ellis-Foster Co., Montclair, N.J., U.S.A. Eng. Pat. 117,016, Nov. 13, 1917. (Appl. No. 16,653 of 1917.)

LIQUID aromatic hydrocarbons containing compounds such as coumarone and indene (e.g., coal tar naphtha distilling between 160° and 180° C.) are treated with a polymerising agent, such as aluminium chloride, or, preferably, sulphuric acid, in sufficient quantity to polymerise such compounds. The unchanged liquid is removed, and the soft resinous mass freed from the more volatile constituents by distillation, leaving a hard resin with an iodine value not much below 47. The distillation is preferably effected slowly up to 180° C. under a reduced pressure of about 29 in. of mercury.—C. A. M.

*Phenols and formaldehyde; Manufacture of condensation products from —.* T. I. K. M. Hiltermann, Amsterdam. Eng. Pat. 117,857, Aug. 16, 1917. (Appl. No. 11,789 of 1917.)

SOLID and transparent phenol-formaldehyde condensation products are obtained from crude material (eresol) without subsequent purification by employing the neutral salts of aromatic hydroxycarboxylic acids as condensing agents. An example of the process given is: 100 parts by weight of phenol, 80 parts by weight of 40% formaldehyde, and 5 to 10 parts of an approximately 50% aqueous solution of neutral sodium or potassium salicylate are heated together until reaction commences, the heat being then reduced. At the termination of the reaction the water contained in the raw materials is driven off, the mass cast into moulds, and hardened in open moulds at 60°—80° C.—A. de W.

*Phenolic condensation products; Process of hardening —.* J. P. A. McCoy, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,269,627, June 18, 1918. Date of appl. July 3, 1914.

A SUBSTANCE capable of decomposing with evolution of formaldehyde, e.g., paraformaldehyde, together

with a tannin condensing agent having no deleterious effect upon the dielectric strength or stability of the resulting product, e.g., catechol-tannin, is added to a fusible phenolic condensation product.

—A. de W.

*Phenol-aldehyde condensation product.* A. W. C. van Voorhout, Delft, Netherlands. U.S. Pats. 1,271,392 and 1,271,393, July 2, 1918. Dates of appl., Dec. 22, 1916, and Jan. 23, 1918.

See Eng. Pat. 110,041 of 1916; this J., 1917, 1185.

## XV.—LEATHER; BONE; HORN; GLUE.

*Chrome [tanning] liquors; Action of neutral salts on —.* A. W. Thomas and M. E. Baldwin. J. Amer. Leather Chem. Assoc., 1918, 13, 248—255.

MEASUREMENTS of hydron concentration were made by means of the hydrogen electrode on chrome liquors to which varying amounts of neutral salts had been added. Sodium and ammonium chlorides, especially the former, caused distinct increases in the hydron concentration, whereas the sulphates of the same metals had a less marked effect in the opposite direction except when the concentration of sodium sulphate was greater than half-molar. Magnesium sulphate in increasing concentration caused first a slight decrease and then a slight increase to nearly the original hydron concentration. The effect of sodium chloride was also studied with regard to time. A rapid increase in hydron concentration takes place immediately on adding the salt, after which there is a slow decrease to a value in excess of that of the original liquor.—F. C. T.

*Leather analysis; Preparation of samples for —.* F. P. Veitch and R. W. Frey. J. Amer. Leather Chem. Assoc., 1918, 13, 232—239.

SAMPLES of leather for analysis were prepared by planing heavy sole leather and also by shredding with a machine constructed of four circular saws with "staggered" teeth mounted together on a shaft. Analysis showed that not only did the shredded samples contain much less moisture than the planed samples, but also less soluble solids, the difference in the latter varying from 0.8 to 4.5%. This effect was attributed to heating, and a new machine was constructed of four  $10 \times \frac{1}{8}$ " saws of the splitting-saw type, with large teeth, about one to the inch. The saws had a slight set and were driven at 1500 revolutions per minute. The cutting action was much improved, only a barely perceptible heating occurred, and the content of soluble matter proved on analysis to be always slightly greater than that of a planed sample.—F. C. T.

*Leather; Determination of fat in —.* L. E. Levi and A. C. Orthmann. J. Amer. Leather Chem. Assoc., 1918, 13, 313—318.

THE new fat solvent proposed by Wilson and Kern (this J., 1918, 343 A), consisting of equal parts of ether and carbon tetrachloride, is more readily inflammable than Wilson and Kern allow. Moreover, it continues to extract soluble matter from leather for a very long time; for instance 50 grms. of leather after extraction for 19 hours with petroleum ether was extracted twice with the new solvent for four hours, and a third time for 15 hours, when the evaporation residue from the last extract amounted to 0.64% of the leather, two-thirds of the residue being soluble in water and the remainder soluble in alcohol. The authors claim

that concordant results with the new solvent are impossible unless the time of extraction is precisely specified for every kind of leather, and also that the solvent extracts much that is neither fat nor oxidised fat. Dry quebracho extract after careful treatment with petroleum ether showed 1.88% of soluble matter on extraction with the new solvent and the residue was very like that obtained similarly from leather. The new solvent always extracts considerable quantities of dyestuff and chrome soap from chrome leathers. One such residue showed 1.57% of ash, mostly chrome oxide. It is maintained that Wilson and Kern's criticism of the authors' method is based on too little experimental evidence.—F. C. T.

## PATENTS.

*Glue and like substances; Manufacture of products adapted to precipitate —.* H. Wade, London. From Deutsch-Koloniale Gerb- und Farbstoff-Ges., Karlsruhe, Germany. Eng. Pat. 116,936, June 29, 1917. (Appl. No. 9420 of 1917.) Addition to Eng. Pat. 8818 of 1914 (see Fr. Pat. 471,924; this J., 1915, 438).

WHITE or slightly coloured products capable of precipitating glue are obtained by condensing formaldehyde with an  $\alpha$ - or  $\beta$ -naphtholmonosulphonic acid, using about 1 mol. of the former to 2 mols. of the latter. The reaction is carried out in strongly acid solution containing at least 25% of water, preferably at the ordinary temperature.

—F. C. T.

*Hides and the like; Apparatus for the treatment of —.* B. H. Tilson and T. Melbourne, Warrington. Eng. Pat. 117,581. (Appl. Nos. 2054, Feb. 5, and 4015, Mar. 7, 1918.)

Two forms of apparatus are described for agitating the liquors with which hides are treated in the pits in the processes of leather manufacture. In one form, a pit with a concave bottom is used, provided at the sides with two swinging arms which are joined at the lower ends by cross-pieces in the form of paddles or blades. The swinging arms are rocked by an oscillating shaft, and hides are hung in the pits between the swinging arms and above the paddles or blades. In the other form of apparatus, the stirrer-device at the bottom of the pit extends over the entire breadth and about half the length of the pit, and the blades of the stirrer are provided with hinged one-way valves, and are rocked by means of an eccentric rod.—F. C. T.

*Fibrous [roofing] material and method of making the same [from scrap leather].* U.S. Pat. 1,269,905. See IX.

## XVI.—SOILS; FERTILISERS.

*Potash in soils; Influence of gypsum upon the solubility of —.* P. R. McMiller. J. Agric. Res., 1918, 14, 61—66.

SURFACE samples of five loam soils of different character were intimately mixed with 1% of gypsum, and sufficient water was added to make the moisture content equal to the moisture equivalent of the soil. The samples, together with controls, were kept in jars for three months. They were then shaken with water, and the amount of potash in the filtered solutions was determined by the platonic chloride method. Marked increases were found in the amounts of soluble potash in the treated over the untreated samples.—J. H. J.



*Nitrate and nitrite assimilation [by plants]. XIII. Iron and oxygen as necessary agents for the reduction of alkali nitrites by auto-oxidisable compounds [sugars, etc.].* O. Baudisch. Ber., 1918, 51, 793—805.

THE author (this J., 1917, 730) has already reported that when solutions of dextrose are boiled with ferrous sulphate and sodium carbonate, deep-coloured solutions are obtained, containing iron in the form of an internal complex ("masked" iron), which have the power of reducing alkali nitrites to nitric oxide and ammonia, or nitrobenzene to aniline. It is now shown that this property is shared by aldoses and ketoses in general, such as dihydroxyacetone, glycollic aldehyde, levulose, and maltose, but is not exhibited by closely related alcohols, such as glycerol or mannitol. In addition, the power of reducing nitrites in the presence of "masked" iron is possessed by catechol, quinol, pyrogallol, gallic acid, quercetin, chrysarobin, etc. All these compounds are auto-oxidisable, that is they take up oxygen and yield hydrogen peroxide in the presence of water, and it is remarkable that, with the exception of the sugars, these compounds can only reduce in the presence of oxygen. In the case of the sugars, the necessary oxygen is supplied by the sugar molecule itself. Furthermore, traces of iron in the masked form must be present; manganese or copper will not do. Some of the above polyhydric compounds can make the complex themselves out of iron hydroxides, but not all of them. Phloroglucinol, for example, cannot produce a complex from ferric hydroxide, but it can from potassium ferrieyanide. It is not necessary that the complex shall involve the auto-oxidisable substance itself; the iron may be applied as a complex with catechol-o-carboxylic acid, for example. The fact remains, therefore, that these auto-oxidisable substances, which are so widely dispersed in nature, can in the presence of oxygen bring about oxidations on the one hand (e.g., methyl alcohol to formaldehyde) and the reduction of nitrites, in the presence of oxygen and iron complexes, on the other, and the significance of this in bio-chemical questions is obvious. (See further J., Chem. Soc., Oct., 1918.)—J. C. W.

*Aluminium ion; Distribution of the — in plants.* J. Stoklasa. Biochem. Zeits., 1918, 88, 292—322.

TABLES are given showing the amount of aluminium in a large number of plants. The author calls attention to the fact that the xerophytes contain only small amounts, whereas the hydrophytes and hygrophilic plants contain relatively large amounts. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.

*Plants; Immunity of — towards the immediate principles which they elaborate.* R. Combes. Comptes rend., 1918, 167, 275—278.

IT was found that plants of *Agrostemma githago* would grow satisfactorily under aseptic conditions in Knop's culture solution containing up to 1% of the glucoside, agrostemmasaponin, which the plant elaborates, whereas plants such as peas, radishes, and buckwheat showed marked signs of injury when the culture solution contained only 0.01% of the glucoside. The injury was shown by the disappearance of the root hairs, arrested development of the roots, and diminution in the dry weight of the root.—W. G.

*Potash salts in Hungary.* Von Konek-Norwall. See VII.

*Basic exchange in permutite.* Rothmund and Kornfeld. See XIX.

## PATENTS.

*Nitrate of ammonia; Treatment of —.* F. W. Howorth, London. From Norsk Hydro-Elektrisk Kvaestofaktieselskab, Christiania. Eng. Pat. 117,823, Apr. 10, 1917. (Appl. No. 5063 of 1917.)

SEE U.S. Pat. 1,263,363 of 1918; this J., 1918, 385 A. The substance added is bog iron ore or kieselguhr, and the mixture may be subjected to a preliminary drying operation at a low temperature (20°—40° C.) before the final drying.

*Phosphatic material; Process of concentrating —.* H. A. Webster, Columbia, Tenn. U.S. Pat. 1,266,730, May 21, 1918. Date of appl., June 4, 1917.

LOW-GRADE phosphatic limestone is calcined until a substantial portion of the carbon dioxide is expelled, and the burnt rock is finely ground and suspended in a moving fluid. The lighter phosphorus-free particles are carried away by the stream whilst the heavier phosphatic material settles out.—C. A. K.

*Process of producing gas and phosphatic fertiliser material.* U.S. Pat. 1,269,795. See IIA.

## XVII.—SUGARS; STARCHES; GUMS.

*Unfermentable sugars (glucose) of cane molasses, their preparation, properties, and determination.* C. Muller. Archief Suikerind. Neder-Indië, 1918, 26, 346—358. Int. Sugar J., 1918, 20, 377—378.

IN order to examine the question of the formation of glucose during the course of manufacture in the cane sugar factory, lime was heated with a 10% solution of pure levulose, with a 20% solution of invert sugar, and with a thick-juice (62° Brix), respectively, imitating in each case the conditions prevailing in practice as closely as possible. The concentration of the sugar, the time of heating, and the quantity of lime used, greatly influenced the yield of "glucose," but the temperature had little effect. An examination of the product obtained from cane thick-juice indicated that it is not simply an isomer of the formula,  $C_6H_{12}O_6$ , as the product obtained from levulose appears to be, nor a single compound to which a formula may be assigned, but rather a mixture of a series of compounds of high molecular weight (containing 12, 18, or more, carbon atoms), each possessing very different properties. For its determination, 50 grms. of cane molasses diluted to 500 c.c. with water was acidified with sulphuric acid, fermented with 25 grms. of pressed bottom yeast, and the volume completed to 550 c.c. After clarifying with normal lead acetate, and eliminating the excess of lead with sodium carbonate, the cupric-reducing power was ascertained by heating for 30 minutes only to 65° C., as recommended by Pellet (this J., 1917, 467). Operating in this way, the amount of "glucose" or unfermentable sugars was found to lie between 3.2 and 4.9% for cane molasses and 0.1 and 0.2% only for the beet product, the quantity in the former case for the same factory being found to vary somewhat according to the ripeness of the cane.—J. P. O.

*Dextrose; Estimation of — by means of hypoiodite.* R. Willstätter and G. Schudel. Ber., 1918, 51, 780—781.

THE dextrose solution is mixed with about twice the amount of N/10 iodine solution necessary for oxidation to gluconic acid,  $C_6H_{12}O_6 + I_2 + 3NaOH = 2NaI + 2H_2O + HO(CH_2(CH_2OH)_4CO_2Na$ , a quantity

of  $N/10$  sodium hydroxide which is 1.5 times as much as the iodine is slowly added, the mixture is left for 12–15 minutes (or 20, if the proportion of sugar is very small) and then the excess of iodine is titrated after acidifying slightly with sulphuric acid. Taking 10 c.c. of sugar solution, the average error is less than 0.1% with 1% solutions or less than 1.5% with 0.1% solutions. Under these conditions ketoses and sucrose are not affected, and, therefore, the method may be applied to the estimation of aldoses in mixtures.—J. C. W.

*Lactose in admixture with sucrose and invert sugar; Determination of —.* J. Grossfeld. Z. Unters. Nahr. Genussm., 1918, 35, 249–256.

From the results of experiments with pure lactose and sucrose, empirical formulae have been deduced for calculating the percentage of lactose in a mixture containing also sucrose and invert sugar, the data required being the polarisation and reducing power of the mixture after inversion. The inversion is always made under the same conditions, namely, 50 c.c. of the solution mixed with 2 c.c. of hydrochloric acid (sp. gr. 1.125) and heated in a boiling water-bath for 30 mins. The reducing power is expressed in terms of invert sugar and the lactose is then calculated from the empirical formula:—

$$L = 1.20Z \frac{D_x - D_1}{D_1 - D_1'}$$
 where  $Z$  is the quantity of "apparent invert sugar" calculated from the reducing power,  $D_x$  = the specific rotation found for the "apparent invert sugar,"  $D$  and  $D_1$  = the specific rotations of the inverted products from lactose (+70.6°) and sucrose (−16.7°), respectively, under the above conditions;  $D_1 - D_1'$  is thus 87.3°, and the formula becomes:  $L = 0.01375Z(D_x + 16.7)$ . The quantity of sucrose present is found from the formula:  $S = 0.01145Z(70.6 - D_x)$ , since 100% of sucrose gives 100% of "apparent invert sugar" under the conditions given. Tables are given which show the rotation and reducing power of the inverted products from various mixtures of lactose with sucrose, etc. Experiments with prepared mixtures of lactose and sucrose by this method gave satisfactory results. To determine lactose in sweetened condensed milk, 20 grms. of the sample is dissolved in water, treated with lead acetate, diluted to 100 c.c., and filtered; 50 c.c. of the filtrate is neutralised with hydrochloric acid, using methyl orange as indicator, 2 c.c. of hydrochloric acid (sp. gr. 1.125) is added in excess, the mixture heated in a boiling water-bath for 30 mins., cooled, treated with a few drops of phosphotungstic acid solution, diluted to 100 c.c., and filtered. After standing for 24 hrs., the filtrate is polarised and its reducing power determined and the results obtained calculated into lactose as described. The percentage of lactose found is multiplied by 0.94 to correct for the volume of the lead acetate precipitate.—W. P. S.

*Nitrate and nitrite assimilation [by plants]. Iron and oxygen as necessary agents for the reduction of alkali nitrites by auto-oxidisable compounds [sugars].* Baudisch. See XVI.

*Detection of cane sugar [sucrose] in milk.* Elsdon. See XIXA.

#### PATENTS.

*Cane-sugar; Process of manufacturing —.* N. M. Thomas, Pymble, N.S.W., and C. G. Petree, Bundaberg, Queensland. U.S. Pat. 1,266,882, May 21, 1918. Date of appl., Jan. 14, 1918.

In a process of crushing cane in stages, with intermediate maceration of the bagasse, the rich juice

from the earlier stages of crushing is defeated and the mud produced is mixed with thin raw juice from later crushings; this mixture is defeated and the mud therefrom is distributed over the bagasse at a stage of crushing when the liquid expressed from the bagasse is richer in sugar than the mud itself.—J. H. L.

*Sugar-refining apparatus [centrifuge].* K. K. Kalta, Lahaina Main, Hawaii. U.S. Pat. 1,267,419, May 28, 1918. Date of appl., Oct. 18, 1917.

A CYLINDRICAL centrifugal basket, open at one end, and surrounded by a casing of similar shape, is mounted with its axis horizontal. Mounted on the same axis is a non-rotating sleeve movable into and out of the basket. This sleeve, equal in length to the basket, bears at one end a disc which just fits into the latter, and at the other end a larger disc which abuts against the open end of the casing when the sleeve is at the end of its travel in the basket. It bears also hoppers for charging massenite into the basket, an adjustable scraper for securing a uniform layer of sugar in the latter, and an adjustable comb for breaking up the sugar layer. The sleeve remains inside the basket throughout the centrifuging process, and is withdrawn to discharge the sugar.—J. H. L.

*[Sugar refining:] Process of treating phosphoric acid to be used as a clarifier [in —].* T. Hayashi and U. Emura, Assignors to Kwanto Sanso Kabushiki Kaisha, Ltd., Tokyo, Japan. U.S. Pat. 1,263,080, June 11, 1918. Date of appl., May 24, 1917.

See Eng. Pat. 108,303 of 1917; this J., 1918, 317 A.

### XVIII.—FERMENTATION INDUSTRIES.

*Alcoholic fermentation; General relationship of aldehydes to — and the co-ferment of yeast.* C. Neuberg. Biochem. Zeits., 1918, 88, 145–204.

A GENERAL confirmation of a previous statement of the author, that aldehydes accelerate the alcoholic fermentation of dextrose and mannose. The fact has been demonstrated by experiments with a large number of different aldehydes. With the exception of vanillin, all acted as accelerators. The author also confirms his former statement that a mixture of keto-acids with potassium phosphates acts as a co-ferment. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.

*Beer [wort]; Utilisation of residues from [Brunswick].* O. Reinke. Chem.-Zeit., 1918, 42, 367.

IN the manufacture of strong Brunswick beer, the wort is evaporated until it contains 60% of extract, and then filtered. The residue thus separated still contains about 54% of soluble matter which may be extracted and used over again, whilst the insoluble portion, when dried and roasted, forms a coffee-substitute.—W. P. S.

*Enzymes; Action of —.* VII. M. Jacoby. Biochem. Zeits., 1918, 88, 35–42.

FOR the development of the catalase of the bacterium *Proteus*, alanine and aspartic acid can be employed as well as leucine. In this respect, the catalase differs from urease, for the development of which leucine is essential. The catalase develops satisfactorily in a medium containing the above-mentioned amino-acids and sodium lactate with the necessary inorganic salts. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.



## PATENTS.

*Alcohol; Process for producing — from burnt or roasted starch-bearing materials, etc.* B. Kazmann, Battle Creek, Mich., Assignor to Kellogg Laboratories. U.S. Pat. 1,267,081, May 21, 1918. Date of appl., Mar. 31, 1917. Renewed Mar. 7, 1918.

The roasted or "burnt" material, after being subjected, if desirable, to re-heating or dry distillation and then cooled by means of air, is treated with saccharifying agents, the saccharine liquid obtained is fermented and the alcohol recovered. For example, the material may be extracted with hot water, the undissolved matter subjected to diastatic action, the saccharine liquid separated from the solid residue, the former sterilised by heat, any dextrins present in it saccharified by means of acid, and the product fermented and distilled.—J. H. L.

*Distilling apparatus [for separating alcohol from beer].* A. L. Straus, Assignor to Baltimore Process Co., Baltimore, Md. U.S. Pat. 1,267,709, May 28, 1918. Date of appl., Aug. 31, 1917.

A DETACHABLE condenser consisting of a water-cooled bundle of vertical open-ended tubes is fixed above the mouth of a vessel such as a wort-copper. The vapours condensed in the tubes flow back into the mouth of the copper but are there caught in a collecting pan situated inside the copper just below its mouth. This pan has a jacket for cooling water and it discharges through a tube, also water-jacketed, which passes through the wall of the copper and delivers the condensed alcoholic liquid into apparatus in which further separation of alcohol from water is carried out.—J. H. L.

*Yeast; Process for pressing —.* E. A. Meyer, Bristol. U.S. Pat. 1,268,865, June 11, 1918. Date of appl., Nov. 12, 1917.

SEE Eng. Pat. 113,628; this J., 1918, 255 A.

## XIXA.—FOODS.

*Cane sugar [sucrose] in milk; Detection of —.* G. D. Elsdon, Analyst, 1918, 43, 292–293.

THE following modification of Gayaux's resorcinol test is capable of detecting 0.02% of sucrose in milk: Five drops of a mixture of 15 c.c. of the milk with 1 c.c. of hydrochloric acid and 0.1 gm. of resorcinol is evaporated to dryness on the water-bath in a depression of a white spotting tile. In the presence of sucrose a red coloration is produced.

—C. A. M.

*Butter fat; The glycerides of —.* C. Amberger. Z. Unters. Nahr. Genussm., 1918, 35, 313–381.

A SAMPLE of butter fat examined by the author contained only 2.4% of triolein. By fractional crystallisation of the glycerides of hydrogenated butter fat from various solvents (alcohol, ether, acetone), it was found that the olein had not been converted into tristearin, but into a mixed glyceride. The greater part of the oleic acid in butter fat is therefore present as a mixed glyceride and not as triolein. The butyric and caproic acids are also present as mixed glycerides; tributyrin and tricaproin could not be isolated from the fat. Butyrodiolein, butyropalmito-olein, and oleodipalmitin are present, as is shown by analysis of the glycerides isolated from the alcohol-soluble portion of hydrogenated butter fat. The author has also isolated another glyceride, m.pt. 67.9° C., from butter fat; this glyceride yielded a mixture of fatty acids, m.pt. 55.5° C.—W. P. S.

*Purine bases in foodstuffs; Estimation of —.* T. von Fellenberg. Biochem. Zeits., 1918, 85, 323–336.

TABLES are given showing the content of purine bases in a large number of foodstuffs. The purine bases were precipitated as cuprous compounds by a method described in detail by the author, and the nitrogen was estimated in this precipitate. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.

*Plant products from Colombia.* Bacharach. See XII.

*Determination of lactose in admixture with sucrose and invert sugar.* Grossfeld. See XVII.

*Utilisation of residues from [Brunswick] beer [wort].* Reinke. See XVIII.

## PATENTS.

*Edible oils and fats; Treatment of — and the production of butter substitutes.* C. Townsend, London. Eng. Pat. 117,104, June 2, 1917. (Appl. No. 7944 of 1917.)

WHEY, preferably obtained as a by-product from butter and cheese making, is evaporated, and the residue dried and powdered for storage. For imparting a butter flavour the powder is mixed with water to the consistency of cream, and soured with lactic bacteria at a suitable temperature, or a suitable amount of lactic acid may be mixed with the whey powder. The product is then incorporated in an emulsifier in suitable proportion, and at an appropriate temperature, with oils, fats, or margarine.—C. A. M.

*Grain; Method of deodorising —.* W. Frick, Höchst, Germany, Assignor to General Electric Co. U.S. Pat. 1,267,204, May 21, 1918. Date of appl., Jan. 8, 1915.

GRAIN which has become wet and musty is subjected to the action of a mixture of ozone and steam, and then dried in fresh air.—W. P. S.

*Juice-asepticising process.* H. Coffman, Nazareth, Pa. U.S. Pat. 1,270,698, June 25, 1918. Date of appl., Apr. 16, 1917.

FRUIT juice, immediately after it has been extracted from the fruit, is placed in bottles, carbon dioxide is introduced into the latter to displace the air, the bottles are sealed, heated in water at 145° F. (63° C.), then cooled, and, after the lapse of 48 hrs., again heated at the same temperature for 15 mins.

—W. P. S.

## XIXB.—WATER PURIFICATION; SANITATION.

*Water; Determination of magnesia in —.* M. Monhaupt. Chem.-Zeit., 1918, 42, 338.

A FEW drops of methyl orange solution are added to 100 c.c. of the water, the latter is neutralised in the cold, 5 c.c. of 3% potassium oxalate solution and a known excess of N/10 alkali solution (equal volumes of N/10 sodium hydroxide and carbonate solutions) are added, and the mixture is diluted to 200 c.c. The quantity of sodium hydroxide added must be more than that of the magnesia present in the water. After filtration, 100 c.c. of the filtrate is treated with 5 c.c. of 2% neutral calcium chloride solution and then titrated, without filtration of the calcium oxalate and carbonate, with N/10 acid.

—W. P. S.

*Phosphoric acid; Colorimetric estimation of* — in potable water. P. N. van Eck. Pharm. Weekblad, 1918, 55, 1037—1040.

Stannous chloride can replace hydrazine sulphate in Riegler's colorimetric method of estimating phosphoric acid (this J., 1915, 796), the same blue coloration being produced.—A. J. W.

*Permutite; Basic exchange in* —. V. Rothmund and G. Kornfeld. Z. anorg. Chem., 1918, 103, 129—163.

The basic exchange which takes place between hydrated aluminosilicates, such as the soil zeolites or the artificial product permutite, and neutral salt solutions is a true chemical action and not a process of adsorption. In the case of permutite, equilibrium is rapidly established between the solid and a neutral salt solution, and the composition of the resulting permutite is found to depend upon the relative concentrations of the interchangeable basic radicals in solution. (See also J. Chem. Soc., 1918, II., 315.)—E. H. R.

*Trypaflavin solutions; The keeping properties of* —. A. Abelmann. Pharm. Zeit., 1918, 63, 270.

The yellow dyestuff Trypaflavin (diaminomethyl-acridinium chloride) is employed therapeutically in the form of a 0.1% solution as a bactericide which is comparatively non-toxic to animal tissues and preserves its activity in presence of serum. The 0.1% solution is extremely sensitive to strong sunlight; it turns brown after exposure for one day, and the brown colour slowly increases on further exposure to light. A solution exposed to light and left open to the air for a month was found to be dark brown in colour and to have developed a growth of *Actinomyces*, in spite of the fact that some of the Trypaflavin was still undecomposed. With the addition of sodium carbonate, sulphite, or chloride, decomposition takes place with the formation of a brownish precipitate, but the liquid remains bright yellow on exposure to light. Trypaflavin solutions keep perfectly well in the dark, and decompose only slowly in diffused daylight; 1% solutions are considerably more stable than 0.1% solutions. Addition of acetic or citric acid or of hydrogen peroxide produces the brown coloration even in the dark, and must therefore be avoided. Adequate protection from light by the use of dark-coloured bottles and preservation in the form of more concentrated solutions are recommended.—J. F. B.

#### PATENTS.

*Water-distilling apparatus.* G. B. Furman, East Orange, N.J. U.S. Pat. 1,268,223, June 4, 1918. Date of appl., Aug. 25, 1915.

The apparatus consists of a main tank and two auxiliary tanks, together with a device for maintaining a constant level of water in the tanks. Steam from a boiler is conducted to a coil in the main tank, connected with a second coil in the first auxiliary tank. A coil in the second auxiliary tank serves to condense water vaporised from the main and first auxiliary tanks. The condensed water from the three coils is collected in a common receiver.—W. P. S.

*Antiseptic dressing.* W. R. Walkey, Westminster. Eng. Pat. 117,750, Oct. 19, 1917. (Appl. No. 15,222 of 1917.)

COTTON wool, fibre, or other suitable material is first treated with a solution of paraffin wax or mixture of waxes in ether or other suitable solvent, and, after the solvent has been driven off, with a solution of Flavine or other dye which has an antiseptic action.—L. A. C.

*Purifying fluids [e.g., drinking water]; Process for* —. B. Schwerin, Assignor to Elektro-Osmose A.-G. (Graf Schwerin Ges.), Berlin. U.S. Pat. 1,269,006, June 11, 1918. Date of appl., Dec. 4, 1916.

SEE Ger. Pat. 291,751 of 1915; this J., 1917, 402.

*Evaporating and distilling apparatus [for water].* Eng. Pat. 111,480. See I.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Strophanthus seeds; The sulphuric acid test for* —. P. Bohrisch. Pharm. Zeit., 1918, 63, 318—319.

The sulphuric acid test for strophanthin, by which a green coloration is obtained immediately with the officinal (Kombé) variety of strophanthus seed, has not been very accurately specified. Especially when the word "cross-section" is interpreted to mean the surface of a seed which has been cut in half, the indications of the test are very variable and indefinite. The author has investigated the various modifications of the test which have been proposed, and recommends the following procedure:—Thin cross-sections of the seeds are placed on an object glass and treated with a little ether to remove the fat; they are then covered with one drop of sulphuric acid which has been diluted with one-fourth of its weight of water. The endosperm and at least the outer portions of the embryo leaves should show a deep green coloration, which is best observed under a low-power microscope or a strong magnifying glass.—J. F. B.

*Alkaloids of the pomegranate tree. V. Resolution of pelletierine and methylisopelletierine into their optical antipodes. Elucidation of Tanret's bases.* K. Hess and A. Eichel. Ber., 1918, 51, 741—747.

PELLETIERINE and methylisopelletierine have been resolved by means of active tartaric acids. Some optically-active modifications of these bases were described by Tanret as occurring among the alkaloids of the pomegranate, but the constants quoted by him were much higher than those now observed. The authors feel more than ever justified, therefore, in their statement that no active modifications are present in the root. (See further J. Chem. Soc., 1918, i., 404.)—J. C. W.

*Arecaidine and arceoline; New method for preparing* —. Elucidation of the constitution of guvacine and arceaine. Synthesis of 1-methyltetrahydropyridine-carboxylic acids. I. K. Hess and F. Leibbrandt. Ber., 1918, 51, 806—820.

WHEN the 1-methylpiperidine-carboxylic acids are brominated in alcohol and the products are heated with sodium ethoxide, 1-methyltetrahydropyridine-carboxylic acids are produced. These are closely related to the alkaloids of the areca nut. For example, arecaidine is 1-methyl-1,3,5,6-tetrahydropyridine-3-carboxylic acid, and arceoline is its methyl ester, and these may be obtained by the above method from methyl 1-methylpiperidine-3-carboxylate (N-methylnipecotinate). Two other alkaloids have been found in the areca nut, namely, the acids guvacine and its N-methyl-derivative, arceaine. The authors have synthesised 1-methyl-1,4,5,6-tetrahydropyridine-2-carboxylic acid from 1-methylpiperidine-2-carboxylic acid (N-methylpipercolinic acid), but have found that whilst this closely resembles arceaine it is not identical with it. When guvacine is hydrogenated with the aid of colloidal



platinum, however, it yields piperidine-4-carboxylic acid. Consequently, guvacine is a tetrahydropyridine-4-carboxylic acid, probably the 1.2.5.6 derivative.

In another paper (Ber., 1918, 51, 1004—1006), Hess shows that the methyl ester of guvacine, "guvacoline," is also present in the areca nut.

On the other hand, Freudenberg (*ibid.*, 976—982) submits evidence to the effect that guvacine is simply demethylated arecaine, in which case arecaine and arecaine are identical. (See further J. Chem. Soc., i., 403.)—J. C. W.

*Catalysts; Influence of foreign substances on the activity of —. IV. Experiments with palladium hydrosol in the presence of mercury and mercuric oxide.* C. Paal and W. Hartmann. Ber., 1918, 51, 711—737.

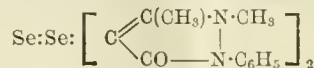
The influence of mercury on the absorptive power of palladium was known even to Dulong and Thénard, and since the advent of colloidal solutions of the platinum metals as catalysts, mercury salts have been classed with the anti-catalysts. In the course of the present work, however, it has been found that palladium hydrosol, protected by sodium protalbate, permanently loses its power of activating hydrogen, and thus stimulating processes of reduction of all kinds, if it is left in contact with mercury, and especially yellow mercuric oxide, but that such a preparation has practically undiminished influence on hydrogen peroxide. A long series of experiments is described which prove that the inactivation of the colloidal palladium is due to its power of sending mercury or its oxide into colloidal solution. This power of "peptisation" is not exerted by sodium protalbate, at any rate on free mercury, although mercuric oxide is taken up if shaken with the salt in an atmosphere of hydrogen.—J. C. W.

*Hydrogenation of aromatic compounds with the help of platinum. III. Hydrogenation with platinum containing oxygen.* R. Willstätter and D. Jaquet. Ber., 1918, 51, 767—779.

Users of platinum in catalytic reductions have begun to recognise that it sometimes matters whether the catalyst is entirely free from occluded oxygen at the outset or has been freely exposed to the air. For example, Hess (Ber., 1913, 46, 3120, 4104) has shown that the rigid exclusion of oxygen is necessary in order to ensure success in the reduction of pyrrole and some of its derivatives. The present paper, however, describes cases of the opposite kind, in which the catalyst must be occasionally activated by exposing it to oxygen. The most interesting example is phthalic anhydride. If this is dissolved in glacial acetic acid and treated with hydrogen in the presence of platinum-black, only a small volume of gas is absorbed, unless the apparatus is opened as occasion requires and the catalyst is agitated in contact with the air. Then the reduction proceeds smoothly by way of phthalide and o-toluic acid, to hexahydrophthalide, hexahydro-o-toluic acid, and hexahydrophthalic acid. Indeed, the influence of phthalic anhydride is such that it even inhibits the hydrogenation of benzene and phthalic acid under ordinary conditions. It appears, therefore, that platinum and oxygenated platinum must be regarded as distinct catalysts. In the case of phthalic anhydride, it is obvious from the above that the oxygen-ring is attacked before the benzene nucleus, but in the case of phthalimide the sole product is hexahydrophthalimide, the side ring remaining intact. The reduction of indole to the utmost limit, represented by octahydroindole ("perhydroindole"), is also described. (See further J. Chem. Soc., 1918, i., 391.)—J. C. W.

*Selenium derivatives of antipyrine; New —.* [Determination of selenium in organic compounds.] F. von Konek and O. Schleifer. Ber., 1918, 51, 842—855.

SELENIUM monochloride reacts with antipyrine as though it had the constitution,  $\text{Se}:\text{SeCl}_2$ , giving diantipyryl diselenide (or "selenoselenide"),



This deposits selenium on boiling with hydrochloric acid, giving diantipyryl selenide (compare Ger. Pat. 299,510). Selenium tetrachloride reacts to form diantipyrylselenium dichloride  $(\text{C}_{11}\text{H}_{11}\text{ON}_2)_2\text{SeCl}_2$ .

The methods for estimating selenium in organic compounds are reviewed, none being found quite satisfactory. The above compounds were analysed by digesting them with pure sulphuric acid (10 c.c. for 0.2 gm.) until a brownish-green colour developed, then diluting to about 200 c.c., saturating with sulphur dioxide, and collecting the precipitated selenium next day. The drawback to this method is the difficulty of determining when to stop the action of the sulphuric acid, as it should not proceed so far that much selenious acid is produced. (See further J. Chem. Soc., 1918, i., 407.)—J. C. W.

*"Perka-glycerol."* [Potassium lactate solution.] P. Panwitz and A. Beythien. Z. Unters. Nahr. Genussm., 1918, 35, 335—337.

A GLYCEROL substitute sold under the name "Perka-glycerol" consists of an aqueous solution containing 65—68% of potassium lactate.—W. P. S.

*Acetone; Detection of —.* I. M. Kolthoff. Pharm. Weekblad, 1918, 55, 1021—1029.

IN Frommer's test for acetone (Berl. Klin. Wochenschr., 1902, 42, 1005), salicylaldehyde can be replaced by vanillin, but not by acetaldehyde or formaldehyde.—A. J. W.

*Keeping properties of Trypaflavin solutions.* Abellmann. See XIXb.

*Detection of glycuronic acid and other acids with similar behaviour by the naphthoresorcinol reaction.* Van der Haar. See XXIII.

*Combined estimation of tyrosine and uric acid in the same solution.* Herzfeld and Klingner. See XXIII.

#### PATENTS.

*Glandular extractive product and process of manufacturing same.* T. B. Aldrich, Assignor to Parke, Davis & Co., Detroit, Mich. U.S. Pat. 1,271,111, July 2, 1918. Date of appl., Mar. 3, 1913.

THE infundibular lobe of the pituitary gland is dehydrated and freed from fat, and then extracted with glycerin. By the addition of a suitable precipitant (alcohol or acetone) to the extract, a substance is precipitated possessing pressor and "oxytocic" properties.—L. A. C.

*Di[hydr]oxydiaminoarsenobenzene; Metallic derivatives of —.* J. Danysz. Sevres, France. U.S. Pat. 1,269,792, June 18, 1918. Date of appl., July 28, 1916.

SEE Eng. Pat. 104,496 of 1916; this J., 1917, 906.

**XXII.—EXPLOSIVES; MATCHES.****PATENT.**

*Explosive materials; Machine for making pellets from pulverulent or granular* —. T. M. Creischere, Glasgow, and R. G. Creischer, Clarkston, Renfrew. Eng. Pat. 117,904, Dec. 15, 1916. (Appl. No. 18,034 of 1916.)

**XXIII.—ANALYSIS.**

*Analysis; New method of qualitative — without the use of hydrogen sulphide.* G. Ahukvist. Z. anorg. Chem., 1918, 103, 221–239.

A new method of qualitative analysis is described in some detail. Although it does not effect a quantitative separation of the different groups and subgroups, it is claimed that all the commoner metals can be readily detected. The principal group reagent is a mixture of potassium hydroxide, potassium carbonate, and hydrogen peroxide. The elements of group I., soluble in this reagent, are As, Sb, Sn, Pb, Zn, Al, and Cr; the elements of group II., insoluble in the reagent, are Ag, Hg, Bi, Fe, Mn, Cu, Cd, Ni, Co, Ca, Sr, Ba, and Mg; group III. comprises the alkali metals and ammonia. Full details for the detection of each metal in the three groups are given. (See also J. Chem. Soc., 1918, II., 333.)—E. H. R.

*Barium; Volumetric determination of — and the solubility of barium chromate in various aqueous solutions.* J. Waddell. Analyst, 1918, 43, 287–289.

Barium chromate, as obtained in the separation of barium from calcium and strontium, may be dissolved in hydrochloric acid and determined volumetrically by a method similar to that used for lead.  $2\text{H}_2\text{CrO}_4 + 12\text{HCl} + 6\text{KI} = 6\text{KCl} + 2\text{CrCl}_3 + 3\text{I}_2 + 3\text{H}_2\text{O}$ . The solution of the barium salt is treated with 30% ammonium acetate solution (10 c.c. to 50 c.c. of a 0.3% solution of barium chloride), and the barium precipitated by adding 15 c.c. of 10% ammonium bichromate to the boiling liquid. The precipitate is washed, dissolved in a mixture of 25 c.c. of strong hydrochloric acid and 75 c.c. of water, and the solution made up to 200 c.c., treated with 6 grms. of potassium iodide, and allowed to stand for 10 mins. The liberated iodine is then titrated with standard thiosulphate solution. This method was used to determine the solubility of barium chromate in water and various aqueous solutions, the following results being obtained:—Water, 0.78; 1% ammonium acetate solution, 2.80; 10% ammonium acetate, 3.58; 20%, 5.07; 10% ammonium nitrate, 4.89; 10% sodium chloride, 2.39; and 1% acetic acid, 11.04 parts per 100,000 of washing liquid. Water is thus the best medium for washing precipitated barium chromate.

—C. A. M.

*Manganese; Microchemical reaction for —.* J. B. Menke. Chem. Weekblad, 1918, 15, 868–869.

MANGANESE sulphate gives with a drop of ammonia containing cyanuric acid a precipitate of manganous hydroxide. After the lapse of a few minutes, or immediately on boiling, colourless unstable needles of high polarising power separate from the mixture.

—A. J. W.

*Bismuth; Electro-analysis of — without platinum electrodes.* P. Poch. Anal. Fis. Quim., 1918, 16, 520–533.

The author has devised a method of estimating bismuth electrolytically. It can be carried out in solution in nitric, tartaric, sulphuric or acetic acid, and also in ammoniacal solution. The experiments were made with platinum electrodes, with a platinum anode and a copper cathode, with a platinum

cathode and an iron or graphite anode, and with a copper cathode and an iron or graphite anode.

—A. J. W.

*Molybdenum; Determination of —.* K. Wolf. Z. angew. Chem., 1918, 31, 140.

The conversion of a precipitate of molybdenum sulphide into  $\text{MoO}_3$  by ignition is not quantitative if the temperature rises above  $450^\circ\text{C}$ ., as sublimation then occurs to an appreciable extent. A temperature of  $400^\circ\text{C}$ . suffices and it is most convenient and certain to ignite the precipitate in an electrically heated furnace at a temperature of  $425^\circ\text{C}$ .

—H. J. H.

*Carbon monoxide; Behaviour and detection of the smallest quantities of — in the "chlorate" pipette.* K. A. Hofmann and H. Schibsted. Ber., 1918, 51, 837–842.

An absorption-pipette for hydrogen has previously been described (this J., 1916, 1002, 1003), which contains porous tubes impregnated with platinum and a little palladium, partially immersed in a solution of sodium chlorate activated by osmium tetroxide. The rate at which hydrogen is oxidised in this pipette is greatly lessened by traces of carbon monoxide, but inasmuch as the active surface of the platinumed tubes becomes smaller as the absorption proceeds and the liquid rises, a direct observation of the rate of absorption does not give a true idea of the influence of carbon monoxide. As the part of the pipette in which the tubes are packed is cylindrical, the active area is proportional to the volume of gas unabsorbed, so it is possible to arrive at a "relative rate of absorption" at any period. With pure hydrogen, this rate is constant, or slowly rises, until half the gas is absorbed, whereas traces of carbon monoxide cause very rapid falls in this rate. If the pipette is standardised, therefore, and a curve is plotted connecting the relative rates of absorption at "half volume" with various proportions of carbon monoxide, the phenomenon can be adapted to analytical purposes.—J. C. W.

*Distilling in [superheated] steam under reduced pressure; Laboratory apparatus for —.* C. Harries and R. Haarmann. Ber., 1918, 51, 788–790.

In the apparatus, which is fully described with diagrams, steam at 3–4 atmos. pressure is admitted through a throttle-valve into a superheating chamber kept at about  $300^\circ\text{C}$ ., and then into the substance in the evacuated distillation flask. The vapours from the distillation flask pass through a wide tube partly filled with short pieces of glass tubing in order to prevent foaming and spraying over of liquid into the condenser.—J. C. W.

*Glycerol; The copper method for estimating —.* F. L. Weiss. Chem. Weekblad, 1918, 15, 862–868.

THERE is no definite chemical compound of copper and glycerol, the proportion of copper in the glycerol-copper complex being determined by four factors, *viz.*, the proportion of glycerol in the solution, the copper content increasing with the glycerol; the temperature, the proportion of copper falling with rise of temperature, especially in very dilute solutions; the degree of dilution of the liquid, and the concentration of the free alkali.—A. J. W.

*d-Glycuronic acid and other acids with similar behaviour; Detection of — by the naphthoresorcinol reaction.* A. W. Van der Haar. Biochem. Zeits., 1918, 88, 205–212.

The substance is first hydrolysed with 5% sulphuric acid, and the solution neutralised with



barium hydroxide, filtered and concentrated. Lead acetate is then added, and after filtration basic lead acetate. The glycuronate is contained in the precipitate thus produced, which is heated with 10 c.c. of 10% hydrochloric acid and 100 mgrms. of naphthorescinol for 1 minute. After cooling to 50° C. the solution is shaken with benzene; if glycuronic acid is present a violet colour is produced in the benzene, which can be characterised by a band in D in the spectrum. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.

*Tyrosine and uric acid: Combined estimation of — in the same solution.* E. Herzfeld and R. Klinger. *Biochem. Zeits.*, 1918, 88, 283—285.

THE two substances are estimated together by Folin's phenol reagent, the tyrosine giving a colour twice as strong as the corresponding amount of uric acid. The latter is then destroyed by hydrogen peroxide and sodium hydroxide, and, under specified conditions, the phenol reagent is added again, and the tyrosine alone estimated. (See also J. Chem. Soc., Oct., 1918.)—S. B. S.

*Determination of acid in woollen fabric.* Woodmansey. *See V.*

*Application of rapidly rotating metallic reductors in the determination of vanadic acid.* Gooch and Scott. *See VII*

*Estimation of oxygen in working cyanide solutions.* White. *See X.*

*Determination of lead in ores, etc.* Stahl. *See X.*

*Pure antimony.* Groschuff. *See X.*

*Determination of turpentine and detection of adulterants.* Tausz. *See XIII.*

*Preparation of samples for leather analysis.* Veitch and Frey. *See XV.*

*Determination of fat in leather.* Levi and Orthmann. *See XV.*

*Unfermentable sugars (glucose) of cane molasses, their preparation, properties, and determination.* Muller. *See XVII.*

*Estimation of dextrose by means of hypiodite.* Willstätter and Schudel. *See XVII.*

*Determination of lactose in admixture with sucrose and invert sugar.* Grossfeld. *See XVII.*

*Detection of cane sugar [sucrose] in milk.* Elsdon. *See XIXa.*

*Estimation of purine bases in foodstuffs.* Von Fellenberg. *See XIXa.*

*Determination of magnesia in water.* Monhaupt. *See XIXb.*

*Colorimetric determination of phosphoric acid in potable water.* Van Eck. *See XIXb.*

*The sulphuric acid test for strophanthus seeds.* Bohrisch. *See XX.*

*Detection of acetone.* Kalthoff. *See XX.*

#### PATENT.

*Viscosity measuring instrument.* H. R. Edgecomb, Wilksburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,270,800, July 2, 1918. Date of appl., Aug. 11, 1913

AN apparatus for measuring the viscosity of heavy oil or grease comprises a vertical jacketed cylinder containing the material to be tested, provided with

a piston supported by an external adjustable spring above it. The cylinder is moved axially at a uniform rate by means of a hydraulic cylinder below it, and the time of its movement is recorded. A record sheet is attached to the cylinder, and the vertical movement of the piston, as modified by the viscosity of the fluid and the resistance of the spring, is transformed by a bell crank lever into a horizontal movement and recorded on the moving sheet.—W. F. F.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Adams. Furnaces. 14,977. Sep. 14.  
Baily and Cope. 14,400. *See X.*  
Bawtree and Hadfield. Apparatus for applying thick coating of fluid, etc., material to the interior surfaces of cylinders. 14,579. Sep. 7.  
Beale and Dunkerley. Refrigeration. 14,875. Sep. 12.  
Candlot. Kilns or shaft furnaces. 14,468. Sep. 5. (Fr., Mar. 23, 1917.)  
Ely. Furnaces. 14,647. Sep. 9.  
Hutchins. Means for treating gases with liquids. 14,933. Sep. 13.  
McIntyre, and Milne and Son. Means for drying, concentrating, and filming solids, liquids, semi-liquids, etc. 14,230. Sep. 2.  
Nederlandsche-Maatschappij. 14,447. *See XIX.*  
Roberts. Kilns or furnaces. 14,791. Sep. 11.  
Saunders and Walsh. 14,422. *See II.*  
Stein et Cie. Oscillating furnaces. 14,248. Sep. 2. (Fr., Oct. 24, 1917.)  
Stein et Cie. Continuous re-heating furnaces. 14,796. Sep. 11. (Fr., Nov. 5, 1917.)  
Stewart. Filtration apparatus. 14,367. Sep. 4.  
Thomson. Colour-heat insulation. 15,001. Sep. 14.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 12,620 (1917). Savage, and Brinjes and Goodwin. Edge-runners, pan and like mills. (118,465.) Sep. 11.  
13,413 (1917). Rimmer. *See XIX.*  
18,620 (1917). Norton. Grinding-machines. (118,537.) Sep. 11.  
4800 (1918). Moore. Apparatus for agitating liquids, powders, granular substances, etc. (118,567.) Sep. 11.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

- Bailey. Method of treating peat, etc., by superheated steam. 14,204. Sep. 2.  
Bargate and Desmond. Desulphurising gaseous distillates. 14,955. Sep. 14.  
Chance. Washing coal and concentrating ores and minerals. 14,645. Sep. 9. (U.S., Aug. 27, 1917.)  
Clark. Method of treating oils. 14,658. Sep. 9.  
Hart. Suction-gas scrubbers and coolers. 14,349. Sep. 4. (Australia, Aug. 28, 1917.)  
Hart. Suction-gas plants. 14,628. Sep. 9. (Australia, Nov. 13, 1917.)  
Hes. 14,663. *See X.*  
Jackson, and Woodall and Duckham. Manufacture of gas in vertical retorts. 14,712. Sep. 10.  
Jackson and Spieer. Liquid fuel for internal-combustion engines. 14,830. Sep. 12.

Mantle. Treating waste, etc., lubricating oils to eliminate sulphur and other impurities. 14,673. Sep. 10.

Nitrogen Products and Carbide Co., and Perkin. Destructive distillation of carbonaceous materials. 14,519. Sep. 6.

Saunders and Walsh. Purification of gases. 14,422. Sep. 5.

Soc. Franco-Belge de Fours à Coke. Flue-bases or plectroits for coking ovens. 14,725. Sep. 10. (Fr., Oct. 22, 1917.)

Stafford. Destructive distillation of wood. 14,708. Sep. 10. (U.S., Sep. 10, 1917.)

Stewart. Explosive substances for internal-combustion engines, etc. 14,283. Sep. 3.

White (Sinclair Refining Co.). Cracking hydrocarbons. 14,865. Sep. 12.

White. Distillation of solid carbonaceous material. 14,773. Sep. 11.

Wilton. Gas plant, coking plant, etc. 14,408. Sep. 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,866 (1917). Ore. Coke ovens. (118,470.) Sep. 11.

13,049 (1917). Dowson and Mason Gas Plant Co., and Webb. Open-hearth gas-producers, etc. (118,689.) Sep. 18.

15,923 (1917). Marbais and Degulde. Extraction and fractional distillation of oils from coal gas. (118,730.) Sep. 18.

17,054 (1917). Hillen. Distillation of coal and other materials for manufacture of motor gas. (118,522.) Sep. 11.

717 (1918). Merz and McLellan, Michie, and Weeks. Solid fuel for large-scale power production. (118,777.) Sep. 18.

4504 (1918). Dempster and Sons, and Brooke. Horizontal and inclined retort settings. (118,804.) Sep. 18.

### III.—TAR AND TAR PRODUCTS.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,621 (1917). Nicorestl. Solid preparations containing cresols and mixtures of cresols, and manufacture thereof by means of salts of fatty acids. (118,667.) Sep. 18.

15,421 (1917). Bull. Sulphonation of aromatic hydrocarbons. (118,727.) Sep. 18.

15,923 (1917). Marbais and Deguide. *See* II.

### IV.—COLOURING MATTERS AND DYES.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,348 (1917). Imray (Soc. Chem. Ind. in Basle). Manufacture of azo dyestuffs capable of being chromed. (118,448.) Sep. 11.

16,246 (1917). Allsebrook. Manufacture of organic colouring matter and of pigments therefrom. (118,735.) Sep. 18.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Ashton. Manufacture of paper, papier mâché, etc. 14,961. Sep. 14.

Marks (Cedaroid Co.). Paper or paper board and its manufacture. 14,784. Sep. 11.

#### COMPLETE SPECIFICATIONS ACCEPTED.

8206 (1917). Lippitt. Obtaining fibres from fibrous material or vegetable matter. (113,084.) Sep. 18.

16,038 (1917). Doverdale. Paper-making machines. (118,511.) Sep. 11.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Barnes, Wrigley, and Spence and Sons. Dyeing, staining, and mordanting. 14,740. Sep. 11.

Farrell. Dye dyes. 14,383. Sep. 4.

#### COMPLETE SPECIFICATION ACCEPTED.

16,508 (1917). Dudley. Dyeing machines. (111,843.) Sep. 11.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Bargate and Desmond. 14,955. *See* II.

Craig. Obtaining potash compounds from blast furnace, etc., slag. 14,412. Sep. 5.

Dawson. Manufacture of nitric acid. 14,359. Sep. 4.

Dawson. Production of sodammonium sulphate from nitre cake, etc. 14,756. Sep. 11.

Ffabetl. Manufacture of platinised asbestos. 14,325. Sep. 3. (Italy, Nov. 5, 1917.)

Marks (Du Pont de Nemours and Co.). Concentrating nitric acid. 14,522. Sep. 6.

Rabinovitch. Extraction of magnesium compounds. 14,556. Sep. 7.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,594 (1917). Maconochie and de Ros. *See* X.

12,595 (1917). Maconochie and de Ros. Production of zinc oxide. (118,665.) Sep. 18.

14,425 (1917). Hutchins. Apparatus for drying salt. (118,712.) Sep. 18.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

British Thomson-Houston Co., and Gray. Devices for discharging predetermined quantities of glass from glass furnaces. 14,862. Sep. 12.

Kraus. Manufacture of refractory material. 14,542. Sep. 6.

Winwood. Pot or receptacle for heating or melting glass. 14,424. Sep. 5.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,845 and 13,846 (1917). Crowell. Dental cements. (118,701 and 118,702.) Sep. 18.

8472 (1918). McCauley. Means for discharging molten metal in the manufacture of glass articles. (118,581.) Sep. 11.

9030 (1918). McCauley. Conveyance of molten glass. (118,585.) Sep. 11.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Swingler. Mixing-machine for concrete, etc. 14,691. Sep. 10.

Thermalloy, Ltd., and Vautin. Sulphur cements. 14,847. Sep. 12.

Waine. Manufacture of bricks and artificial stones. 14,869. Sep. 12.

Wingvist. Rotating kilns for burning cement, etc. 14,625. Sep. 9. (Sweden, June 12.)

#### COMPLETE SPECIFICATION ACCEPTED.

216 (1918). Ardagh. Manufacture of fire-resisting plastic material and its application to constructional purposes. (118,768.) Sep. 18.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Alpha Products Co. Manufacture of metallic cerium, etc. 14,238. Sep. 2. (U.S., Sep. 1, 1917.)

Bally and Cope. Melting-furnaces. 14,400. Sep. 4. (U.S., Sep. 4, 1917.)

Barfield and Peakman. Determination of decalescence in treatment of steel. 14,797. Sep. 11.

Besford and Day. Soldering aluminium, duralumin, etc. 14,979 and 14,980. Sep. 14.

Bolitho. Roasting and refining tin. 14,233. Sep. 2.

Chance. 14,645. *See* II.

Craig. 14,442. *See* VII.

Dehn (National Lead Co.). Smelting-furnace. 14,246. Sep. 2.



Frankignoul. Blast furnaces. 14,694. Sep. 10.  
Gill (Fansteel Products Co.). Cleaning and circulating system for reducing tungsten trioxide. 14,614. Sep. 9.

Giton. Furnace for re-fusing and refining metal. 14,621. Sep. 9.

Hes. Metallic alloys for explosive, incendiary, or illuminating purposes. 14,663. Sep. 10.

Neill. Casting steel ingots. 14,481. Sep. 6.

Paetschke. Alloys and method of purifying same. 14,575. Sep. 7.

Stein et Cie. 14,796. *See I.*

#### COMPLETE SPECIFICATIONS ACCEPTED.

8366 (1917). Greaves and Etchells. Converting iron ores directly into steel. (118,647.) Sep. 18.

12,594 (1917). Maconochie and de Ros. Oxidation of tin. (118,664.) Sep. 18.

16,072 (1917) and 1905 (1918). Wild and Barfield. Indicating condition of steel, iron, etc., during heat treatment. (118,732.) Sep. 18.

17,367 (1917). Gaunt, Brookfield, and Tylor and Sons. Smelting furnaces and the like. (118,750.) Sep. 18.

6271 (1918). Ramen. Mechanical roasting-furnaces. (118,812.) Sep. 18.

7667 (1918). Clevenger. Electrolytic production of zinc. (115,847.) Sep. 18.

8289 (1918). Milliken. Alloys and process of making same. (118,825.) Sep. 18.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Angold and Rilling. Electro-magnetic control of electric arcs. 14,965. Sep. 14.

Bibby. Electric-arc furnaces. 14,235. Sep. 2.

Bradley. Electrical treatment of gases. 14,943, 14,944, and 14,947. Sep. 13. (U.S., June 21, 1916.)

Fuller Accumulator Co., and Jones. Electrodes for primary and secondary batteries. 14,859. Sep. 12.

Kayser and Macdonald. Means for improving conductivity at joints of electrodes. 14,372. Sep. 4.

Pearson and White. Electric accumulators. 14,530. Sep. 6.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9489 (1917). Berry. Treatment of magnetic material for use in static transformers and other electrical apparatus. (118,425.) Sep. 11.

12,566 (1917). Worsnop. Electric accumulators (118,464.) Sep. 11.

13,172 (1917). Hamilton, Blackwell, and Turner. Electrodes for electric furnaces, etc. (118,690.) Sep. 18.

7667 (1918). Clevenger. *See X.*

### XII.—FATS; OILS; WAXES.

#### APPLICATIONS.

Clark. 14,658. *See II.*

Naaml. Vennootsch. Jurgens' Vereen. Fabr. 14,329. *See XIX.*

Phillips. Extraction of oil from cottonseed. 14,537. Sep. 6.

#### COMPLETE SPECIFICATION ACCEPTED.

12,472 (1917). MacGregor, and Scott and Son. Extraction of oils, wax, rosins, etc., from bone charcoal or other substances. (118,461.) Sep. 11.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATION.

Dorel. Manufacture of gelatinous compositions for reproduction of line documents. 14,262. Sep. 2. (Fr., Dec. 7, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,472 (1917). MacGregor and others. *See XII.*  
16,246 (1917). Allsbrook. *See IV.*

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

Turner. 14,435. *See XV.*

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Dorel. 14,262. *See XIII.*

Posnett. Treatment of hides, skins, etc. 14,817. Sep. 12.

Turner. Utilising waste leather and rubber. 14,435. Sep. 5.

### XVII.—SUGARS; STARCHES; GUNS.

#### COMPLETE SPECIFICATION ACCEPTED.

9509 (1917). Hopkinson. Manufacture of glucose (118,651.) Sep. 18.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Bodman. Food product. 14,827. Sep. 12.

Campbell and Glover. Vegetable food products. 14,260. Sep. 2.

Kestner. Edible product obtained from beet. 14,969. Sep. 14.

Kestner. Food product containing cacao. 14,970. Sep. 14.

Lensvelt. Manufacture of cattle cake from yellow mustard seed. 14,940. Sep. 13. (Holland, Dec. 21, 1917.)

Marquis. Food compound. 14,353. Sep. 4.

Naaml. Vennootsch. Jurgens' Vereen. Fabr. Process for purifying casein simultaneously with obtaining fat and lecithin, and obtaining emulsifying agent. 14,329. Sep. 3. (Holland, Apr. 23.)

Nederl. Maatsch. tot voortzetting der Zaken van der Linde & Teves. Drying agricultural, etc., products. 14,447. Sep. 5. (Holland, May 29.)

Powerie. Roller driers for treating foodstuffs, etc. 14,595. Sep. 9.

Sutherland. Treating mill or flour and milling-products. 14,863. Sep. 12. (Holland, Jan. 2.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

10,897 (1917). Remington. Preparatory treatment of wheat for milling purposes. (118,431.) Sep. 11.

12,624 (1917). Edsen, Tucker, and Minerals Separation, Ltd. Softening water. (118,668.) Sep. 18.

12,732 (1917). Fairweather (Pacific Evaporator Co.). Evaporated vegetable products. (118,466.) Sep. 11.

12,733 (1917). Fairweather (Pacific Evaporator Co.). Evaporated fruit. (118,673.) Sep. 18.

12,734 (1917). Fairweather (Pacific Evaporator Co.). Evaporated apples. (118,671.) Sep. 18.

12,971 (1917). Espallat de la Mota. Processes of preserving eggs. (118,684.) Sep. 18.

13,413 (1917). Rimmer. Apparatus for distilling and sterilising liquids, especially water. (118,482.) Sep. 11.

18,235 (1917). Barnabe and Bozzelli. Fodder or alimentary mixture. (118,757.) Sep. 18.

18,279 (1917). Melhuish. Manufacture of soya bean milk. (118,555.) Sep. 11.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATION.

Comp. des Prod. Chim. d'Alais et de la Camargne. Continuous preparation of monochloroacetic acid from trichloroethylene. 14,513. Sep. 6. (Fr., May 4.)

#### COMPLETE SPECIFICATION ACCEPTED.

14,034 (1917). Marks (Du Pont de Nemours and Co.). Process for purifying camphor. (118,489.) Sep. 11.

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATIONS.

Hes. 14,663. *See X.*

James. Matches. 14,461. Sep. 5.

Stewart. 14,283. *See II.*

## I.—GENERAL; PLANT; MACHINERY.

*Effect of gravitation upon the drying of ceramic ware.* Washburn. See VIII.

## PATENTS.

*Separating dust or powder from gases or air containing it; Apparatus for —.* P. O. Rowlands, Liverpool. Eng. Pat. 117,957, Aug. 13, 1917. (Appl. No. 11,593 of 1917.)

SEVERAL sets of pervious vertical tubes provided with crossheads near the top are connected by flexible portions above the crossheads to an upper horizontal trunk. The tubes are arranged within a casing which is provided with doors on each side to allow access to the tubes, the lower ends of which are connected to a trunk provided with a screw conveyor for removing deposited dust. The tubes are provided at their upper ends with spring valves and with shaking tappets which are operated by a series of cams and levers.—W. H. C.

*Air-washer.* D. T. Hastings, Assignor to Holley Bros. Co., Detroit, Mich. U.S. Pat. 1,272,434, July 16, 1918. Date of appl., Mar. 8, 1917.

The washing liquid is contained in a receptacle having an air inlet and outlet. By means of a float the air inlet is maintained in constant relation to the level of the washing liquid and is closed when the liquid has fallen to a predetermined level. Means are provided for separating liquid from the air in the space above the liquid level, and fresh liquid is introduced through a "filler," so placed that liquid is limited to a level below the separating device. The "filler" has a removable closure, and may be used as an auxiliary air inlet when the normal air inlet is cut off.—J. H. P.

*Dust collector.* F. A. Marascio, Phillipsburg, N.J. U.S. Pat. 1,272,489, July 16, 1918. Date of appl., Feb. 14, 1918.

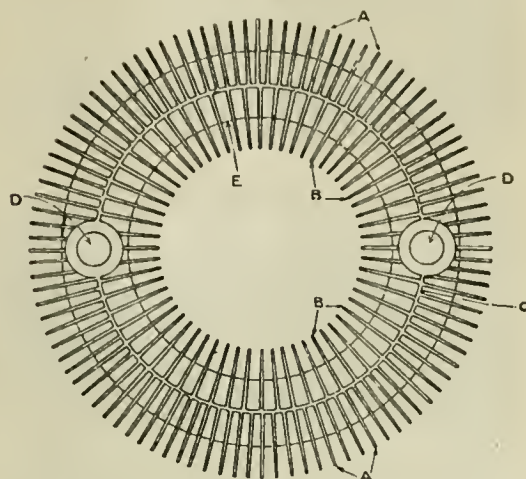
The apparatus comprises a casing with a door and a water pan which can be removed through the door. The dust-laden air is conducted through a hollow shaft into a perforated drum which rotates in the water in the pan.—J. H. P.

*Gases; Art of separating materials from —.* E. R. Knowles, Brooklyn, N.Y. U.S. Pat. 1,273,791, July 23, 1918. Date of appl., Feb. 14, 1917.

A MIXTURE of gas and heavier material is passed through a stationary chamber containing a centrifugal fan. Each fan blade consists of a solid or perforated radial portion, and a perforated circumferential portion extending towards but not reaching the next blade.—W. F. F.

*Transmission or exchange of heat; Apparatus for the —.* A. H. Bruce and C. Baynes, London. Eng. Pat. 118,375, Nov. 28, 1917. (Appl. No. 17,609 of 1917.)

An apparatus for the transmission of heat between fluids, e.g., steam, water, or air, comprises an endless circular pipe, B, with inlet and outlet connections, D, at opposite ends of a diameter. The pipe is provided with a short external cylindrical flange, C, co-axial with the axis of the pipe ring, and projecting on both sides of the pipe. Semicircular ribs, A, and B, are provided on both sides of the flange. The provision of the flange, C, facilitates



the casting of the apparatus in one piece with a larger number of ribs, A, than the number of ribs, B, so as to equalise the spacing as shown.

—W. F. F.

*Crusher.* W. K. Liggett, Assignor to The Jeffrey Manufacturing Co., Columbus, Ohio. U.S. Pat. 1,268,479, June 4, 1918. Date of appl., July 28, 1914.

The material is fed from a hopper to the upper part of the space between a rotary crushing roll and a breaker plate with a concave working surface mounted at one side of the roll. The plate is pivoted at its lower end and held yieldingly against the material being crushed. The upper end of the plate is connected with gearing by which the plate can be moved downwards, inwards, outwards, and upwards, and has a projection which moves the material towards the crushing roll.

*Tube-mills; Feeder for —.* H. W. Hardinge, New York. U.S. Pat. 1,272,801, July 16, 1918. Date of appl., May 11, 1916.

The material is fed into the axial inlet opening of a tube-mill from a rotary conical casing having an internal helico-spiral trough conveyor.—W. H. C.

*Mixing apparatus; Chemical —.* J. R. Milson, Watertown, Mass., Assignor to J. J. Burns, Boston, Mass. U.S. Pat. 1,267,102, May 21, 1918. Date of appl., Nov. 13, 1916.

The apparatus comprises a number of containers connected with a mixing chamber by pipes provided with weighted valves so that, when the containers are heated, a predetermined pressure may be generated in each. Gases or vapours leave the containers under pressure and pass to the mixing chamber, which is provided with an agitator, and thence flow to a water-cooled reservoir or condenser.—J. H. P.

*Mixer and dryer.* W. B. Ruggles, Bayonne, N.J. U.S. Pat. 1,269,120, June 11, 1918. Date of appl., Mar. 9, 1911.

MATERIAL to be mixed and dried is passed through a rotary mixer, and thence through a passage to an adjacent rotary dryer through which hot gas is also passed. The gas leaving the dryer is passed through an auxiliary heater to preheat the material. The passage between the mixer and dryer is controlled by a plate which may be operated during rotation of the apparatus to open or close the passage.—W. F. F.



*Steam; Apparatus for the generation of* —. P. E. Tucker, Bremerton, Wash. U.S. Pat. 1,270,990, July 2, 1918. Date of appl., July 25, 1917.

ALTERNATE charges of combustible gas and small quantities of water are delivered to a combustion chamber in such a manner that the burning gas converts the preheated water into steam and forces the latter into an adjacent reservoir.—A. B. S.

*Filter.* W. H. Duryea, Tiffin, Ohio. U.S. Pat. 1,271,681, July 9, 1918. Date of appl., Aug. 17, 1916.

A CYLINDRICAL porous filter chamber having one flat and one hemispherical end is contained within an outer casing of similar shape. The deposit which forms on the surface of the filter chamber is washed off by a multiple jet of liquid introduced through the centre of the curved end of the outer chamber and is discharged through a special outlet provided for the purpose.—W. H. C.

*Filter.* F. K. Atkins, New York, and E. F. Atkins, Arlington, N.J. U.S. Pat. 1,273,127, July 23, 1918. Date of appl., Sep. 18, 1917.

THE filter plates are provided with central openings and are arranged on a common axis. The solid material deposited on the plates is removed by a series of cutters mounted on a central shaft extending through the openings. The cutters are normally arranged in the spaces between the plates, in alignment with the central openings, and may be moved over the active surfaces of the plates by rotation of the shaft.—W. F. F.

*Ammonia condenser for ice machines.* W. H. Manns, Assignor to Frick Co., Waynesboro, Pa. U.S. Pat. 1,272,286, July 9, 1918. Date of appl., July 31, 1914.

THE condenser comprises a series of coils connected at the inlet end with an ammonia gas header and at the outlet end with a liquid ammonia header, both headers being arranged on a level with the top of the coil. A valve interposed near the gas inlet regulates the flow of liquid ammonia absorbed by the liquid in the inlet pipe outside of the valve.

—J. H. P.

*Gas-absorption apparatus.* H. B. Slater, Los Angeles, Cal. U.S. Pat. 1,272,321, July 9, 1918. Date of appl., Feb. 19, 1916.

THE apparatus comprises a casing having an inlet at the top and an outlet at the bottom for the absorbing liquid. A trough receives the liquid from the inlet and distributes it in a descending sheet, which is caught by a deflector and caused to form a second descending sheet. The outlet for the liquid is provided with aspirating means, and the gas to be absorbed is aspirated through the descending sheets of liquid.—J. H. P.

*Fume condenser.* G. C. Richards, Berkeley, Cal. U.S. Pat. 1,272,528, July 16, 1918. Date of appl., Apr. 30, 1917.

THE condenser comprises a long box-like structure with a number of vertical baffle plates. A number of frames covered with fabric are placed angularly between the baffle-plates and form a covering for the box. The fumes to be condensed are forced into the box and water is sprayed on the frames and into the box, and is kept at a constant level in the box.—J. H. P.

*Conveyor for furnaces; Material* —. T. B. Cram, Assignor to The Railway Materials Co., Chicago, Ill. U.S. Pat. 1,272,918, July 16, 1918. Date of appl., July 26, 1915.

THE hearth of the furnace has a number of narrow longitudinal slots communicating with relatively large chambers below which open at both ends to the atmosphere. Refractory elements are mounted on supports in these chambers and project into the slots, and are caused to move in an arcuate path upwards and forwards in the slots by means of eccentrics mounted at both ends of the hearth. Heating gases are supplied to the furnace, and the supports are cooled by air circulated through the chambers below the slots.—J. H. P.

*Registering apparatus chiefly for use in registering the amount of feed of granular and like substances.* J. E. Lea, Manchester. Eng. Pat. 118,333, Aug. 27, 1917. (Appl. No. 12,258 of 1917.)

*Separation of suspended particles from gases; Process and apparatus for* —. H. A. Burns, London. Eng. Pat. 118,334, Aug. 27, 1917. (Appl. No. 12,281 of 1917.)

SEE U.S. Pat. 1,250,088 of 1917; this J., 1918, 114 A.

*Cooling, humidifying, and filtering air or the like; Apparatus for* —. H. F. Brown, Sheffield. U.S. Pat. 1,271,271, July 2, 1918. Date of appl., Jan. 19, 1918.

SEE Eng. Pat. 112,653 of 1917; this J., 1918, 114 A.

*Drying materials in sacks and the like; Method and apparatus for* —. H. P. Dinesen, Herlev, Denmark. U.S. Pat. 1,271,293, July 2, 1918. Date of appl., Jan. 25, 1916.

SEE Eng. Pat. 100,697 of 1916; this J., 1916, 915.

*Crystallisation; Apparatus for effecting continuous* — from solutions. E. Hansen, Rjukan, Assignor to Norsk Hydro-Elektrisk Kvaestof-aktieselskab, Christiania, Norway. U.S. Pat. 1,273,058, July 16, 1918. Date of appl., Sep. 3, 1915.

SEE Fr. Pat. 479,668 of 1915; this J., 1916, 1145.

*Emulsifying apparatus.* U.S. Pat. 1,269,399. See XIXA.

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal dust and air mixtures; Laboratory determination of explosibility of* —. J. K. Clement and J. N. Lawrence. U.S. Bureau of Mines, Tech. Paper 141, 1917. 35 pages.

A LABORATORY method is described by which it is possible to determine whether dusts are capable of giving rise to explosions in the mine, and the percentage of pulverised shale that must be added to the dust to render it non-explosible. The method has been standardised against certain coal dusts of which the "ignition limit" and "propagation limit" mixtures with shale dust have been previously determined by large-scale experiments in the mine. The term "ignition limit" is used to designate the highest percentage of shale dust with which a given coal dust can be mixed and yet be ignited by the flame of a blown-out shot of black powder and give rise to an explosion. The "propagation limit" of a dust is the highest percentage of shale dust with which it can be mixed and still be capable

of propagating an explosion set up in a cloud of more inflammable dust or in an explosive gas mixture. The apparatus consisted of an explosion globe (capacity 1400 c.c.) with an electrically heated lighting coil at the centre, the temperature of the ignition coil being 1200° C. The dust to be tested was puffed up on to the coil from an injector connected to an oxygen cylinder. Standard quantities of 1 gm. or 3 grms. of dust were used, all dusts being pulverised to pass through a 200-mesh sieve. The pressure produced in the globe by the combustion of the dust was registered by a Crosby indicator, and was taken as a measure of the inflammability of the dust in question. Suitable arrangements were also made for determining the explosibility of dusts when the explosion globe contained a mixture of air plus a small percentage of natural gas. The pressures developed by different dusts varied from a few tenths of a pound to 20 pounds. By noting the pressures developed in the apparatus by 1 gm. or 3 grms. of the ignition or propagation limit mixtures of a standard coal (as determined in the experimental mine), and assuming that the similar limit mixtures of any other coal would develop the same respective pressures, it was possible to predict the explosibility of a coal, by determining the pressures developed by a series of its mixtures with shale dust under standard conditions. A small percentage of gas in the atmosphere rendered necessary the addition of more shale dust to make the coal dust mixture non-explosible. The relative inflammabilities of 600 coals, including road and rib dusts and shale dust mixtures, have been determined by the method. No definite relation was found between inflammability and chemical composition. The effect of size of particles of dust on explosibility is touched upon. A series of curves is given showing graphically the relation between inflammability and percentage of shale dust in the mixtures for several typical coals.—T. F. E. R.

*Incombustible matter in coal and rock-dust mixtures in mines; Quick determination of* — A. C. Fieldner, W. A. Selvig, and F. D. Osgood. U.S. Bureau of Mines, Tech. Paper 144, 1918. 36 pages.

A VOLUMETER method is described depending on the relative specific gravities of coal and rock dusts. It is rapid, and gives results of reasonable accuracy if calibrated for the prevailing constituents of the dust. The true sp. gr. of coal rarely varies beyond the limits of 1.25 and 1.35, while the sp. gr. of incombustible matter found or applied in mines varies from 1.9 (clay) to 5.0 (pyrite). A definite weight (15 or 20 grms.) of dust is introduced into the flask of a volumeter (essentially a specific gravity bottle having an extended calibrated stem); 25 c.c. of alcohol is allowed to flow on to the dust, and the mixture gently shaken until the air bubbles have escaped. Another 25 c.c. of alcohol is then added through the measuring tube and the level of the meniscus read. A value for the apparent specific gravity is thus obtained, from which the proportion of incombustible dust may be estimated by reference to calibration curves of synthetic mixtures.

—C. A. K.

*Coals; Weights of various* — S. B. Flagg. U.S. Bureau of Mines, Tech. Paper 184, 1918. 13 pages.

The testing of many kinds of American coals for Government purposes has afforded the opportunity of observing the relative weights of different varieties from the point of view of storage. General conclusions only are justified, as many factors are involved. Slack coal composed of a mixture of small pieces up to nut size is denser than screened nut coal, as the voids are more com-

pletely filled. Coal with a high moisture content is usually lighter than a similar sample with less moisture. Higher densities are to be expected for coals of high fixed carbon content, and the reverse applies to coals with a large percentage of ash; younger coals, in general, are lighter than those of earlier formation.—C. A. K.

*Coal; Determination of ash of* — H. Mastbaum. Chem.-Zeit., 1918, 42, 385—386, 391—392.

INCINERATION in a muffle-furnace does not always give the same result in the determination of the ash of coal as is obtained when the sample is ignited over a Bunsen burner; the latter method always gives a higher result when the coal contains much calcium carbonate, since this is not decomposed completely and at the same time sulphur compounds are absorbed from the products of combustion of the gas. If a muffle-furnace is not available, the coal should be ignited over a spirit flame or in an electric furnace. For commercial purposes, it is desirable that the method of determining the ash of coal and the temperature employed should be prescribed.—W. P. S.

*Bituminous coal; Storage of* — J. H. Anderson. Trans. Inst. of Marine Eng., 1918, 30, 81—98.

An account is given of the measures adopted to avoid spontaneous combustion of stacked coal with special reference to a pile, in the open, of 16,000 tons of mixed coals—Scotch, North of England, and Yorkshire. The maximum depth of the pile was 16 ft. Vent pipes were inserted at various points. They were 3 in. in diameter and 8 ft. long, with perforations in the side, and the lower end hammered, chisel-shape, so as to leave an aperture of  $\frac{1}{2}$  in. Smaller tubes of 1 in. piping, reaching to the bottom, were inserted over the area of the stack to permit of temperature measurements, which were recorded systematically every day. The temperature was taken at a depth of 7 ft., which was the warmest stratum. Action was taken as soon as a temperature of 90° F. (32° C.) was recorded. Other temperature-measuring tubes were then driven in at points round about until the source of the heating was located. When found, a vent pipe was inserted and the rise of temperature was thereby arrested. If, however, a temperature of 100° F. (38° C.) was found, the stack was trenched above the spot to a depth of 1 ft. and this was repeated daily until the rise of temperature was arrested. The author recommends as maximum depths 12—14 ft. for small graded coal, 9—12 ft. for unwashed mixed coal. He has found slacks to heat when stacked only 6 ft. deep.—H. J. H.

*Coal; Absorption of methane and other gases by* — S. H. Katz. U.S. Bureau of Mines, Tech. Paper 147, 1917. 22 pages.

SAMPLES of air-dried Pittsburgh bituminous coal (moisture 1.61%, volatile matter 37.67%, fixed carbon 55.38%, ash 5.34%) were pulverised to pass a 200-mesh sieve and exposed in closed vessels to atmospheres of air or nitrogen containing known quantities of methane. Computations were made from pressure changes and from gas analyses made with a Haldane apparatus. In the air-methane atmosphere containing 3% methane, an equilibrium was established between the methane and the coal in about four hours, but oxygen was continuously absorbed by the coal and minute quantities of carbon dioxide and monoxide were formed. Freshly pulverised coal, which already contained some methane, absorbed 0.08 c.c. per grm., but coal which had previously been exposed to the



atmosphere for five weeks and had consequently lost some methane absorbed 0.16 c.c. per gram. In the nitrogen-methane mixtures the coal (after five weeks' exposure to the atmosphere) was first introduced into a pure nitrogen atmosphere and left until the absorption of nitrogen ceased and the pressure remained constant. A percentage of methane was then added and comparatively rapid absorption occurred at first, but after two hours an evolution commenced and continued for seven days, after which the pressure (compensated for ordinary expansion) in the closed vessel rose and fell with the temperature. The volume of methane absorbed increased with increase in the partial pressure of the methane, and nitrogen was simultaneously evolved, in amounts somewhat less than the volume of methane absorbed. For methane and nitrogen a definite equilibrium exists, for each temperature, between the partial pressures of the gases and the quantity absorbed by the coal. So far as investigated, the absorption of gases by Pittsburgh coal is closely analogous to the absorption of gases by charcoal.—T. F. E. R.

*Coke; Formation of* — G. Charpy and M. Godchot. *Comptes rend.*, 1918, 167, 322–324.

NON-CAKING coals containing a low percentage of volatile matter can be made to yield a satisfactory coke when they are mixed with a suitable proportion of coal containing an excess of volatile matter which would yield a very bulky and friable coke. Thus a Brassac coal containing 11% of volatile matter when mixed with a Durham coal containing 24% of volatile matter gave the following results by coking at 700° C. For mixtures containing 25% of Durham coal the coke had a resistance of 24 kilos. per sq. cm.; containing 44% a resistance of 45 kilos.; and containing 51% a resistance of 80 kilos. per sq. cm.; above this content the resistance rapidly fell to zero. The content of volatile matter in a mixture giving a coke with the maximum resistance was found to vary considerably with the different coals used. The coking properties of a coal poor in volatile matter may also be improved by addition of the requisite amount of pitch or tar coming from a previous distillation. In the case of a coal too rich in volatile matter, if the latter be suitably reduced by a partial distillation at 450° C., a satisfactory coke can then be obtained by coking at 700° C.—W. G.

*Coking of Illinois coals.* F. K. Oritz. U.S. Bureau of Mines, Bull. 138, 1917. 71 pages.

THE first part of the report deals with the quality and the possible uses of the coke produced from Illinois coal, alone, or when mixed with other coals low in volatile matter. The value of the coals for the production of gas and by-products and the effect of impurities in the coal, on the coke and gas made, are discussed. The second part deals with the nature, physical properties, and chemical composition of Illinois coals and special reference is made to the non-homogeneous structure of the coal. The last part describes in detail the large-scale tests made in beehive ovens, by-product ovens, and gas retorts respectively. Coke from Illinois coals is usually light and friable and contains a large amount of non-fused material, e.g., shale, etc. Mixing with Eastern coal of low volatile content to the extent of 20% Illinois coal produced coke of improved quality. High carbonising temperature appears to improve the coke but the results are not conclusive. Addition of 5% of pitch improves the coke. The yield of gas in by-product ovens ranged from 11,000 to 13,000 cub. ft. per ton (2000 lb.). The average yield of ammonia in by-product ovens was 26.6 lb. of sulphate and in gas retorts about 23 lb.; while the

average tar was 11.9 galls. per ton in the former and about 10–12 galls. in the latter method of carbonisation. Owing to the high sulphur and ash content of Illinois coals their use for coking is limited, and mixing with low sulphur coals is generally necessary. Washing is also usually necessary, and about 25% of the sulphur and 20–30% of the ash are removed during the process. The cost of washing is 5 to 10 cents per ton and from 10 to 25% of the coal is lost during the operation. Crushing the coals before using produced better coke, because the non-fusible layers of the coal were broken up and cemented together by the fusible portions of the coals. Illinois coals contain 37.5 to 46.5% volatile matter; sulphur 1.42 to 5.55%; ash about 10%; oxygen about 11% (all on the dry basis) and low phosphorus. About 40% of the volatile matter (14% of the coal) is "inert" and is driven off largely as water of decomposition, carbon dioxide, and nitrogen. The paper contains eleven plates illustrating the nature of the coke from Illinois coal and its mixtures with other coals, and a selected bibliography.—T. F. E. R.

[*Heat;*] *Apparatus for measuring the loss [of —] up the chimney and the elements constituting this loss.* M. Chopin. *Comptes rend.*, 1918, 167, 335–338.

THE apparatus consists essentially of a pyrometer and an ammeter so arranged that their pointers cross between their scales. In the area where these points of intersection occur are plotted curves from which the percentage loss up the chimney may be read off. The pyrometer registers the temperature of the gases at the base of the chimney, and the ammeter registers the carbon dioxide content of the flue gases, being controlled by the electrical resistance of a solution of sodium hydroxide before and after a known volume of the gases has been bubbled through it.—W. G.

*Flames; Propagation of* — through tubes of small diameter. W. Payman and R. V. Wheeler. *Chem. Soc. Trans.*, 1918, 113, 656–666.

WHEN an inflammable mixture of methane and air, contained in a tube closed at one end, is ignited near the open end of the tube, the flame is found to travel with uniform speed towards the closed end of the tube. The speed of the uniform movement diminishes as the diameter of the tube is reduced as a result of the loss of heat by conduction through the walls of the tube. Further reduction prevents the development of the "uniform movement," and when the diameter is sufficiently small, the flame is not able to spread any measurable distance from the point of ignition. Experiments made with gas mixtures containing from 7.6 to 12.0% methane and tubes varying in internal diameter from 3.6 to 9.0 mm. showed that the range of gas mixtures, in which the uniform movement is developed, becomes smaller as the diameter of the tube is decreased, until with a tube of 4.5 mm. diameter, only one of the mixtures tested (9.95% methane) was found to propagate the flame. The speed with which the flame is propagated tends towards a constant value of about 35 cm. per second as the limiting mixtures are approached. This speed is considered to represent the smallest speed at which continued propagation of flame is possible in mixtures of methane and air. When mixtures of methane and air, contained in horizontal tubes open at both ends, are ignited near one end of the tube, flame begins to travel with increasing speed towards the other end. When a certain distance has been traversed, the flame vibrates backwards and forwards and in tubes of small diameter such vibration leads to the extinction of the flame. The

distance travelled before vibrations begin increases with the length and the diameter of the tube and decreases as the percentage of methane in the mixture approaches that corresponding with the maximum speed of propagation. The relations between these quantities are such that a length of tube of diameter small enough to prevent the passage of flame in a mixture containing say 10% of methane may fail to do so when the mixture contains only 7% of methane, although the former mixture has a greater speed of propagation. In an attempt to realise approximately the conditions under which an inflammation of firedamp takes place within a miner's safety lamp, experiments were also made to determine the length of brass tube which is required to prevent the passage of flame when a 10% methane-air mixture is exploded. The mixture was contained in tubes 2 cm. in diameter and was ignited at the closed ends of these tubes. By varying the lengths of these explosion tubes it was possible to vary the speed acquired by the flame by the time it reached the brass exit tube. Results obtained with brass tubes of 4 to 8 mm. diameter showed that the increased speed of the flame which is obtained by the use of a long explosion tube, reduces materially the protection against inflammation of the external atmosphere which is afforded by brass tubes. An increase in the diameter of the explosion tubes was found to be without influence on the transmission of inflammation. (See also J. Chem. Soc., Oct., 1918.)—H. M. D.

*Water determination in mineral oils; Modification of benzol method of —.* F. C. Fuchs. Eng. and Min. J., 1918, 106, 357.

In the determination of water by distillation with a non-miscible liquid (e.g., benzene), drops of water may remain, in suspension in the benzene, on the walls of the measuring burette and are not easily detected. The addition of a colouring substance soluble in benzene but not in water (e.g., natural asphalt) shows the colourless drops distinctly in the coloured benzene, and by gentle tapping the drops are made to descend into the aqueous layer.—C. A. K.

*Tar oil in Diesel engines.* Moore. See III.

*Use of nitre cake in the manufacture of sulphate of ammonia.* Parrish. See VII.

*Test of a producer gas-fired kiln.* Harrop. See VIII.

*Use of the interferometer in gas analysis.* Selbert and Harpster. See XXIII.

#### PATENTS.

*Fuel from waste materials and making and utilising the same.* J. E. Hughes, Liverpool. Eng. Pat. 117,921. (Appl. Nos. 9701, July 5, and 15,495, Oct. 25, 1917.)

GROUND anthracite, shale, with or without slack, peat, spent tan, or other vegetable refuse, is mixed with a binder such as waste from the manufacture of caustic soda, scum from beet sugar manufacture, or other chemical waste consisting largely of calcium carbonate. The mixture is pressed into pellets which may be burned in a forced draught with or without steam. The pellets may be used for household fires with occasional rekindling with a blow lamp, or larger pieces may be used for furnaces or for generating power gas on motor vehicles.—W. F. F.

*Fuel briquette.* W. Kennett and E. T. Krebs, Carson City, Nev. U.S. Pat. 1,273,619, July 23, 1918. Date of appl., Feb. 13, 1918.

A FUEL briquette consists of crushed oil shale and a heavy semi-liquid binder of carbonaceous matter containing a resinous material in solution.

—W. F. F.

*Furnaces [; Gas —].* A. C. Ionides, jun., London. Eng. Pat. 117,923, July 9, 1917. (Appl. No. 9893 of 1917.)

BACK firing of a mixture of air and gas is prevented by forming a cavity or depression in the furnace wall immediately opposite to the burner nozzle, so as to maintain a stratum of more or less stagnant and consequently cooler gas between the burning jet and the wall opposite thereto.—W. H. C.

*Burner for gas-fired furnaces.* H. J. Yates, Birmingham, S. N. and E. R. Brayshaw, Manchester. Eng. Pat. 118,239, Oct. 16, 1917. (Appl. No. 4812 of 1918.)

THE burner comprises a tubular casing open at one end and provided with a gas nozzle of T-shape projecting into it, the horizontal arm of the T being co-axial with the casing and open at both ends. The orifice nearest the open end of the casing is of larger diameter than the other. An air nozzle is fixed some distance behind the gas nozzle and co-axial with it, so that the greater part of the air passes around the gas nozzle, and a portion passes through it and mixes with the gas. The mixed gases are ignited on issuing from the casing.

—W. F. F.

*Producer gas; Method of making —.* J. W. Hornsey, Summit, N.J., Assignor to General Reduction Gas and By-Products Co. U.S. Pat. 1,270,949, July 2, 1918. Date of appl., Oct. 9, 1911. Renewed Nov. 24, 1917.

CARBONACEOUS material and an oxidising agent are fed continuously through a heated horizontal rotating vessel. The material is retarded in its longitudinal travel by transverse radial ribs on the wall of the vessel, and is lifted and allowed to fall by longitudinal ribs between the transverse ribs. The ribs of adjacent series are not in longitudinal alignment, so that a more intimate mixing with the oxidising agent is obtained.—W. F. F.

*Gas-producer.* A. M. Gow, Duluth, Minn. U.S. Pat. 1,272,252, July 9, 1918. Date of appl., Dec. 20, 1917.

THE fuel in the coking zone of a gas-producer is maintained in a porous condition by the vertical reciprocation of a transverse chopper. The chopper is hollow and carried by a hollow vertical shaft through which a cooling medium circulates, and the shaft is reciprocated vertically by means of a piston working in a cylinder and operated by fluid pressure. The fluid pressure also operates a pawl and ratchet, so that after each stroke the chopper is partly rotated about its vertical axis when raised above the fuel bed.—W. F. F.

*Gas-producers; Charging and poking mechanism for —.* M. J. Owens, Assignor to The Toledo Glass Co., Toledo, Ohio. U.S. Pat. 1,273,177, July 23, 1918. Date of appl., Nov. 5, 1914.

A GAS-PRODUCER is provided with a cover plate movable horizontally and provided on one half with a number of pockets to distribute coal uniformly over the grate. The other half of the cover plate carries a series of vertical guide tubes containing



pokers which are mounted on a common crosshead and reciprocated by fluid pressure. The cover may be moved so that either half is over the producer. Each coal pocket is provided with slides at top and bottom, and a complementary series of supply shoots is arranged at the side of the producer to supply the pockets when the cover is displaced so that the pokers are over the grate.—W. F. F.

*Gaseous mixtures; Separation of the constituents of* — [e.g., methane from natural gas]. B. S. Lacy, Seward, N.J., Assignor to The Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,272,059, July 9, 1918. Date of appl., Sep. 20, 1916.

To separate methane from natural gas, the compressed gas is passed through one channel of a temperature exchanger, when it is expanded and the liquefied hydrocarbons are separated. The non-liquefied gas is passed through a second channel of the exchanger in an opposite direction to the first. The liquefied hydrocarbons are evaporated at a rising temperature by passing them through the lower part of a third channel of the exchanger also in an opposite direction to the first, the gas produced passing on through the upper part of the channel.—J. H. J.

*Gas; [Electrolytic] process of making combustible* — C. F. Euler, Topeka, Kans. U.S. Pat. 1,273,050, July 16, 1918. Date of appl., Apr. 4, 1916.

An electric current is passed through water containing insufficient acid or salt to liberate any substantial amount of gas other than oxygen and hydrogen. The anode and cathode are horizontal, the cathode being below the anode, so that the gas liberated from the cathode rises past the anode and mixes intimately with the gas liberated uniformly from it. The gases are withdrawn from the closed container without admixture of extraneous gases.—W. F. F.

*Coal gas retorts and generator plants; Operation of* — H. Strache, Vienna. Ger. Pat. 305,721, Sep. 28, 1916. Under Int. Conv., Sept. 30, 1915.

The producer is operated with bituminous coal, and the mixture of producer gas and coal gas produced during the period of blowing with dry air is utilised for heating coal gas retorts. As soon as the temperature in the lower part of the producer is sufficiently high, the current of air is replaced by steam and the water gas, which is also enriched with coal gas on passing through the layer of coal in the upper part of the producer, is led off through another outlet and mixed with coal gas from the gas retorts.—L. A. C.

*Moist fuel; Drying of and gas production from* — A. Eckardt, Zwickau. Ger. Pat. 306,060, Mar. 4, 1916. Addition to Ger. Pat. 302,827 (this J., 1918, 330 A).

The furnace used for drying the fuel is utilised also for heating the mixture of steam and air delivered beneath the grate of the producer.—L. A. C.

*Gases obtained from coal or other carbonaceous fuel; Recovery of cyanogen bearing compounds from* — E. V. Espenhahn, Brighton, Victoria, Australia. Eng. Pat. 109,254, July 25, 1917. (Appl. No. 10,703 of 1917.) Under Int. Conv., Aug. 8, 1916.

The gas to be purified is passed through centrifugal or other washers to remove tar, and then through a strong solution of sodium carbonate containing

ferrous sulphide in suspension at a temperature of 40°–90° C. Sodium ferrocyanide is produced by the action of hydrogen cyanide gas, the sludge is removed by filter-pressing, and the salt crystallises on cooling. The clear liquid is returned to the washing tank with the addition of fresh sodium carbonate and ferrous sulphide. The sodium ferrocyanide may be treated with sulphuric acid to produce hydrogen cyanide, which is subsequently converted into alkali cyanides and sodium ferrous ferrocyanide. The latter may be used with sodium carbonate for the washing tank, when sodium ferrocyanide and ferrous sulphide are produced by the action of hydrogen sulphide. The ferrous sulphide then reacts with sodium carbonate and hydrogen cyanide as described above. In a modification, the gas may be first treated for the recovery of ammonia and a portion of the gas may be passed through a solution of ferrous sulphate to produce ammonium sulphate free from cyanides by the action of ammonia and hydrogen sulphide. Ferrous sulphide is also formed and may be mixed with sodium carbonate for washing the gas as above. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 10,876 and 26,566 of 1902 and 2929 of 1906; this J., 1903, 691, 1243; 1906, 536.)—W. F. F.

*Petrol; Manufacture of volatile oils such as* — M. A. II. de Dampierre, Paris. Eng. Pat. 109,796, Aug. 15, 1917. (Appl. No. 11,745 of 1917.) Under Int. Conv., Sep. 6, 1916.

HYDROGEN is injected into heavy oil whilst this is being heated in a still, and the mixed vapours and gas ascend through a water-cooled vertical pipe containing a number of perforated nickel discs. A safety valve situated at the upper end of the pipe allows a determined pressure to be maintained in the still and pipe. The vapours of the heavy oil condense and run back into the still, whilst the hydrogen and the vapours of volatile oils pass through the valve to a condenser.—T. St.

*Liquid fuel [for internal combustion engines].* S. Dyson, Elland, Yorks. Eng. Pat. 118,021, Sep. 12, 1917. (Appl. No. 16,272 of 1917.)

MINERAL oil, including olefines, paraffins, or other hydrocarbons not of coal tar origin, 70 parts, and carbon bisulphide, 30 parts, are mixed with slaked lime 5–10 parts, and the mixture agitated and then washed with water which is drained off with the impurities.—W. F. F.

*Hydrocarbon oils; Process of refining* — H. T. Maitland, Marcus Hook, Pa., Assignor to Sun Co., Philadelphia, Pa. U.S. Pat. 1,272,979, July 16, 1918. Date of appl., Jan. 13, 1916.

A STREAM of sulphuric acid is broken up into an extremely finely divided state by a stream of gas, and the resulting mist discharged into an inverted funnel, the open end of which dips below the surface of the oil. The body of oil is independently agitated to ensure thorough distribution of the acid particles which are forced below its surface by the pressure exerted by the stream of gas.—T. St.

*Still for crude bituminous materials.* C. P. Price, Assignor to American Tar Co., Malden, Mass. U.S. Pat. 1,273,091, July 16, 1918. Date of appl., Dec. 3, 1912.

THE still is provided with a trough at its upper edge, and a conical cover seating in the trough. The distillate runs down the cover into the trough and is withdrawn at a number of points by right-angled branch pipes into a common collecting pipe.

The short vertical section of each branch pipe leading into the collecting pipe carries a funnel at its upper end. The still is heated by a pipe coil through which steam is passed.—W. F. F.

*Producer-gas; Process of manufacturing* —. E. Fleischer, Dresden-Alttstadt, Germany. U.S. Pat. 1,273,474, July 23, 1918. Date of appl., May 13, 1914.

SEE Fr. Pat. 471,341 of 1914; this J., 1915, 414.

[*Mineral*] *oils; Process and apparatus for purifying* —. E. C. R. Marks, London. From The Swan Process Oil Co., Deventer, Colo., U.S.A. Eng. Pat. 118,353, Sep. 25, 1917. (Appl. No. 13,784 of 1917.)

SEE U.S. Pat. 1,250,526 and 1,250,527 of 1917; this J., 1918, 117A.

*Art of separating materials from gases.* U.S. Pat. 1,273,791. See I.

*Process and means for producing oils for different purposes from sharp, middle, cresotic, or similar oils.* Eng. Pat. 117,914. See III.

## II.B.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Carbonaceous substances; Apparatus for the destructive distillation of* —. S. N. Wellington, London. Eng. Pat. 117,929, July 30, 1917. (Appl. No. 10,974 of 1917.)

A SAFETY device for insertion in a gas main comprises a pressure chamber fitted with one or more mushroom valves. Each valve rod is connected to the valve of a whistle operated by compressed air, so that the whistle is blown when the pressure in the main becomes excessive.—W. F. F.

*Shale or the like; Apparatus for the treatment [distillation] of* —. R. M. Catlin, Franklin, N.J., Assignor to Catlin Shale Products Co., Wilmington, Del. U.S. Pat. 1,272,377, July 16, 1918. Date of appl., Aug. 9, 1917.

AN inclined tubular retort for the distillation of shale or the like is provided with a concentric helical conveyor of diameter slightly less than the retort and of varying pitch. The material is fed forward more quickly and is thus spread out more thinly as it approaches the outlet end.—W. F. F.

*Gas lighting. Gaseous combustion.* A. C. Ionides, jun., London. Eng. Pat. 118,313. (Appl. Nos. 11,891, Aug. 18, and 16,806, Nov. 15, 1917.)

Gas supplied by a blower passes through a governor, which maintains the pressure between predetermined limits, to a double beat valve and thence to one side of a floating bell. Air is supplied by another blower at a higher pressure to another double beat valve and thence to the other side of the floating bell. The bell actuates the two valves so as to maintain the relative pressures of gas and air at any desired ratio. The gas and air pass by separate pipes to any system of burners, each burner being adjusted to pass the correct proportions of gas and air at the given pressure ratio. Temporary variations in the quality of gas are compensated by adjusting a cut-off valve on the air supply pipe. When any

permanent adjustment of all the burners is required a test light or test flame is adjusted at the same time, so that if there is a temporary variation in the quality of the gas the necessary adjustment of the air valve may be made with reference to the test light.—W. F. F.

*Incandescence mantles; Collodion coating for* —. R. Langhans, Berlin. Ger. Pat. 306,103, Nov. 21, 1917.

COLLODION to be used as a protective coating for burnt-off incandescence mantles, is mixed with a molecular compound of phthalic anhydride with an aromatic amino or hydroxy compound.

—J. F. B.

*Incandescence lamps; Manufacture of [electric]* —. F. Blau, Charlottenburg, Germany, Assignor to General Electric Co. U.S. Pat. 1,269,520, June 11, 1918. Date of appl., Apr. 18, 1916.

BARIUM azoimide is introduced into the bulb of an incandescence lamp and decomposed by heating to a temperature below the softening point of the glass, so as to produce metallic barium and anhydrous barium oxide on the internal surface of the bulb, the barium oxide acting as a drying material.—B. N.

*Electrode for arc-lamps.* H. R. Edgecomb, St. Marys, Pa., Assignor to Speer Carbon Co. U.S. Pat. 1,272,401, July 16, 1918. Date of appl., Dec. 7, 1916.

THE electrode comprises an inner core, and an outer shell of carbon capable of burning at approximately the same rate as the core, the shell being coated with a material of greater electrical conductivity. An electrically-conducting heat-insulating film containing carbon is disposed between the core and the shell.—B. N.

*Film conductor [for electrical discharge apparatus] and the method of making the same.* I. Langmuir, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,273,628, July 23, 1918. Date of appl., June 6, 1914. Renewed May 1, 1917.

GAS-FREE adherent metallic conducting films, for use as anodes in electrical discharge apparatus, are deposited upon insulating surfaces, by heating a highly refractory metal to a temperature near its melting point in a vacuum having a residual gas pressure below 0.001 mm. of mercury, i.e., too high to permit of the passage of a disruptive discharge, the surface upon which the film is to be deposited being exposed to the metallic vapour.

—B. N.

*Incandescence lamps; Method of exhausting* —. I. Langmuir, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,273,629, July 23, 1918. Date of appl., Dec. 5, 1914.

SEE Eng. Pat. 1088 of 1915; this J., 1916, 105.

[*Electric lamps;*] *Leading-in conductor [for —]*. C. G. Fink and W. E. Koerner, East Orange, N.J., Assignors to General Electric Co. U.S. Pat. 1,273,758, July 23, 1918. Date of appl., Sep. 25, 1915.

SEE Eng. Pat. 104,758 of 1916; this J., 1917, 498.

*Decolorising carbons and process of producing the same.* Eng. Pat. 116,253. See XVII.

*Process of manufacturing decolorising carbon.* Eng. Pat. 117,828. See XVII.



### III.—TAR AND TAR PRODUCTS.

*Tar oil in Diesel engines.* H. Moore. Diesel Engine Users' Assoc., May 26, 1918. [Reprint.] 24 pages.

Almost all heavy tar distillates are suitable for use in Diesel engines. The gross heat value of tar oils of British origin averages 10·2% less than that of the petroleum oils sold in this country, but the increase in consumption is not higher by this amount for the reasons that tar oil generally yields a higher thermal efficiency than petroleum oil (probably due to its lower viscosity), and owing to the average hydrogen content of tar oil being only about 6·9% as against about 11·5% for petroleum oils, the difference between gross and net calorific values is not so great as with petroleum oils. The average ignition point of tar oils is about 480° C. as against 260° C. for petroleum oils, and it is this difference which is responsible for the troubles in obtaining ignition of tar oils. With a normal tar oil, petroleum is necessary for starting with compressions below 670 lb. per sq. in., but good running is obtained at over  $\frac{3}{4}$  load with a compression of 460 lb. per sq. in. Shale oil is particularly serviceable as an ignition oil with pilot ignition gear on account of its low ignition point. High temperatures of the circulating water help the combustion of tar oil to a small extent. Heating the cycle air lessens output of the engine by rarefying the air, but aids the combustion of tar oils. Advancing the fuel valve is a distinct advantage. The best results have been obtained with 9° advance when burning tar oil alone, and about 5° advance when using pilot ignition gear (running engine at 250 revs.). Mixing oils is of doubtful advantage, as it is necessary to employ more than half petroleum to obtain moderately good results, even when oils are such as will stand mixing without forming deposits. Hot blast is by far the most helpful modification for burning tar oil, but is rather dangerous, as it might cause explosions in the fuel valve casing. The fuel valve face suffers a little more with hot blast than in normal practice. Heating the fuel is useful from the point of view of increasing efficiency by reducing viscosity and as a means of dissolving naphthalene. Improved running on tar oil at low loads is obtainable by restricting the flame plate, but this is only at the expense of the full load results. Wear on exhaust valve is not very serious with tar oils, but is a much more important matter when burning raw tars. It only becomes serious when firing is uncertain, and is generally much worse when running on tar oil alone than with the pilot ignition system. Oils which give an abrasive ash on ignition may cause trouble even when this amounts to less than 0·08%, but when the ash consists largely of sodium sulphate (arising from washing tar acids from creosote), much higher ash contents are permissible. Tar oils containing pitch cause many troubles, but it is almost entirely the free carbon which affects the use of such mixtures in internal combustion engines.—T. St.

#### PATENTS.

*Sharp, middle, ercosote or similar oils; Processes and means for producing oils for different purposes from —.* A. F. Martin, London, Eng. Pat. 117,914, June 7, 1917. (Appl. No. 8319 of 1917.)

The oil is mixed with aqueous sodium hydroxide solution, agitated and heated, and the mixture allowed to settle. The liquid portion is run off into a still and distilled for fractions boiling respectively below 180° C., from 180° to 225° C., and from 225° to 360° C. The first fraction is mixed with an equal amount of benzol or petrol, and the mixture redistilled. The fraction distilling between

90° and 180° C. is a high-grade motor fuel, having a flash point of 35°—45° F. (about 2°—7° C.), or over, and sp. gr. approximately 0·7—0·75. By again distilling the latter fraction from 90° to 180° C., a high-grade aeroplane motor spirit oil is produced. The second main fraction is suitable, either without further treatment, or, if dark in colour, after redistilling, for use as a Diesel engine oil. The third main fraction is gently heated and agitated with an addition of a quantity of finely disintegrated commercial pitch, whereby a good boiler fuel oil is obtained. A combination of apparatus, and devices for economically carrying out the several steps of the process, are described. —T. St.

*Aniline-reducer.* A. H. Stevens, Brooklyn, N.Y., Assignor to Stevens Bros., New York. U.S. Pat. 1,267,819, May 28, 1918. Date of appl., Mar. 25, 1918.

AN apparatus for the production of aniline by reduction of nitrobenzene with iron filings and hydrochloric acid comprises a tank provided with a false bottom and a removable lining to allow of easy renewal after corrosion. A horizontal bar passes through a slot in the lower end of a vertical shaft mounted within the tank and this bar carries rabble blades which are mounted on the bar in such a way that the latter is practically completely enclosed and thus largely protected from corrosion.

*Aromatic hydrocarbons; Process of producing —.* R. L. Hill, Chester, Pa., Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,269,439, June 11, 1918. Date of appl., July 26, 1915. Renewed Mar. 28, 1918.

ANOMATIC hydrocarbons of higher boiling point than toluol (e.g., heavy naphtha) are heated with an alkaline-earth carbide, such as calcium carbide, at a temperature exceeding 500° C., and under a pressure greater than atmospheric pressure. Hydrocarbons of lower boiling point are produced. —C. A. K.

*Still for crude bituminous materials.* U.S. Pat. 1,273,091. See IIA.

*Manufacture of impregnated material.* U.S. Pat. 1,273,673. See XIII.

### IV.—COLOURING MATTERS AND DYES.

*Dyestuffs; Natural —.* Bull. Imp. Inst., 1918, 16, 1—16.

*Brazilletto wood* from the Bahamas. This dye-wood is derived from a species of *Casalpinia*. Three samples were received in pieces 16—38 ins. long and  $1\frac{1}{2}$ —3 ins. thick. They all yielded colours similar to those obtained with commercial Brazil wood, the only difference being in the depth of colour obtained on wool mordanted with alum. The aqueous extract ranged from 8 to 11%. These Brazilletto woods were quite different from a sample of "Brazilada" wood previously received from the Bahamas, which resembled logwood in appearance and properties. They are not likely to be of much commercial interest. A sample of logwood from the Bahamas was judged to be of good quality equal to the best Jamaica wood.

*Indigo* from Nyasaland and Nigeria. Two samples from Nyasaland contained respectively 20·4 and 36·1% of indigotin, the former showing 22·2% of ash, principally earthy matter. They were classed commercially as "low" and "middling," and had been prepared from wild plants. A sample from

Nigeria showed 31·3% of ash, 27·5 of indigotin, and 1·5 of indirubin (Indigo Red); it was classed as rather low grade, but marketable.

"Indian madder" from S. Africa. A sample of roots of *Rubia cordifolia* from Natal was considered to be equal in quality to the Indian product and stronger than commercial ground madder (*R. tinctorum*); the tints obtained with the Natal roots differed somewhat from those given by the standard samples of madder.

*Lichen* from the Falkland Islands. A sample of bushy lichen, very abundant in the Falkland Islands, was identified as *Neuropogon melanzanthum*, giving buff, orange, and brown shades on boiling with unamordanted wool. Prolonged boiling gave darker colours, especially with the addition of ammonia. This lichen possesses dyeing properties which make it suitable for local use, but it would be of no interest for export.—J. F. B.

*Colouring matter of the glumes of the sweet Sorghum; Industrial application of the —.* A. Piédallu. Comptes rend., 1918, 167, 345—348.

The glumes of the sweet Sorghum and of several varieties of Sorghum with black glumes contain a dye, which can be extracted with dilute acid and which dyes wool, silk, leather, and cotton. The dye is extracted by heating the glumes in an autoclave with 1% sulphuric or hydrochloric acid or with 2% sodium bisulphate. The colours obtained on wool and silk are bright and strong, the shades being from violet to red, from beige (natural wool colour) to brown, and from pearl grey to deep grey varying with the mordant. The dye may be used with or without mordants, the most satisfactory mordant being stannous chloride, which gives bright amaranth and crimson tints. All the shades are fast to washing. They pass a little towards violet under the influence of alkali, the tint being restored by dilute acid.—W. G.

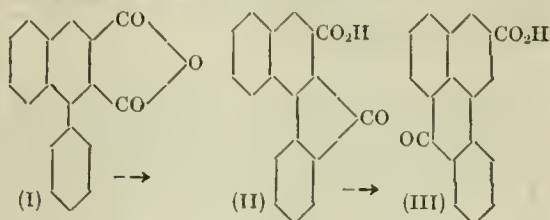
*p-Phenylenemethyldiamines; Acylated —.* G. T. Morgan and W. R. Grist. Chem. Soc. Trans., 1918, 113, 688—694.

METHYLANILINE is prepared by treating aniline with formaldehyde in presence of zinc dust and caustic soda (Eng. Pat. 102,834; this J., 1917, 207) and the secondary base is converted into acyl derivatives by heating for several hours with concentrated formic acid, acetic acid, etc. The corresponding *p*-phenylenemethyldiamine derivatives may be obtained by known methods, the most convenient being by nitration of the acylmethylaniline and reduction of the *p*-nitro compound, which is the main product of the nitration with ordinary nitrating mixture at 0°—10° C. *p*-Aminomethylformanilide, m.pt. 132° C., and *p*-aminomethylacetanilide, m.pt. 63° C., when treated with nitrous anhydride in dry acetone solution, give colourless diazonium hydroxides which combine with dry  $\beta$ -naphthol with elimination of water. Azo dyestuffs are formed by diazotising the *p*-aminomethylacylanilides in the ordinary way and coupling with various phenolic compounds. (See also Eng. Pat. 111,321; this J., 1918, 5A.)—J. F. B.

*Benzanthrone; A new synthesis of —. II. Transformation of 1-phenylnaphthalene-2,3-dicarboxylic acid anhydride into benzanthronecarboxylic acid.* A. Schaarschmidt and E. Korten. Ber., 1918, 51, 1074—1082.

WHEN 1-phenylnaphthalene-2,3-dicarboxylic acid anhydride (I) is heated with 91% sulphuric acid for 3 hours at 155° C., it yields benzanthronecarboxylic acid (III) and a sulphonated 3,4-benzofluorenone-1-carboxylic acid (II). As this acid (II)

is the product formed by the prolonged action of sulphuric acid in the cold, and gives the same result if heated as above, the chief reaction may be represented thus:



It is an example, therefore, of the rupture of a weak 5-ring and establishment of a 6-ring. 3,4-Benzofluorenone itself suffers a similar change when heated with sulphuric acid, and the imide corresponding with the above anhydride exhibits the phenomenon most readily. (See further J. Chem. Soc., 1918, 1, 434.)—J. C. W.

*Benzanthrone; A new synthesis of —. III. A.* Schaarschmidt and E. Georgeacopol. Ber., 1918, 51, 1082—1087.

WHEN 3,4-benzofluorenone is fused with potassium hydroxide, the 5-ring is ruptured in the two possible ways, the products being salts of 1-phenylnaphthalene-2-carboxylic and *o*- $\alpha$ -naphthylbenzoic acids. If the free acids are treated with phosphorus pentachloride and then aluminium chloride, the former yields 3,4-benzofluorenone again and the latter benzanthrone. (See further J. Chem. Soc., 1918, 1, 434.)—J. C. W.

*Quinonimide dyes; Nature of cyclic —.* F. Kehrman. Annalen, 1917, 414, 131—188.

A THEORETICAL paper containing a discussion of the results of investigations of the constitution of azonium, azthlonium, azoxonium, carboxonium, carbothlonium, and acridonium compounds. They are regarded in general as desmotropic substances which acquire the ortho- or the para quinonoid structure according to the conditions. (See also J. Chem. Soc., 1918, 1, 449.)—C. S.

#### PATENTS.

*Lignine sulphonic acid or its salts; Method of preparing azo colouring matters from —.* E. Oman, Stockholm. Eng. Pat. 103,822, Jan. 4, 1917. (Appl. No. 201 of 1917.) Under Int. Conv., Jan. 28, 1916. Addition to Eng. Pat. 103,479.

AFTER the coupling has been made according to the process specified in Eng. Pat. 103,479 (this J., 1918, 407A), an inorganic or organic base is added, with stirring, to the reaction mixture until slightly alkaline, whereby the corresponding salt of the azo compound is precipitated, or may be precipitated by means of a salt, e.g., a soluble carbonate. If the reaction be carefully carried out at a low temperature, the base may be added to the diazotised amine before coupling. The colour of the precipitated compound varies according to the base employed.—J. F. B.

*Mono-azo dyestuffs of the pyrazolone series able to be chromated and process of making same.* E. Reber, Assignor to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,270,325, June 25, 1918. Date of appl., Sep. 11, 1917.

NEW mono-azo dyes of the pyrazolone series are prepared by combining diazo-derivatives of anthranilic compounds with hydroxyaryl substituted



pyrazolones. In particular, the dyestuff obtained by coupling the diazo-derivative of anthranilic acid with 1-(5'-sulpho-3'-carboxy-2'-hydroxy)-phenyl-3-methyl-5-pyrazolone dyes wool yellow tints in an acid bath which turn to brownish-yellow on being after-chromed.—L. A. C.

*Vegetable dye and process of making same.* H. Warkup, Kingston, N.Y., Assignor to J. S. Young and Co., Hanover, Pa. U.S. Pat. 1,273,293, July 23, 1918. Date of appl., Dec. 20, 1917.

A QUERCITRON extract is evaporated to form a thick liquid, which is then treated at the boiling point with a number of mordanting substances. After standing for some time, the liquid is neutralised or rendered slightly alkaline with sodium carbonate, heated to expel carbon dioxide and finally evaporated *in vacuo* to about 51° Tw. (sp. gr. 1.255). The bath produced by dilution of the product can be used for the direct mordanting and dyeing of cotton.—L. A. C.

*Ortho[hydr]oryazo dyestuffs; Substantive — and a process of making same.* E. Anderwert, H. Fritzsche, and H. Schobel, Assignors to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pats. 1,265,030 and 1,265,031, May 7, 1918. Date of appl., Jan. 24, 1917.

SEE Eng. Pat. 12,250 of 1915; this J., 1916, 1008.

*Gallocyanine dyestuffs; Condensation product of — with amines, and process of making same.* A. Grob and J. Würzler, Assignors to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,268,453, June 4, 1918. Date of appl., July 7, 1917.

SEE Eng. Pat. 116,754 of 1917; this J., 1918, 407A.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Silks; Differentiation of natural and artificial —.* A. Herzog. Kunststoffe, 1917, 7, 277—278. Z. angew. Chem., 1918, 31, Ref., 225.

A PHYSICAL method of differentiation is described which is based on the differences between the refractive indices and which is applicable in the case of dyed fibres. A fibre of natural silk mounted in aniline and examined under a microscope with a Nicol prism is almost invisible when its longitudinal axis is at right angles to the plane of polarisation of the prism, and becomes more and more visible when the stage is rotated, until it reaches a maximum of visibility at right angles to the original position. Artificial silk fibres have a refractive index considerably below that of aniline and are distinctly visible at all positions of the revolving stage.—J. F. B.

*Rape straw as a paper and fodder material.* E. Heuser and T. Blasweiler. Papier-Zeit., 1918, 43, 593, 613. Chem.-Zeit., 1918, 42, Rep., 108.

THE examination of the rape plant showed, on the dry substance, cellulose, 30.31; pentosans, 24.10; lignin, 40.06, and ash, 5.53%. The composition of the plant substance is somewhat similar to that of cereal straw, which suggests that rape, like straw, might be used as a raw material both for paper pulp and for fodder. A pulp prepared by the soda process had the composition: cellulose, 55.60; pentosans, 30.50; lignin, 10.10; ash, 3.77%. In consequence of the higher proportion of lignin in the rape straw as compared with cereal straw, a sharper treatment is necessary in order to obtain

a similar degree of resolution. The pulp would be described from a technical point of view as a "half-cellulose," such as is employed for the manufacture of boards.—J. F. B.

*Utilisation of rice and its by-products.* See XIXA.

## PATENTS.

*Textile fabrics, wood, leather, and other materials; Treatment of —.* W. M. Gillard, Chelsfield, and M. Heslam, Upper Holloway. Eng. Pat. 117,640. (Appl. Nos. 5769, Apr. 24, and 14,675, Oct. 10, 1917.)

IN order to render material such as canvas, wood, leather, etc., more suitable for the reception of photographic prints, hand painting, etc., it is coated with a preparation containing a filling material such as kaolin, a binding medium such as gelatin, and substances such as formaldehyde and glycerin, to impart insolubility and flexibility. —B. V. S.

*Wool scouring or like effluents or liquors; Process for the separation of grease from —.* G. G. Jarman, Huddersfield. Eng. Pat. 118,332, Aug. 25, 1917. (Appl. No. 12,220 of 1917.)

Wool scouring liquor or other effluent containing grease is first passed, preferably just below the boiling temperature, through a hydro-extractor or centrifugal filter in order to separate the sand and heavy foreign impurities, and is then delivered, preferably from an overhead feed tank into which the filtered liquor is pumped, to a cylindrical or bowl centrifugal separator, having a feed jet and baffle-plate at the bottom, in which the grease is separated from the aqueous layer and both are discharged through separate delivery channels at the top.—J. F. B.

*Fibre; Method of treating vegetable —.* M. W. Marsden, Philadelphia, Pa. U.S. Pat. 1,269,476, June 11, 1918. Date of appl., Feb. 6, 1917.

FOR the manufacture of pulp, the incrusting and other difficultly soluble bodies are removed by subjecting the prepared stock to the solvent action of a mixture of sodium chloride and lime in the presence of heat, moisture, and pressure.—J. F. B.

*Fibre plants and woody material; Process of retting, degumming, and drying —.* B. Loomis, Hartford, Conn. U.S. Pat. 1,271,591, July 9, 1918. Date of appl., Aug. 2, 1915.

THE material is placed in layers on grids in a closed tank, and hot water and fatty acid solvents including acetic acid are circulated around and between the layers until the gummy and resinous matter has been released; this is floated off and separated from the water, and the solvent liquor is repeatedly reheated and circulated through or in contact with the fibre material until retting and degumming are completed.—J. F. B.

*Fibre; Process for treating —.* R. R. Roberts, Washington, D.C., Assignor to United Textile Co. U.S. Pat. 1,272,851, July 16, 1918. Date of appl., July 30, 1917.

THE fibres are boiled under pressure in an alkaline solution containing kerosene, then treated in an acid bath, washed in a saponaceous solution, rinsed, and dried.—J. F. B.

*Silk; Process for degumming* — L. Elkan Erben Ges., Charlottenburg. Ger. Pat. 305,920, Aug. 26, 1917.

SOLUTIONS of "lignic acid" are employed in the degumming baths.—J. F. B.

*Cuprammonium cellulose solutions for artificial threads; Preparation of stable* — by the use of sugars. Glanzfäden Akt.-Ges., Petersdorf. Ger. Pat. 306,107, Oct. 13, 1917.

MONOHYDROSE sugars of the dextrose group are employed in the cuprammonium solutions as well as sugars of the sucrose group, in such quantities that the spinning qualities of the solutions are not impaired. Even at normal temperature the monosaccharides exert a reducing action which impedes the oxidation of the copper compound and the cellulose during the preparation of the solution, so that the solubility of the copper oxide in ammonia is greater and the cellulose dissolves more rapidly and completely; the stability of the cellulose solution is also improved. A proportion of 0.25% of starch glucose or invert sugar calculated on the weight of the cellulose is sufficient to counteract the tendency to oxidation during the preparation of the cellulose solution.—J. F. B.

*Pulp [from bracken] for making paper, paper boards and the like; Manufacture of* — W. T. Moore, Bromley, Kent. Eng. Pat. 118,291, Sep. 12, 1917. (Appl. No. 3651 of 1917.)

BRACKEN is collected either in the autumn during the withering period or in the winter when the fronds are dead, and the pinnae and pinnules (primary and secondary leaflets) are stripped off the stems by drawing the fronds through an aperture formed by two plain or toothed pieces of wood or metal placed parallel in the same plane, in a frame, at a suitable distance apart. The leaflets fall on one side of the frame and the stems on the other side. The leaf material is boiled separately with water without pressure and without the use of alkali, and the stems are digested, preferably without pressure, with 5–10% of their weight of soda, preferably in the form of caustic soda. The two materials are broken-in, beaten, bleached, and washed and may be blended together at any stage after the breaking process.—J. F. B.

*Treating to an impregnating fluid, particles of non-uniform moisture content; Apparatus for* — [e.g. in the preparation of wood pulp]. G. H. Hess, La Grange Park, Ill. U.S. Pat. 1,265,330, May 7, 1918. Date of appl., May 17, 1917.

THE apparatus comprises a dryer in which the material, e.g., wood chips, is partially dried; a bin in which the partially dried material is retained until the moisture content becomes uniform throughout; a storage bin to receive the contents of the preceding bin, and a vessel in which the material is subjected to the action of an impregnating fluid, e.g., a digester for the manufacture of wood pulp.

*Paper pulp; Manufacture of* — S. D. Wells, Madison, Wis. Dedicated to the Government and people of the United States. U.S. Pat. 1,269,350, June 11, 1918. Date of appl., Apr. 30, 1918. (Dedicated to the public.)

IN the process of digesting wood or other fibrous material, the desired amount of moisture is introduced into the digester during the process, in direct combination with the steam, whereby the cellulose is protected to a marked degree from the action of the digestion liquors, while their action on the incrusting matters is not diminished.

—J. F. B.

*Paper; Safety* — E. E. Schmidt, Boston, Mass., Assignor to Paramount Safety Paper Co., Chicago, Ill. U.S. Pat. 1,269,863, June 18, 1918. Date of appl., Feb. 6, 1917.

THE paper is coated with a composition containing ingredients capable of being decomposed by acid and alkaline ink eradicators to release substances which stain the paper and to render writing ink containing iron indelible. Suitable ingredients are a soluble ferrocyanide, an iodide, glycerin, ox-gall, tapioca flour, and caustic soda; the ox-gall is for the purpose of rendering the paper permeable to the composition.—J. F. B.

*Paper containing salicylic or benzoic acid; Manufacture of* — L. Elkan Erben Ges., Charlottenburg. Ger. Pat. 305,956, June 27, 1917.

PAPER is treated with easily soluble salts of salicylic or benzoic acid. The absorption by the fibre is so complete and permanent that no visible efflorescence of crystals occurs.—J. F. B.

*Paper textiles; Process for cleaning old* — T. Goldschmidt A.-G., Essen. Ger. Pat. 306,000, Sep. 12, 1917. Addition to Ger. Pat. 305,427 (this J., 1918, 410 A).

AFTER cleansing with bleach liquor or chlorine water, any looseness of texture produced by the treatment is corrected by impregnating the textiles with a mixture of gelatin solution and glycol, before finishing by mechanical treatments.

—J. F. B.

*Cardboard; Process for rendering* — grease- and waterproof. L. Buchbinder, Vienna. Ger. Pat. 306,028, Dec. 2, 1917.

THE proofing composition consists of a mixture of a glue precipitate with a decoction of Iceland moss containing gypsum ("Federweiss") or magnesia, or with a concentrated alcoholic solution of resin. The composition is applied warm, e.g. with a brush in the case of containers intended for fatty substances which readily solidify, or by filling the container with the composition and pouring out after 3–4 minutes, in the case of those intended for substances of low melting point. The coating must be dried at the ordinary temperature. The mixture of magnesia and glue-tannin fills up the pores of the paper and makes the container watertight; if it has to withstand mineral oil, a second coating of waterglass is applied. An alternative method consists in treating the cardboard with a mixture of precipitated glue and magnesia and then soaking in a decoction of Iceland moss containing formaldehyde.—J. F. B.

*Drying paper, pasteboard, wood-pulp, and other materials; Apparatus for* — L. A. Lyche, Sandviken, Norway. U.S. Pat. 1,271,738, July 9, 1918. Date of appl., Oct. 12, 1917.

SEE Eng. Pat. 5855 of 1915; this J., 1916, 41.

*Collodion coating for incandescence mantles*. Ger. Pat. 306,103. See 11b.

*Method of preparing azo colouring matters from lignine sulphonic acid or its salts*. Eng. Pat. 103,822. See IV.

*Process of fermenting sulphite [cellulose] liquor*. U.S. Pat. 1,269,287. See XVIII.

*Process of obtaining alcohol from sulphite [cellulose] liquor*. U.S. Pat. 1,273,392. See XVIII.



## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Laundry [textile] goods; Oxygen tendering of —.*  
P. Heermann. Chem.-Zeit., 1918, 42, 337–338, 342–343.

THE author replies to various objections which have been raised against his previous work (this J., 1918, 233A) and brings forward evidence to show that washing powders containing available oxygen or bleaching agents are more injurious to fabrics than are those free from these substances. In practice, it is quite possible to have conditions under which the bleaching powders will produce local destruction and perforation of fabrics.

—W. P. S.

*Natural dyestuffs.* See IV.

*Industrial application of the colouring matter of the glumes of the sweet Sorghum.* Piédallu. See IV.

### PATENTS.

*Kiers; Vomiting —.* E. D. Jefferson, Boston, Mass., U.S.A. Eng. Pat. 112,260, Sep. 26, 1917. (Appl. No. 13,894 of 1917.) Under Int. Conv., Dec. 29, 1916.

A VOMITING kier of the type described in Eng. Pat. 25,307 of 1904 (U.S. Pat. 772,450; this J., 1904, 1202) is provided with an external trap into which the liquor accumulating at the bottom of the kier can penetrate through a non-return valve. The top of the trap is connected with a mechanically time-controlled valve through which a determined quantity of high-pressure steam is admitted at intervals above the liquid collected in the trap. An external vomit-pipe leads from the bottom of the inside of the trap into the top of the kier, and the liquid is forced up by the steam pressure and sprayed over the top of the goods. A narrow vent pipe is provided between the top of the trap and the top of the kier to equalise the pressure when the trap is filling up. A battery of kiers may be arranged with the steam valves of all the kiers controlled at one place by a single motor, with means for throwing the control mechanism of any kier out of action.—J. F. B.

*Mordanting of vegetable fabrics and fibres.* The Calico Printers' Association, Ltd., and F. Ashton, Manchester. Eng. Pat. 117,715. (Appl. Nos. 12,510, Aug. 31, 1917, and 3287, Feb. 25, 1918.)

CHROMIUM mordants, either alone or in conjunction with other mordants, are fixed on vegetable fibres by first preparing the fabric in a solution of a chromium salt and subsequently immersing in a caustic soda solution of a minimum density of 20° Tw. (sp. gr. 1.10) and allowing the material to lie batched or piled long enough to complete the fixation. If desired, the material, after the treatment with caustic soda, may be subjected, without drying, to the action of steam before batching or piling.—J. F. B.

*Aniline Black upon vegetable fibres; Production of —.* The Calico Printers' Association, Ltd., and E. A. Fourneaux, Manchester. Eng. Pat. 117,987, Aug. 31, 1917. (Appl. No. 12,509 of 1917.)

FOR the production of Aniline Black developed by drying with hot air or "ageing" and after-treatment with sodium chromate, the ingredients mentioned in Eng. Pat. 116,562 (this J., 1918, 463A) are mixed within the limits of the following molecular proportions:—The primary arylamines other than aniline should not exceed 3 mols. out of a total 11 mols. of amines; the proportion of strong mineral acid should not exceed one-half of that required to

neutralise completely the bases used; for 1 mol. of aniline or the equivalent mixture of bases, the boric acid may range from 0.4 to 1.0 mol., the formic acid should not be less than 0.75 mol., the organic catalyst (*p*-phenylenediamine or *p*-aminophenol) not less than 0.03 mol., the soluble copper salt not less than 0.1 mol., and the chlorate between 0.3 and 0.45 mol. Suitable thickening agents are made into a paste and boiled with a little formic acid; the aniline, the acids, and the salts of the bases are then dissolved in the warm thickening, and the sodium chlorate in solution is added after cooling; the copper salt is added only immediately before use.—J. F. B.

*Beam dyeing machine.* E. and L. Holt, Burlington, N.C. U.S. Pat. 1,269,934, June 18, 1918. Date of appl., July 1, 1915.

A PERFORATED beam for holding a quantity of warped or wound yarn is mounted in a closed vat, and liquid is circulated from the inside to the outside of the beam and *vice versa*. Means are provided for introducing compressed air above the surface of the liquid for simultaneously forcing the treating agents from the vat and for oxidising them on the yarn as they escape from the vat; means are also provided for introducing compressed air into the bottom of the beam.—J. F. B.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,271,144, July 2, 1918. Date of appl., Aug. 3, 1917.

WITHIN a receptacle having non-perforated sides are two foraminous plates, spaced apart, each plate comprising a series of extended hollow cones having vertical openings in the sides. A distributing chamber converging downwards is spaced below the lower foraminous plate, and contains a series of parallel curved blades converging downwards and inwards. A pump for forcing liquid through the apparatus is connected with the lower portion of the distributing chamber and an overflow pipe is provided above the upper foraminous plate. A smaller testing device exterior to the dyeing chamber, similar and proportionate to the larger vessel, is connected with the latter below the lower foraminous plate.—J. F. B.

*Dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,271,549 and (B) 1,271,550, July 9, 1918. Dates of appl., May 16 and June 30, 1917.

(A) A RECEPTACLE contains a series of revolving fibre chambers each having foraminous walls with members extended inwards, and with openings between the members, a foraminous member spaced from the inner wall of the fibre chamber and integral therewith, a series of tubes, each tube of which opens upon a portion of the inner face of the foraminous member and extends outwards through the hollow core of the device, and a second series of similar tubes extending through the core in the opposite direction. Means are provided for maintaining the fibre chambers beneath a liquid within the receptacle, for continuously passing a liquid through the tubes and fibre chambers, for passing air through them, and for revolving the fibre chambers, foraminous member, and tubes. (B) A dyeing chamber contains a series of fibre chambers each comprising a foraminous bottom having non-perforated sides and members extending upwards, and a foraminous top plate to each series of fibre chambers having members extending downwards. There are also a reserve tank, the upper portion of which is connected with the upper and lower portions of the dyeing chamber and the lower portion with a pump, a second dyeing chamber adjacent to the first and connected thereto at the top and the bottom, and means for passing a liquid through the fibre chambers in opposite directions.

—J. F. B.

*Fabrics; Method for sponging, setting, and finishing* — E. Heidler, Garfield, N.J., Assignor to Columbia Conditioning Co., Inc., New York. U.S. Pat. 1,270,153, June 18, 1918. Date of appl., Aug. 9, 1916.

Fabric in the width is unrolled and passed with a continuous travelling motion through the following operations in the sequence stated: brushing both faces of the fabric, heating it by exposure to the action of steam, sponging it with water, heating it by passing round a heated surface, ironing it on both faces by heated cylinders running in the opposite direction to the travel of the fabric, allowing the fabric to accumulate in folds, subjecting the fabric to tension, supplying steam to the fabric, cooling it gradually and supplying it with a natural amount of water by passing it over moistened rollers, spreading it in a transverse direction, and finally winding up the fabric in finished condition.—J. F. B.

*Sericin soap; Manufacture of a substitute for* — [for use in silk dyeing]. Gebr. Schmid, Basle, Switzerland. Eng. Pat. 117,042, Jan. 14, 1918. (Appl. No. 805 of 1918.) Under Int. Conv., Sep. 4, 1917. Addition to Eng. Pat. 100,169, Mar. 12, 1915 (this J., 1916, 734).

SEE Ger. Pat. 305,239 of 1917; this J., 1918, 461A.

*Vegetable dye and process of making same.* U.S. Pat. 1,273,293. See IV.

*Preparation of dilute bleaching liquors from sodium carbonate solution and chlorinc.* Ger. Pat. 306,193. See VII.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid; Manufacture of* — by the ferric oxide contact process. H. Ditz and F. Kanhäuser. Z. angew. Chem., 1918, 31, 149—150, 153—156.

In the manufacture of sulphuric acid by the ferric oxide contact process (this J., 1902, 1137; 1905, 729; 1908, 402) the burner gases are first brought into contact with ferric oxide at a high temperature, 50 to 60% of the sulphur dioxide being converted into sulphur trioxide, whilst the arsenic is retained by the contact mass. The gases are then cooled, and, after absorption of the sulphur trioxide by means of sulphuric acid, are brought into contact with a platinum mass whereby the oxidation is completed. Samples of sulphuric acid deposited in the fan casing of a plant had the following composition:—Sulphuric acid, 97.82 and 97.01; lead sulphate, 1.28 and 1.04; selenium, 0.104 and 0.006; selenium dioxide, 0.161 and 0.058; selenic acid, 0.008 and 0.014; arsenious oxide, 0.014 and 0.007; and sulphur dioxide, 0.012 and 0.064% respectively. Experiments to determine the volatility of lead sulphate showed that when heated with sulphuric acid alone, it was only slightly volatile, but that in the presence of selenium, especially in the elementary form or as  $\text{SeO}_2$ , the volatility was greatly increased. The high proportion of lead in the acid was contrary to recorded statements. According to Lunge 100 parts of sulphuric acid of sp. gr. 1.841 dissolves 0.039 part of lead sulphate, whilst Marshall gives the solubility in 98% acid as 0.09%, but the authors have shown (this J., 1917, 641) that 98 to 100% sulphuric acid is able to dissolve a large proportion of lead. The variations in the proportions of selenium in the acid are attributable to the variable composition of the pyrites or to variations in the working of the plant. The presence of selenium has probably an unfavourable influence on the oxidation of the

sulphur dioxide. The process should convert up to 95% (usually about 92%) of the sulphur burned, but in a plant studied by the authors the yield was sometimes less than 90%. This was probably partly due to the toxic action of the arsenic in the gases upon the platinum contact mass, but it was also possible that selenium and lead compounds in the burner gases had an influence on the activity of the ferric oxide contact mass.—C. A. M.

*Sodium sulphate, ammonium sulphate and water; The ternary system* —. Utilisation of nitre cake for the production of ammonium sulphate. H. M. Dawson. Chem. Soc. Trans., 1918, 113, 675—688.

WHEN finely powdered Glauber's salt and ammonium sulphate are brought into intimate contact at the ordinary temperature, a pasty mass is obtained and the temperature of the mixture falls considerably. This is due to the formation of sodium ammonium sulphate:  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O} + (\text{NH}_4)_2\text{SO}_4 = \text{Na}_2\text{SO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} + 6\text{H}_2\text{O}$ . The lowest temperature at which this reaction occurs is  $-16^\circ\text{C}$ . Above  $59.3^\circ\text{C}$ . the double salt decomposes with the formation of the anhydrous simple salts. The composition of the saturated solutions which characterise the ternary system has been determined at temperatures between  $-19.5^\circ\text{C}$ . (the eutectic temperature) and  $100^\circ\text{C}$ . The solutions which are saturated simultaneously with respect to the double salt and sodium sulphate show a very pronounced minimum in the ammonium sulphate content and a maximum in the sodium sulphate content at  $26.5^\circ\text{C}$ ., which is the temperature at which Glauber's salt is transformed into anhydrous sodium sulphate in presence of the double salt. Although the double salt may be crystallised out from solutions containing the mixed sulphates at any temperature between  $-16^\circ\text{C}$ . and  $59.3^\circ\text{C}$ ., if the ratio between the salts is suitably adjusted, yet the double salt is decomposed by water between  $-16^\circ$  and  $20^\circ\text{C}$ . with the separation of Glauber's salt, and between  $41.5^\circ$  and  $59.3^\circ\text{C}$ . with the separation of anhydrous sodium sulphate. Saturated solutions of the double salt containing equivalent quantities of sodium and ammonium sulphate are only obtainable, in the absence of supersaturation, between  $20^\circ$  and  $41.5^\circ\text{C}$ . In view of the fact that solutions saturated with respect to Glauber's salt and ammonium sulphate (or the double salt) at low temperatures contain a large excess of ammonium sulphate, it is possible to crystallise out a large proportion of sodium sulphate from a solution containing the two sulphates by cooling. If the mother liquor is subsequently evaporated at about  $100^\circ\text{C}$ . pure ammonium sulphate may be obtained so long as the ratio of ammonium sulphate to sodium sulphate in the residual hot liquor does not fall below about 2.7. By repeating the cooling and evaporating processes, complete separation of the sodium and ammonium sulphates may be effected. By the use of this process it is possible to make use of nitre cake for the absorption of ammonia in the production of ammonium sulphate, Glauber's salt being obtained as a by-product—H. M. D.

*Nitre cake; Use of* — in the manufacture of sulphate of ammonia. P. Parrish. Gas J., 1918, 143, 395—396.

IN order to produce a saleable mixed sodium and ammonium sulphate, the method has been successfully adopted at the Stafford Gas-Works of introducing ground solid nitre cake into the acid-charge pot. The following precautions were found necessary for the satisfactory application of the method: The sulphuric acid content of the bath should not



be allowed to fall below 6%, as determined by titration; the nitre cake should be added regularly; frequent tests should be made to ascertain the presence of undecomposed nitrates, which results in corrosion of the lead. As an alternative to the introduction of the solid cake, it is suggested that it might be dissolved in the feed acid in lead-lined tanks, provided the nitre cake is sufficiently pure and the acid free from arsenic. The method adopted at the Llwynpia works of the Glamorgan Coal Co. is to feed a nitre cake solution of 6% sulphuric acid content along with the free acid in certain fixed proportions into the saturator. As soon as deposition of crystals begins in the saturator, as indicated by an excess back pressure, some of the liquor is run into the mother liquor well, where, on cooling to 46° C., practically the whole of the sodium sulphate crystallises out. A further supply of solution is then run into the saturator. The mother liquor containing ammonium bisulphate and small quantities of sodium bisulphate, is again introduced into the cycle of operations. Ammonium sulphate containing 23.8%  $\text{NH}_3$  is obtained as the final product. Trial has been made on a Mond recovery plant at Langwith of the exclusive use of nitre cake for extraction of the ammonia. The cooled gases, freed from tar, are scrubbed in a tower with a 3 to 4% sulphuric acid solution. On reaching the saturation point, the solution is evaporated to crystallise out the ammonium sulphate. The mother liquor, maintained at about 80° C. is used to wash the nitre cake, the conditions being such that very little sodium sulphate goes into solution. The liquor is then used again in the tower, diluted with washings from the sodium sulphate sludge. Ammonium sulphate containing 24.5%  $\text{NH}_3$  is thus obtained.

—L. A. C.

*Chlorine; Determination of — in mixtures containing silicates.* G. Brulins. Z. angew. Chem., 1918, 31, 156.

CHLORINE may be determined by titration with silver nitrate in the presence of dissolved or gelatinous silicic acid (water glass, detergents, etc.), provided that the liquid is made neutral to phenolphthalein with nitric acid and any resulting gelatinous mass is finely distributed by trituration or shaking.—C. A. M.

*Chlorates, peroxides, etc.; Source of error in Bunsen's method for the determination of —.* E. Rupp. Z. anal. Chem., 1918, 57, 226–230.

THE results obtained by this method are from 0.5 to 2% too low when the substance is distilled directly with hydrochloric acid, the chlorine collected in potassium iodide solution, and the liberated iodine titrated. This is due to reaction between chlorine and steam with the formation of hydrochloric acid and oxygen. Better results are obtained when potassium iodide is added to the distillation mixture and the iodine distilled over and titrated, since iodine does not decompose water vapour. The concentration of the hydrochloric acid has an influence, however, and the results in some cases tend to be too high owing to slight decomposition of the iodide by hydrochloric acid. To obtain concordant results the distillation must always be carried out under the same conditions.

—W. P. S.

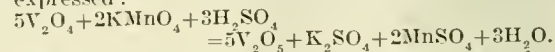
*Nitrates and nitrites; Estimation of —.* W. Strecker. Ber., 1918, 51, 997–1004.

THE solution to be analysed, containing nitrites and nitrates equivalent to not more than 0.11 grm.  $\text{NaNO}_2$  and 0.18 grm.  $\text{KNO}_3$ , is dropped into a boiling, concentrated solution of ammonium

chloride, at least 100 times as much of this salt being taken as there is nitrite present, the solution being in a flask which is connected with an apparatus for making air-free carbon dioxide, on the one hand, and a water-cooled Schiff's nitrometer, containing potassium hydroxide, on the other. After adjusting the pressure and noting the volume of nitrogen, a solution of iron in concentrated hydrochloric acid is introduced and the nitric oxide is collected.—J. C. W.

*Vanadium compounds; Quantitative analysis of — with the aid of carbon tetrachloride.* P. Jannasch and H. E. Harwood. J. prakt. Chem., 1918, 97, 93–137.

VANADIUM in various vanadium compounds can be estimated by heating the latter in a current of carbon dioxide and carbon tetrachloride vapour. The compound is placed in a silica boat inside a silica tube and the resulting volatile vanadium oxychloride and tetrachloride are collected by passage into two receivers containing dilute nitric acid and water respectively. The resulting solution of vanadic acid is evaporated to dryness, the residue being subsequently dissolved in dilute sulphuric acid, treated with a slight excess of potassium permanganate to remove traces of organic matter, and then reduced by a current of sulphur dioxide. After the expulsion of the excess of sulphur dioxide by boiling, the vanadyl sulphate which remains is estimated by titration at 70° C. with potassium permanganate solution. A repetition of the reduction and titration may be made with the same solution. The reaction may be expressed:



Details are given of the conditions necessary for the satisfactory decomposition of various vanadium compounds by carbon tetrachloride. It is also possible to expel phosphoric acid from sodium phosphate by heating a mixture of the latter with sodium chloride or silica in carbon tetrachloride vapour; boric acid and titanate acid can be displaced in a similar manner, but the details necessary to render the process capable of application to quantitative analysis are not yet completed.

—D. F. T.

*Hydrogen peroxide; Neutralisation of — with sodium borate.* J. Cambe and H. Diacono. J. Pharm. Chim., 1918, 18, 12–17.

CERTAIN samples of hydrogen peroxide when mixed even with an excess of sodium borate did not give a red coloration to phenolphthalein. These samples contained hydrofluosilicic acid or were freshly prepared and contained "active" oxygen, and the non-appearance of the red coloration of the phenolphthalein when sodium borate was added was due to the presence of these substances; other indicators were not affected. (See also J. Chem. Soc., Oct., 1918.)—W. P. S.

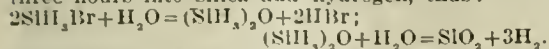
*Bismuth tellurides. II.* M. Amadori. Atti R. Accad. Lincei, 1918, 27, I., 131–134.

THERMAL investigation shows that fused mixtures of  $\text{Bi}$ ,  $\text{Bi}_2\text{S}_3$ , and  $\text{Bi}_2\text{Te}_3$  give no compounds analogous to the natural sulphotellurides, tetradimite, grunlingite, and joseite. (Compare J. Chem. Soc., Oct., 1918.)—T. H. P.

*Silicon hydrides. V. Decomposition of silicon hydrides by water. Action of hydrogen bromide on monosilane.* A. Stock and C. Somleski. Ber., 1918, 51, 989–996.

THE silanes (silicon hydrides) are practically unaffected by water during several days, unless the

glass renders the mixture alkaline, when they decompose in a few hours into hydrogen and silicic acid. Monosilane reacts very readily with hydrogen bromide in the presence of aluminium chloride to give the lower bromides, thus:  $\text{SiH}_4 + \text{HBr} = \text{SiH}_3\text{Br} + \text{H}_2$ , and  $\text{SiH}_4 + 2\text{HBr} = \text{SiH}_2\text{Br}_2 + 2\text{H}_2$ . A mixture of monosilane, hydrogen bromide, and bromomonosilane can be analysed by means of water. The last-named reacts immediately to form the gas disiloxane, which decomposes after two or three hours into silica and hydrogen, thus:



If the volume of the mixture is  $a$ , the volume after the initial contraction is  $b$ , and the final volume is  $c$ , then  $c - b$  gives the volume of  $\text{SiH}_3\text{Br}$ , and  $a - b - \frac{1}{2}(c - b)$  the volume of  $\text{HBr}$ . (See further J. Chem. Soc., Oct., 1918.)—J. C. W.

*Catalysts; Influence of foreign substances on the activity of —.* V. *Experiments with palladium hydrosol in the presence of the hydroxides of iron, copper, and zinc.* C. Paal and W. Hartmann. Ber., 1918, 51, 894–906. (Compare this J., 1918, 560 A.)

The hydroxides mentioned are peptised by sodium protalbate but differ in their influence on the activity of palladium hydrosol, protected by this colloid, in the union of hydrogen and oxygen. Ferric hydroxide has no action at all. Precipitated copper hydroxide lowers the activity of the catalyst but large quantities would be necessary to spoil it altogether; colloidal copper hydroxide actually causes an increase in the activity of the palladium at first, but this is followed by a rapid decrease. Zinc hydroxide depresses the activity during the first hour to about one-half, but a remarkable recovery ultimately takes place, and the catalyst actually becomes more than normally active. (See further J. Chem. Soc., Oct., 1918.)—J. C. W.

*Iodine; Enrichment of — during its purification.* F. Lenzi. Boll. Chim. Farm., 1918, 57, 121–123.

In the purification of iodine, sublimation removes most of the cyanogen iodide—which sublimes first—and also of the bromine and moisture. Chlorine and bromine are eliminated by sublimation in presence of potassium iodide. The loss of iodine accompanying these processes may be avoided as follows (see Tarugi, this J., 1918, 284 A). When qualitative analysis indicates the presence of iodine monochloride and iodic acid, the water used for washing the iodine is treated with an acid (sulphuric) solution of ferrous sulphate. If, however, cyanogen iodide is present, the washing water is first left in contact with a sufficient quantity of iodine monochloride for 24 hours and then treated with the acid ferrous sulphate in the usual way. If the iodine contains iodine bromide, this is converted into hydrobromic acid and iodine by the ferrous sulphate.—T. H. P.

*Effect of the iron content of ammonium persulphate on its photographic reducing power.* Sheppard. See XXI.

#### PATENTS.

*Sulphuric acid; Manufacture of — from nitre cake.* F. A. Freeth, Northwich. Eng. Pat. 117,649, June 29, 1917. (Appl. No. 9347 of 1917.)

NITRE cake, calcium sulphate, and water are mixed in suitable proportions (e.g., 689:180:313) and heated to 50° C. with constant stirring until a solution saturated with sodium bisulphate, the double salt  $\text{CaSO}_4 \cdot \text{Na}_2\text{SO}_4$ , and calcium sulphate is produced. The precipitated double salt containing some calcium sulphate is separated and the

solution cooled to 25° C. until sodium bisulphate separates out. This is removed and the solution is concentrated until it contains 70–75% of sulphuric acid and is then cooled to 25° C., the remaining sodium sulphate being deposited as  $\text{Na}_2\text{SO}_4 \cdot 3\text{H}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ . After separation of this substance the solution contains 75–80% of sulphuric acid with less than 1% of sodium sulphate. The sodium bisulphate and the  $\text{Na}_2\text{SO}_4 \cdot 3\text{H}_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$  are treated with more calcium sulphate and the cycle of operations repeated. The double salt  $\text{CaSO}_4 \cdot \text{Na}_2\text{SO}_4$  is decomposed by extracting it with hot water, the sodium sulphate going into solution and the greater part of the calcium sulphate remaining insoluble. Temperatures much higher or lower than 50° C. are unfavourable to the main reaction.—A. B. S.

*Sulphuric anhydride and the like; Apparatus for the manufacture of —.* E. L. Wilke, Buffalo, N.Y. U.S. Pat. 1,270,997, July 2, 1918. Date of appl., Oct. 4, 1916.

SULPHUR dioxide gas is passed tangentially into an outer annular chamber at a point midway of its height, and passes into an inner chamber containing a heated contact material, through a number of passages opening into the upper end of the outer chamber and into the lower end of the inner contact chamber. Dust separates from the gas in the outer chamber. The outlet for gases leaving the inner contact chamber is provided with a damper to control the pressure in the chamber, the volume of gas passing being kept constant.

—A. B. S.

*Metallopyrophosphoric acids or their salts associated with boron compounds or containing boron; Manufacture of — and the application of the products in tanning.* B. Levin, Hale, Chester. Eng. Pat. 117,693, Aug. 3, 1917. (Appl. No. 11,224 of 1917.)

METALLOPYROPHOSPHORIC acids containing boron or their salts may be made by heating pyrophosphoric acid (Eng. Pat. 116,735; this J., 1918, 416 A) or a mixture of boric acid and pyrophosphoric acid with a compound of a suitable metal such as chromium, aluminium, iron, tungsten, or manganese at a temperature not exceeding 300° C. For example, a chromium salt, such as chrome alum, is mixed with boric oxide and commercial orthophosphoric acid in the proportions Cr : B :  $\text{P}_2\text{O}_5$  = 52 : 44 : 600 and heated until a heavy mobile mass is obtained and the temperature exceeds 250° C. The product is poured on to trays which are heated until a substantially anhydrous mass is obtained. The products are less hygroscopic and more stable than the metallopyrophosphates previously known. In tanning, the use of a metallopyrophosphoric acid containing boron and a metal known to have a tanning action is advantageous, because it is more soluble, and has a greater penetrative power than ordinary pyrophosphates and may be rendered insoluble in the pores of the hides by neutralising the acidity of the tan-liquor.—A. B. S.

*Potassium compounds; Manufacture of —.* S. R. Scholes, Beaver, Pa., U.S.A. Eng. Pat. 117,755, Nov. 5, 1917. (Appl. No. 16,157 of 1917.)

FELSPAR or other potash-bearing mineral is reduced to a fine powder and mixed with sufficient hydrated potassium or sodium carbonate to supply one equivalent of  $\text{K}_2\text{O}$  or  $\text{Na}_2\text{O}$  to every two equivalents of  $\text{SiO}_2$ . The mixture is fused, poured into water, and then ground to powder. The product is boiled with water, and crude carbon dioxide or waste gas rich in carbon dioxide is passed through.



the mixture until the amount of dissolved alkali has reached a maximum. The liquid is filtered, the filtrate evaporated, and the potassium and sodium carbonates recovered. When sodium carbonate is used, an amount of the evaporated product equal in weight to the sodium carbonate employed may be added to a fresh quantity of rock and the process repeated. By this means, the proportion of potassium carbonate in the evaporated product will be increased from about 20% to about 40%. This re-use of part of the evaporated product may be repeated five or six times until most of the sodium present has been eliminated, after which only potassium carbonate is added to fresh charges. Alternatively the mixed sodium and potassium carbonates may be separated by evaporating the solution until a concentration is reached at which most of the sodium carbonate crystallises out. The filtered solution is then evaporated to dryness and the product used for treating fresh quantities of potash-bearing mineral as previously described. An important feature of the process is the removal at the end of each cycle of a quantity of mixed carbonates equal to the amount of potassium carbonate extracted from the rock.—A. B. S.

*Potassium salts; Process for the recovery of — from blast furnace flue dust or the like.* Stafford Coal and Iron Co., Ltd., and J. R. Richmond, Stoke-on-Trent. Eng. Pat. 117,991, Sept. 8, 1917. (Appl. No. 12,887 of 1917.)

BLAST-FURNACE flue dust is extracted with hot water in a suitable apparatus, e.g., a hydro-extractor, steam-heated tanks, etc., and the filtered solution is blown with air and treated with lime until it shows an alkaline reaction, whereby iron, manganese, and magnesium are precipitated, after which it is evaporated to dryness. The residue is baked, thus removing any soluble organic matter and rendering the iron insoluble, re-dissolved in water and filtered. The clear solution is evaporated until crystallisation begins, when it is allowed to cool. High-grade potassium salts crystallise out, and, after separation of the crystals, further quantities of crude salts may be obtained by evaporation of the mother liquor. Ammonia may be recovered, either as the chloride by heating the dust before extraction, or, as gas, when lime is added to the liquor.—L. A. C.

*Potassium aluminate, alumina, and potassium carbonate; Manufacture of —.* E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 118,155, Aug. 18, 1917. (Appl. No. 11,887 of 1917.)

A POWDERED mixture of potassium chloride (450 parts), bauxite or any aluminous substance (e.g., calcined bauxite containing about 70%  $\text{Al}_2\text{O}_3$ , 400 parts), calcium hydroxide (222 parts), and coke (40 parts) is heated to redness in a retort or muffle, and 133 parts of arsenic trioxide vapour is introduced. Arsenic trichloride, potassium aluminate, hydrogen, and calcium carbonate are produced. The potassium aluminate is removed by lixiviation and aluminium hydroxide precipitated from the solution by passing in carbon dioxide. The potassium carbonate solution remaining after filtration of the alumina is evaporated to dryness.

—J. H. P.

*Sodium aluminate, alumina, and sodium carbonate; Manufacture of —.* E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 118,156, Aug. 18, 1917. (Appl. No. 11,888 of 1917.)

In the process described in Eng. Pat. 118,155 (see preceding abstract) sodium chloride (351 parts) is used in place of potassium chloride.—J. H. P.

*Borates; Method of separating — from their gangue.* H. W. Faulkner, Ryan, Cal., Assignor to Pacific Coast Borax Co., Reno, Nev. U.S. Pat. 1,269,170, June 11, 1918. Date of appl., June 26, 1917.

A MIXTURE of borate and gangue is dehydrated by heat to such a degree that the specific gravity of the borate is altered, and the two components are then separated, if necessary after crushing, by subjecting the mixture to a jigging operation, dependent on the specific gravities of the components.

—E. W. L.

*Aluminium chloride; Process of making —.* D. F. Smith and H. Essex, Assignors to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,270,226, June 18, 1918. Date of appl., June 22, 1917.

A MATERIAL containing alumina is heated with carbon in an electric furnace so as to form aluminium carbide and dissociated aluminium carbide, and the product is treated with a gas containing chlorine, under such conditions that aluminium chloride is produced, vaporised, and condensed in another part of the apparatus used. No external heating is required for the conversion of the carbide into chloride.—A. B. S.

*Ammonia; Apparatus for the catalytic oxidation of —.* M. Tallani, Isola Liri, Italy. U.S. Pat. 1,270,989, July 2, 1918. Date of appl., Apr. 26, 1918.

THE catalytic material is placed in the middle of a chamber comprising two funnel-shaped chambers united at their larger ends. Air and ammonia are admitted at one of the smaller ends and are given a whirling motion which mixes them and distributes them uniformly over the catalyst. A cooling coil may be fitted inside the apparatus, close to the catalyst. A valve is provided for controlling the pressure in the chamber.—A. B. S.

*Ferrie phosphates; Process of producing —.* W. H. Allen, Detroit, Mich. U.S. Pat. 1,271,002, July 2, 1918. Date of appl., Sep. 25, 1916.

FINELY divided currents of air are passed through an aqueous solution of ferrous phosphate and phosphoric acid so as to oxidise the ferrous salt to ferrie phosphate.—A. B. S.

*Perechloric acid; Method of electrolytic production of —.* E. C. Walker, Brookline, Mass. U.S. Pat. 1,271,633, July 9, 1918. Date of appl., Apr. 23, 1917.

A SOLUTION of hydrochloric acid, of strength not greater than N/1, is subjected in successive stages to electrolytic action with a current-density of 0.5 amp. per sq. cm., and at a temperature not materially exceeding 50° C. in the initial stage, the temperature being reduced considerably in a subsequent stage. A flow of electrolyte is maintained through the initial stage, to and through the subsequent stage, and the perchloric acid solution so produced is finally concentrated.—L. A. C.

*Ammonium perchlorate; Method of producing —.* G. C. Given, Tamaqua, Pa., Assignor to Atlas Powder Co., Wilmington, Del. U.S. Pat. 1,273,477, July 23, 1918. Date of appl., Sep. 10, 1917.

AMMONIUM perchlorate is produced by the interaction of a soluble perchlorate such as sodium perchlorate, with carbon dioxide in presence of ammonia, or by the action of ammonium bicarbonate on a soluble perchlorate.—J. H. P.

*Silicon tetrachloride; Method for the production of* — O. Hutchins, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,271,713, July 9, 1918. Date of appl., Oct. 30, 1917.

Dry chlorine is passed through a mass of silicon carbide heated electrically to at least 1000° C.

—L. A. C.

*Sulphur dioxide from furnace gases; Method of recovering* — H. Howard, Brookline, and F. G. Stantlal, Melrose, Mass. U.S. Pat. 1,271,899, July 9, 1918. Date of appl., May 12, 1917.

Non-gaseous substances, e.g., sulphur trioxide, are precipitated from the furnace gases, and the residual gases scrubbed with a dilute alkaline solution. The solution is then treated with lime, producing calcium sulphite and an alkaline solution, which is used for the treatment of further quantities of the gas.—L. A. C.

[*Rare-earth fluorides;*] *Purification of precipitates [of —]*. D. Burns, Lakewood, Ohio, Assignor to National Carbon Co. U.S. Pat. 1,272,375, July 16, 1918. Date of appl., Sep. 19, 1914.

Precipitated fluorides of rare-earth metals containing also sulphates of rare-earth metals are mixed with sodium fluoride and calcined above 500° C. The sulphates are converted into soluble salts which may be removed by washing.—W. F. F.

*Titanic oxide concentrate and method for obtaining the same*. A. J. Rossi, Niagara Falls, N.Y., Assignor to The Titanium Alloy Manufacturing Co., New York. U.S. Pat. 1,272,855, July 16, 1918. Date of appl., Nov. 27, 1917.

To a solution of titanic sulphate are added an extract of vegetable matter and a sufficient quantity of a calcium compound nearly to neutralise the excess of sulphuric acid. The mixture is boiled and the precipitate separated and dehydrated.

—L. A. C.

*Magnesium oxide; Process for obtaining — from dolomite, etc.* A. M. Mitchell, Tuckaboe, N.Y., Assignor to Mitchell and Grenelle, Inc., New York. U.S. Pat. 1,273,110, July 16, 1918. Date of appl., Mar. 25, 1916.

Ground dolomite is subjected to a tumbling and grinding operation while being heated to about 1000° F. (about 540° C.). The crystalline calcium carbonate is then readily separated from the magnesium oxide which is formed. The magnesium oxide is again calcined at a temperature sufficient to expel any remaining carbon dioxide.

—L. A. C.

*Salt manufacture*. C. L. Weil, Port Huron, Mich. U.S. Pat. 1,273,208, July 23, 1918. Date of appl., Nov. 3, 1917.

Small crystals of salt are first produced by "flashing" the brine, which is then cooled in a closed space provided with disc evaporators so as to produce relatively large crystals of salt, and to build up the small crystals upon the larger ones. By regulating the rate of rotation of the discs during cooling, the proportions of large and small crystals in the product may be controlled.—T. H. B.

*Nitrogen compounds from metal carbides; Process for the production of* — V. Thrane, Christiania, Norway. U.S. Pat. 1,273,690, July 23, 1918. Date of appl., Nov. 10, 1916.

A layer of inert material is fed on to a moving conveyor, and a mass of metal carbide is fed on

to this layer. The moving mass is subjected to a current of nitrogen, and is heated to a temperature sufficient to start a chemical reaction between the nitrogen and the carbide. Once started, the exothermic reaction propagates itself through the mass.

—J. H. P.

*Ammonia; Preparation of synthetic* — C. Ellis, Montclair, N.J., Assignor to C. S. Lutkins, Rye, N.Y. U.S. Pat. 1,273,772, July 23, 1918. Date of appl., May 13, 1916.

A mixture of nitrogen and hydrogen is passed through a catalytic agent comprising nickel-cobalt carbide.—J. H. P.

*Carbon dioxide; Preparation of — from neutral or basic carbonates*. J. Behrens, Bremen, Ger. Pat. 305,417, May 11, 1917.

Neutral or basic carbonates are heated with water to 150° C. or over in closed vessels. The gas evolved is released from time to time and cooled and dried.

—L. A. C.

*Bleaching liquor; Preparation of dilute — from sodium carbonate solution and chlorine*. Deutsche Solvay-Werke Act.-Ges., Bernburg. Ger. Pat. 306,193, Oct. 20, 1916.

A bleaching liquor ready for immediate use, in which between 1 and 2 mols. of combined chlorine are present per 1 mol. of sodium carbonate used, is prepared by passing the requisite amount of chlorine gas into a solution of sodium carbonate; by preparing two solutions in which 1 and 2 mols. of combined chlorine respectively are present per 1 mol. of sodium carbonate used and then mixing the two solutions; or by passing chlorine gas into a solution of sodium carbonate until 2 mols. of chlorine are combined per 1 mol. of sodium carbonate, and then adding the requisite amount of sodium carbonate.—L. A. C.

*Potassium persulphate; Preparation of — by electrolysis*. O. Neher & Co., and O. Nydegger, Mels, Switzerland. Ger. Pat. 306,194, Feb. 7, 1917. Under Int. Conv., Jan. 25, 1917.

Sodium and/or ammonium sulphate or bisulphate is added to the solution of potassium bisulphate used as electrolyte in the preparation of potassium persulphate, the yield thereby being increased 1½ to 2 fold, and a purer product being obtained.

—L. A. C.

*Nitrogen and oxygen gases; Apparatus for the separation of — from liquid air*. G. James, W. T. Vint, and H. Alexander, Leeds. Eng. Pat. 117,937, Aug. 3, 1917. (Appl. No. 11,256 of 1917.)

Inside a wooden casing are two superposed tanks, preferably of equal dimensions, connected by a number of vertical pipes. Above the upper tank, which is open, is a hood provided with a valved outlet pipe passing through the casing, so arranged that it can be lowered on to the tank. At the top and bottom of the casing are adjustable shutters, whereby the passage of air over the apparatus can be regulated. An inlet pipe passes into the upper tank, and from the bottom of the lower tank an outlet pipe, fitted with a valve, leads to a helical pipe, also enclosed in a casing, the latter being open at the bottom, and having on the top an adjustable lid. In order to work the apparatus, the upper shutters and the lid above the helical pipe are closed, the lower shutters being left open. The valve connecting the lower tank with the helical pipe and the valve over the hood are both closed, the hood being raised above the upper tank.



Liquid air is poured into the upper tank, and the vapours at first formed flow out of the top of the tank over the apparatus, displacing the warmer air in the casing, which escapes through the lower shutters. As soon as the liquid air ceases to vaporise, the valve connecting the lower tank with the helical pipe is opened, and as soon as the helical pipe has cooled down to the temperature of the liquid air the hood is lowered and the valve above it opened. On opening the upper shutters and the lid of the casing enclosing the helical coil, a downward current of air through the casings evaporates the liquid air in the apparatus, nitrogen gas at approximately  $-191^{\circ}\text{C}$ . passing out through the valve above the hood and oxygen gas at approximately  $-183^{\circ}\text{C}$ . at the upper end of the helical pipe. Two heat-interchangers, each consisting of a helical coil of pipe having a pipe of smaller diameter throughout its interior, may also be attached to the apparatus, one replacing the helical coil. Nitrogen gas at  $-191^{\circ}\text{C}$ . and liquid oxygen at the same temperature are caused to pass upwards respectively through the inner tubes and air at ordinary temperature is forced into the upper end of the outer tube. Liquid carbon dioxide and oxygen are tapped off at the points at which the temperature reaches  $-90^{\circ}\text{C}$ . and  $-183^{\circ}\text{C}$ . respectively, and nitrogen gas at  $-191^{\circ}\text{C}$ . issues from the lower end.—L. A. C.

*Sulphur; Process for recovering* —. A. F. Hoffman, Pittsburgh, Pa. U.S. Pat. 1,273,370, July 23, 1918. Date of appl., Apr. 8, 1916.

Pyritic ore is heated to drive off sulphur as sulphur dioxide, which is passed into a heated container where it is burned with a gaseous hydrocarbon to produce sulphur, carbon dioxide, and water. The container may be heated by open flame gas jets, and a catalyst, such as a mixture of calcium sulphide and sulphite, may be used.

—J. H. P.

*Bleaching-powder; Manufacture of* —. J. W. Moore, Runcorn, Assignor to Castner-Kellner Alkali Co., Ltd., London. U.S. Pat. 1,272,880, July 16, 1918. Date of appl., Feb. 5, 1918.

SEE Eng. Pat. 113,457 of 1916; this J., 1918, 206 A.

*Nitrogen; Production of* — from the air. C. Andreucci, Rome. U.S. Pat. 1,272,181, July 9, 1918. Date of appl., Nov. 6, 1917.

SEE Eng. Pat. 117,333 of 1917; this J., 1918, 507 A.

*Recovery of cyanogen bearing compounds from gases obtained from coal or other carbonaceous fuel.* Eng. Pat. 109,254. See IIa.

*Fuel from waste materials and making and utilising the same.* Eng. Pat. 117,921. See IIa.

*[Electrolytic] process of making combustible gas.* U.S. Pat. 1,273,050. See IIa.

*Method of treating waste pickle liquor. Process of producing by-products from waste pickle liquor.* U.S. Pats. 1,269,441 and 1,269,442. See X.

## VIII.—GLASS; CERAMICS.

*Optical glass; Special pots for melting* —. A. V. Bleininger. J. Amer. Ceram. Soc., 1918, 1, 15–24.

SATISFACTORY pots were made from a mixture of 25% of kaolin, 25% of plastic bond clays, 7% of

felspar, and 43% of "a calcine" made by firing a mixture of 80% of equal parts of North Carolina, Florida, and Georgia kaolins, 10% of flint, and 10% of felspar at cone 14. Waste bisque of white ware potteries using a body composed of kaolin 35%, ball clay 15%, felspar 14%, and flint 36% is an efficient substitute for the above-mentioned "calcine" if not too finely ground. The following mixture is used for hand-made glass pots at the U.S. Bureau of Standards: White ware bisque (through No. 10-mesh) 35%, grog from old glass pots (through 10-mesh) 10%, felspar 3%, flint 4%, Tennessee ball clay No. 5 15%, Illinois bond clay 5%, kaolin 28%. The materials are mixed and then tempered in a wet pan with the rollers  $\frac{1}{4}$  in. above the pan so as to reduce grinding to a minimum. The paste is passed through a vertical pug-mill and stored as long as possible, but not less than one month. The pots are built up by hand in the usual manner and allowed to dry for about four weeks. For pots made by casting, the body consists of white ware bisque 48%, plastic bond clay 23%, kaolin 24%, felspar 5%, made into a slip containing 80% of solids by means of an aqueous solution of 0.20% (on the dry weight of the body) of a mixture of equal parts of sodium carbonate and sodium silicate. The core of the mould must be held firmly in position by means of cross beams and tie-rods. The slip is supplied to the mould through three funnels arranged symmetrically around the circumference. Absorption is complete after about 16 hrs., and the core may then be removed. The outer mould may be removed after 24 hrs. The cast pots may be dried easily in three weeks. Previous to use, a new pot should be vitrified by heating in the glass furnace to at least  $1400^{\circ}\text{C}$ .; the temperature is then lowered and the charge introduced. Unless this is done the pots are liable to corrode unduly. The rods used for stirring the molten glass are made of the same composition as the pots.—A. B. S.

*Temperature control in glass-melting furnaces; Method of* —. C. N. Fenner. Phys. Rev., 1918, 11, 141. J. Soc. Glass Tech., 1918, 2, Abs., 85.

THE use of platinum and platinum-rhodium thermo-elements introduced through the furnace walls proved unsatisfactory, probably owing to contamination of the thermo-elements by furnace gases. Good results have been obtained with a Leeds and Northrup optical pyrometer. In comparative tests at the temperatures ( $1300^{\circ}$ – $1400^{\circ}\text{C}$ .), at which the important operations of glass-making are performed, the optical pyrometer gave indications agreeing almost exactly with those furnished by a thermo-junction placed in a porcelain tube introduced into the furnace, thus showing that the radiation given out by the walls of the glass furnace corresponded with black-body radiation.

*Alumina; Influence of* — on the fusibility of glasses. F. Singer. Keram. Rundsch., 1917, 25, 142. J. Soc. Glass Tech., 1918, 2, Abs., 53–55.

IN continuation of previous work (this J., 1917, 873), the effect of adding increasing quantities of alumina (from 0.05 mol. to 0.2 mol. in steps of 0.05 mol.) to two glasses of the formulae  $0.5\text{Na}_2\text{O}$ ,  $0.5\text{CaO}$ ,  $3\text{SiO}_2$  and  $0.4\text{Na}_2\text{O}$ ,  $0.6\text{CaO}$ ,  $3\text{SiO}_2$  respectively was studied. In both cases the addition of alumina rendered the melts more fusible, the effect being marked even with only 0.05 mol.  $\text{Al}_2\text{O}_3$  and increasing with the quantity of alumina added. The results were the same whether the alumina was added as aluminium hydroxide or as kaolin. A suitable source of alumina is pegmatite, which is comparatively cheap and also introduces some of the required alkali at the same time. Alumina

(0.15 mol.) was found also to increase the fusibility of a glass of the composition,  $0.2\text{Na}_2\text{O}$ ,  $0.3\text{K}_2\text{O}$ ,  $0.1\text{MgO}$ ,  $0.2\text{CaO}$ ,  $0.1\text{SrO}$ ,  $0.1\text{BaO}$ ,  $3\text{SiO}_2$ . It is probable that in the case of other types of glass also, e.g., lead crystal, borate, or phosphate glasses, the fusibility would be improved by addition of alumina, but the limiting quantities of alumina would vary with each particular type of glass. In the case of heavy lead glasses a small quantity of alumina considerably improves the resistant qualities of the glass without having any very marked effect on the fusibility.

*Selenium as a decolorising agent [in glass].* W. Frommel. *Keram. Rundsch.*, 1917, 25, 95. *J. Soc. Glass Techn.*, 1918, 2, Abs., 51–52.

GLASSES decolorised by means of selenium are more brilliant than similar glasses decolorised by means of manganese or nickel oxides, but acquire a brownish tinge on prolonged exposure to bright sunlight. The decolorising action is due to the presence of minute particles of selenium which tend to give a red colour to the glass and thus neutralise the faint green coloration due to iron. Selenium is readily oxidised, and hence can be used with advantage only in the absence of oxidising agents (nitre, etc.); preferably arsenic trioxide is added as a reducing agent. When sodium selenite is used, this is decomposed with liberation of selenium dioxide, and the latter is reduced to selenium either by the arsenic trioxide or by the furnace gases. The colouring effect of 1 kilo. of iron oxide can be neutralised by 0.208 gm. of selenium. For ordinary glass of good quality 0.1 gm. of selenium or 0.2 gm. of sodium selenite may be used for 250 kilos. of glass. The portion of the glass batch containing the selenium should be added to the pot at the second filling on. Glass decolorised by selenium may usually be recognised by its brilliance and by the reddish tinge it acquires on prolonged heating.

*Glass; Structure and properties of —.* G. Quincke. *Ann. Physik*, 1915, 46, 1025. *J. Soc. Glass Techn.*, 1918, 2, Abs., 61–62.

THE author applies his "foam wall" theory of the structure of solids (compare this J., 1905, 1239; 1906, 932) to explain the physical and mechanical properties of glass. It is considered that during the cooling of glass there separate, at short intervals, thin invisible layers of very viscous, oily liquids, rich in silicic acid. These liquids each contain several allotropic modifications of silicic acid, the concentrations varying in the different layers. In consequence of surface tension effects at the boundaries between the different layers, "foam cells" enclosed by "foam walls" are produced, and the properties of the glass depend largely upon the character (size and shape) of the "foam walls" and the number of such "foam walls" in unit volume.

*Ceramic ware; Effect of gravitation upon the drying of —.* E. W. Washburn. *J. Amer. Ceram. Soc.*, 1918, 1, 25–34.

A PIECE of moist clay weighing 10 grms. and containing 30% of water was suspended by a wire in a stoppered bottle containing a little water, the clay being about 6 cm. above the water. When kept at a constant temperature the clay gradually lost water, at the rate of about 30 mgrms. per day, during eleven days. The clay was then lowered so as to be at the same level as water contained in an annular vessel inside the bottle. The clay then increased in weight. The author suggests that in the first part of the experiment the water distilled out of the clay under the influence of gravitation, and that it returned to the clay in the second experiment. It is concluded that in order to keep clay

moist by enclosing it in a chamber containing a vessel of water, the latter should not be at a lower level than the clay. Cloths hung around the sides of the chamber with their lower ends in a vessel of water and kept wet by capillary action will not prevent the drying of the clay by gravitation (as in the first experiment), as the water held up by capillary force is under tension and therefore at a low vapour pressure, which is identical with that at the surface of the water in the vessel. The behaviour of the clay is analogous to that of a gel which has been soaked in water until swelling has reached a maximum (Schroeder: *Proc. Amsterdam Acad.* 1913, 15, 1078; 1914, 17, 92). In such cases Wolff and Buchner (*Z. physik. Chem.* 1912, 89, 271) found that when precautions were taken to prevent the action of gravitation acting differently on the gel and the water, no drying occurred. If the rate of drying is determined by the difference,  $\Delta p$ , between the vapour pressure of the water in the clay and the partial pressure of water in the surrounding air at the height,  $h$ , above the surface of the water, and if  $D$  is the density of saturated water vapour at the temperature of the clay and water, and  $g$  the acceleration due to gravity,  $\Delta p = gDh$ . The rate of drying by gravity would then vary, as the temperature and a gravity dryer filled with saturated steam at  $100^\circ\text{C}$ . would dry more rapidly than the same dryer at  $0^\circ\text{C}$ . Hence, the rate of drying in a gravity dryer is determined by the vapour pressure gradient and not by the dryness of the air in the dryer, and, therefore, the more moisture there is in the air of the dryer the more rapid will be the drying.

In the discussion, A. S. Watts pointed out that potters keep clay cups moist indefinitely in a chamber with a bucket of water at the bottom, which appears to contradict the author's conclusions, but suggested that there might be a circulation of air in the potter's cupboard owing to the temperature not being kept uniform as in the author's experiments. Linbarger stated that he had found that there is more evaporation in a dryer with a relative humidity of 90% than when the humidity is 65%—a paradox which may possibly be explained by the action of gravity.—A. B. S.

*Kiln; Test of a producer gas-fired periodic —.* C. B. Harrop. *J. Amer. Ceram. Soc.*, 1918, 1, 35–64.

THE kiln was fired by means of a gas-producer of the round pressure type, 10 ft. 6 in. diam., with a double system of blast pipes delivering steam and air to both the bosh and the centre tuyère. The maximum rate of gasification was 1250 lb. of coal per hr., steam at 25 lb. gauge pressure being supplied at the blower. The gas had a water-gauge pressure of 0.14 in. and an average temperature of  $580^\circ\text{C}$ . The fuel used was an Indiana "nut" coal charged at intervals of 25 mins. Air for combustion of the gas was supplied by a No. 9 centrifugal blower through a 12 in. pipe which encircled the kiln near the top of the wall, with 4 in. pipes leading to the burners. The air pressure was 0.95 in. water gauge. The kiln was a typical one of the round downdraught type, 30 ft. diam., with 8 furnaces and a perforated floor. An Underwood's patent gas-burner was fitted to each furnace. The heating was started with four burners for 12 hrs., after which the remaining burners were lighted. For the first 12 hrs. a brick was laid across each 4 in.  $\times$  15 in. opening above the top of the hob in each furnace; 18 hrs. after starting two bricks were laid across these openings; after 30 hrs. the openings were blocked up completely. The burning was completed in  $95\frac{1}{2}$  hrs., a temperature of  $995^\circ\text{C}$ . being first attained, after which it was slowly increased during 24 hrs. The



contents of the kiln consisted of 9522 common and 62,184 facing bricks made by the stiff plastic process and weighing just under  $5\frac{1}{2}$  lb. each; they were set in the usual manner in 2-brick benches. The bricks settled 11 in. in a total height of 14 ft., the gas being shut off as soon as this amount of settlement was reached. The heat expenditure and losses were: In producer and flues, 13.2%; in flue gases, 28.8%; in moisture and waste gases, 0.3%; consumed in dehydration of clay, 2.2%; in ware at end of burning, 20.6%; in kiln structure above ground level at end of burning, 6.3%; in kiln bottom at end of burning and loss by radiation and convection, 28.6%, of which only about 15% was due to radiation and convection. The coal consumption averaged 989 lb. per 1000 bricks. Owing to the loss of heat in the producer and flues, the use of producer gas is less efficient than the direct combustion of coal, but advantages are gained through the centralisation of coal and ash, the greater cleanliness of the yard, the reduced space required for the kilns, the greater control of the combustion, the more rapid burning and the saving in labour when more than one burner is employed. Either oxidising or reducing conditions to any degree can be obtained as desired, and the change from one to the other may be made rapidly. The producer gas has a lower flame temperature than coal burned direct, but temperatures up to  $1150^{\circ}\text{C}$ . and possibly higher may readily be obtained.—A. B. S.

*Refractory bricks; Testing and inspection of* —. C. E. Nesbitt and M. L. Bell. Amer. Soc. for Testing Materials, June, 1918. Blast Furnace and Steel Plant, 1918, 6, 341–345. (See also this J., 1917, 1048, 1049.)

Tests should be made during manufacture to ensure uniformity in grinding, drying, and burning. Silica bricks are not usually subjected to slugs, and for them a spalling and a hot crushing test are sufficient, but fireclay bricks should also be subjected to slag penetration tests. The authors do not favour the hot crushing test for fireclay bricks, but prefer the load or ball impression test. In addition, all bricks should be carefully inspected, and cracked, warped, poorly moulded, off-sized, and badly burned bricks should be eliminated. Serious lack of uniformity in the products of a single works is shown by the great variations in the results of spalling and crushing tests of individual or successive deliveries. The present methods of manufacture do not produce a uniform product, and specifications should, therefore, be used by purchasers.—A. B. S.

*Baryta glazes; Experiments on* —. H. Fritz. Sprechsaal, 1918, 51, 51. Z. angew. Chem., 1918, 31, Ref., 239.

MIXTURES of baryta and quartz, or baryta, alumina, and quartz, do not melt at Seger cone 4a–6a ( $1160^{\circ}$ – $1200^{\circ}\text{C}$ ). The addition of felspar causes superficial sintering, but not fusion. On addition of zinc in the presence of at least 0.6BaO, and an "acidity" not less than  $0.1\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ , good glazes are formed at this temperature; these have a pale blue tint which produces a good white on a stoneware body, and may render the use of cobalt unnecessary.—A. B. S.

*Zinc sulphide; Use of* — in white and luminous enamels. J. Schaefer. Keram. Rund., 1917, 28, 75. J. Soc. Glass Techn., 1918, 2, Abs., 50.

Zinc sulphide may be used with excellent results as an opacifying agent in enamels for sheet iron, provided metallic oxides capable of decomposing it are not present. The opacity, which is due to the sulphide as such and not to zinc oxide produced by oxidation, is equal to that obtained with tin

oxide, but the zinc sulphide enamels have not the whiteness and brilliancy of tin enamels. Zinc sulphide should not be used in coloured enamels or in those containing oxides of lead, arsenic, antimony, or bismuth. Enamels in which luminous zinc sulphide has been used remain unaltered or with only a very slight loss of luminosity for years.

#### PATENTS.

*Glass for absorbing ultra-violet radiations; Manufacture of* —. R. Haddan, London. From Corning Glass Works, Corning, N.Y., U.S.A. Eng. Pats. (A) 118,397 and (B) 118,398, Apr. 15, 1918. (Appl. Nos. 6398 and 6399 of 1918.)

A GLASS for absorbing ultra-violet rays is made by adding to an ordinary lime, barium, lead, or zinc glass batch: (A) at least 6% of titanium dioxide, an oxidising salt (nitre) in sufficient quantity to prevent the reduction of the  $\text{TiO}_2$ , with or without cerium oxide and/or vanadium oxide; or (B) at least 1% of a higher oxide of vanadium than  $\text{V}_2\text{O}_5$ , with or without an oxidising agent (nitre) and a complementary, but absorptively inert substance ( $\text{MnO}_2$ ,  $\text{CoO}$ ,  $\text{NiO}$ ,  $\text{UO}_2$ ) to change the hue of the glass from green to amber. Titanium dioxide is about half as effective as cerium oxide. A lime-soda glass containing 7.7%  $\text{TiO}_2$  and 7.7%  $\text{Ce}(\text{NO}_3)_3$  is brilliantly clear yellow, and in plates 2 mm. thick completely cuts off the 0.365 ultra-violet line. A glass containing both  $\text{Ce}(\text{NO}_3)_3$  and  $\text{TiO}_2$  has a more brilliant colour and greater desirable absorption than a glass containing one of these substances superimposed on a glass containing the other. The addition of borax to the glass batch increases the refractive index of the glasses containing  $\text{TiO}_2$ . Reduction of higher vanadium oxides to  $\text{V}_2\text{O}_5$  must be prevented, as the latter does not cut off the ultra-violet rays. A large proportion of borax in the glass containing vanadium makes the colour muddy. A glass containing 1% vanadium oxide completely cuts off the ultra-violet 0.365 line in plates of 4 mm. thickness, and if 2% of vanadium oxide is used the same cut-off is effected in plates 2 mm. thick.—A. B. S.

*Lehr*. W. L. Clause, Assignor to Pittsburgh Plate Glass Co., Pittsburgh, Pa. U.S. Pat. 1,268,217, June 14, 1918. Date of appl., June 15, 1912.

A GLASS-ANNEALING lehr has a bottom wall supported above the ground by several vertical walls spaced apart to permit the passage of air between them. Conduits for heating gases are provided beneath the bottom wall for heating the lehr and a heat-storing arrangement beneath these conduits, with its thickest part adjacent to the front end of the lehr and gradually diminishing in thickness to the other end. Cold air may also be passed through conduits beneath the heat storage element.

—A. B. S.

*Glass; Method of making coloured* —. H. T. Bellamy, Chicago, Ill. Assignor to Western Electric Co., Inc., New York. U.S. Pat. 1,271,652, July 9, 1918. Date of appl., Feb. 5, 1917.

A BATCH of sand, red lead, sodium nitrate, antimony oxide, borax, tin oxide, potash, lime, and gold in suitable proportions is heated to  $2400^{\circ}\text{F}$ . ( $1315^{\circ}\text{C}$ .), cooled, and then reheated until plastic.

—A. B. S.

*Gas and air mixer for glass furnaces*. E. Majot, Maumee, Ohio. U.S. Pat. 1,273,171, July 23, 1918. Date of appl., Aug. 28, 1917.

A COMBINED mixer and heater for a glass furnace consists of two flues—the upper one for hot air and the other for producer gas—with outlets in a vertical plane and a third hot air flue, with a horizontal

outlet close to the lower outlets of the other two flues, and with a shutter for regulating the amount of air passed through this flue.—A. B. S.

*Dryer [for bricks].* C. H. Klein, Chaska, Minn. U.S. Pat. 1,272,676, July 16, 1918. Date of appl., Sep. 17, 1917.

A DRYING chamber is provided with doors at the ends for the introduction of trucks. Each truck carries a number of superimposed trays, fitting closely against the side walls of the chamber, and adapted to hold pallets of bricks spaced apart. Hot air or gas, entering the chamber under pressure just above the lowest tray, travels to an outlet at the top by passing from side to side of the chamber across each tray in succession, the spaces between consecutive trays being in communication by means of ducts alternately in one or other of the two side walls. Other ducts in these walls enable the moist gases to be reinforced by introduction of fresh dry gases at certain stages in their travel.—J. H. L.

*Refractory ceramic article and method of making the same.* A. W. Oliver, Assignor to Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,273,529, July 23, 1918. Date of appl., Feb. 16, 1918.

PLASTIC clay, a refractory material (e.g., carborundum and graphite), water, and tar are mixed until a high degree of plasticity is developed and the mixture is then moulded into the desired form.—A. B. S.

*Muffle kiln; Tunnel — for ceramic products.* A. Krautzberger, Wistritz, Bohemia. Ger. Pat. 306,055, May 17, 1917.

ONE or more externally heated muffles are built at the side of the tunnel. The goods to be heated are placed on cars and pass along the tunnel in the usual manner until they reach one of these muffles, when (by means of an opening opposite the muffle) they are pushed into the latter and remain there until they attain the desired maximum temperature. They are then drawn back on to the cars, are gradually taken through the remainder of the tunnel, and finally pass out of the kiln. The goods only remain in the side muffle for about 10 mins., so that the operation of the kiln is practically continuous. If each part of the tunnel in front of and behind the muffle holds six cars, the goods pass completely through the kiln in 2 hrs.—A. B. S.

*Glass; Apparatus for manufacture of sheet —.* T. J. McCoy, Shinglehouse, Pa., U.S.A. Eng. Pat. 117,194, Oct. 29, 1917. (Appl. No. 15,735 of 1917).

SEE U.S. Pat. 1,246,764 and 1,255,450; this J., 1918, 53 A, 208 A.

*Cane-glass manufacture. Manufacture of glass rods and tubes.* J. T. Fagan, Cleveland, Ohio, Assignor to General Electric Co. U.S. Pat. 1,273,345 and 1,273,346, July 23, 1918. Dates of appl., June 3, 1913, and July 20, 1916.

SEE Eng. Pat. 17,602 of 1913; this J., 1914, 831.

## IX.—BUILDING MATERIALS.

*Anhydrite and dead-burned gypsum; Hydration of —.* A. C. Gill. J. Amer. Ceram. Soc., 1918, 1, 65—71.

IN a wide-mouthed 250 c.c. bottle with a metallic screw cap, but no rubber washer, was placed 50—60 grms. of anhydrite crushed small enough to pass through an 8-mesh sieve. A second similar bottle was charged with several lumps of alabaster which

had been heated for  $3\frac{1}{2}$  hrs. to  $560^{\circ}\text{C}$ ., the lumps being 1 in. or more in diameter, and having an aggregate volume of 60—75 c.c. Both bottles were filled with water and closed on Oct. 23, 1911. Late in 1917 it was found that all the water had evaporated, and that in the first bottle the small fragments of anhydrite were so firmly united by gypsum crystals that they could not be removed without crushing them. Glistening crystals of gypsum were seen in the interstices, and brilliant acicular crystals attaining a maximum length of  $\frac{1}{2}$  in. projected into the free space which had been occupied by the water. About 34% of the original anhydrite had been hydrated into gypsum. The pieces of burned alabaster in the second bottle were covered completely with fine bristling gypsum needles about  $\frac{1}{2}$  in. long, and the inner portion of the mass had set harder than ordinary plaster of Paris. About 87% of the calcium sulphate had been rehydrated, this hydration having been effected much more readily than in the case of anhydrite.—A. B. S.

### PATENT.

*Artificial stone; Process of making —.* P. M. Sharples, West Chester, Pa., Assignor to White Heat Products Co., Plainbrook, Pa. U.S. Pat. 1,273,547, July 23, 1918. Date of appl., Feb. 2, 1918.

ARTIFICIAL stone is made by mixing calcined sand with disintegrated glass, and forming, pressing, and heating the mixture until the glass fuses.

—A. B. S.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron; Reaction between pure carbon monoxide and pure electrolytic — below the A1 inversion.* H. C. H. Carpenter and C. C. Smith. Iron and Steel Inst., Sep. 1918. [Advance copy.] 53 pages.

WHEN electrolytic iron is heated at  $650^{\circ}\text{C}$ . in an atmosphere of carbon monoxide, a substance believed to be  $\text{Fe}_3\text{C}$  is formed:  $3\text{Fe} + 2\text{CO} = \text{Fe}_3\text{C} + \text{CO}_2$ , the reaction probably being facilitated by a preliminary dissociation of the carbon monoxide. It is possible that the stable condition at  $650^{\circ}\text{C}$ . is iron and graphite, the carbide formed being an intermediate metastable product. The presence of sufficient carbon dioxide in the carbon monoxide prevents the formation of the carbide, while hydrogen facilitates it.—F. C. Th.

*Iron-carbon alloys; Formation of graphite in —.* J. E. Hurst. Engineering, 1918, 106, 217—218.

A CONSIDERATION of the various iron-carbon diagrams suggested leads to the conclusion that graphite does not separate out directly from the liquid, but is formed as a result of decomposition of a previously separated carbide. The Upton thermal-equilibrium diagram (this J., 1908, 1114) is considered to possess a considerable amount of truth. The author considers that in cast iron graphite is precipitated from the austenite and not from the cementite. On reheating grey cast iron to  $900^{\circ}\text{C}$ . part of the graphite is re-absorbed, forming austenite, without any visible intermediary formation of carbide. In grey cast irons containing ferrite the latter is deposited after the graphite and around it.—F. C. Th.

*Steel; Influence of hot-deformation on the qualities of —.* G. Charpy. Iron and Steel Inst., Sep., 1918. [Advance copy.] 19 pages. (See also this J., 1918, 469 A.)

FORGING under the hammer or press results in very marked differences in the extension



produced at different points. The effects of such treatment are exceedingly irregular, especially towards the ends of the bars. Rolling produces practically regular deformation throughout the whole length of the billet. Results are given which indicate that the effect of hot working is to produce deterioration in the test-bars cut transversely, and the progressive distortion of the primary dendritic structure as the working is increased is shown. The influence of the direction of the rolling, and hence of these dendrites upon the resistance to impact, is shown by the following results. When the notch was perpendicular to the direction of rolling, the impact value of a certain steel was 13.5 kilogrammetres; when notched at an angle of 45°, 3.4; at an angle of 20°, 1.5; and parallel to the direction of rolling, 1.3.—F. C. Th.

*Steels; Slow contraction of hardened carbon* —. T. Matsushita. Sci. Rep. Tohoku Imp. Univ., 1918, 7, 43—52.

AFTER being quenched, hardened steel is in a highly strained state, the strain subsequently undergoing gradual relief with concurrent generation of heat and contraction (compare Brush and Hadfield, this J., 1917, 505). In well hardened steels the gradual contraction is always observed, but in imperfectly hardened specimens there is a gradual elongation often associated with a subsequent contraction. This effect is attributed to an incomplete A1 transformation by the moderately rapid cooling, the subsequent gradual conversion of austenitic structure to pearlitic being accompanied by an expansion; the contraction is due to the gradual separation of an unstable cementite; if the temperature be raised to 100° C. this separation is greatly accelerated, occupying less than 2 hours, and no subsequent contraction is then observable. The separation of the unstable cementite does not appreciably affect the hardness of the steel, but the separation of the more stable cementite (which is possible at 340° C.) is accompanied by a loss of hardness. Quenching causes a considerable increase in the electrical resistance of steel by increasing the amount of cementite in solid solution, whilst tempering has the reverse effect. The evolution of heat which invariably accompanies the gradual elongation or contraction of quenched steel arises from the heat of transformation, and is not due merely to the yielding strain as was formerly supposed.—D. F. T.

*Hardened steel; Rate of change at 100° C. and at ordinary temperatures in the electrical resistance of* —. E. D. Campbell. Iron and Steel Inst., Sep., 1918. [Advance copy.] 6 pages.

THE rate of change of electrical resistance of four steels containing from 0.57 to 1.05% C, and hardened from 900° C., was determined at ordinary temperatures and at 100° C. A selection of the results (microhms per cm. cube) is given in the following table:—

Treatment	Carbon, per cent.			
	0.57	0.76	0.95	1.05
Quenched from 907° C.	21.83	32.24	36.32	39.73
Tempered 1 hr. at 100° C.	20.67	27.47	29.62	32.97
Tempered 12 hrs. at 100° C.	19.87	25.75	27.31	29.19
After standing at 25° C.:				
For 7 days .. ..	21.17	32.48	38.53	39.87
For 24 months ..	20.60	30.33	32.53	36.47

—F. C. Th.

*Nickel steels; Thermal and electric conductivities of* —. K. Honda. Sci. Rep. Tohoku Imp. Univ., 1918, 7, 59—66.

AN investigation of the thermal and electric conductivities (compare Honda and Simidu, this J., 1918, 125 A) of a complete series of nickel-iron alloys made by fusing commercial nickel with various proportions of low-carbon steel. The curves for the two conductivities follow similar courses, each reaching a marked minimum at 70% Fe and 30% Ni; the ratio of the two conductivities is not constant, but the variation is not great. Specimens once cooled in liquid air show a small maximum and minimum in each curve at 21% and 27% Ni respectively, the curves then declining to the marked minimum already mentioned; specimens once heated to 900° C. do not exhibit a secondary maximum, a slight inflexion forming the only irregularity. Experiments were also made as to the intensity of magnetisation with these alloys.

—D. F. T.

*Hardness [of metals]; Relation between ball hardness and scleroscope* —. A. F. Shore. Iron and Steel Inst., Sep., 1918. [Advance copy.] 20 pages.

UNDER normal conditions of testing it has not been found possible to obtain a satisfactory curve for converting Brinell to scleroscope hardness numbers. If the load to produce a constant indent of 1 mm. diameter is used as a measure of ball hardness, a smooth curve is obtained when this value is plotted against the scleroscope value:—30 on the scleroscope scale then corresponds to 90 tons per sq. in., 60 to 212 tons, 90 to 425 tons. The substitution of a diamond ball under a load of 750 kilos. for the usual hardened steel ball results in much more consistent values being obtained for very hard steels. It is claimed that the scleroscope measures a value closely connected with the elastic limit, the Brinell number being more nearly allied to the maximum stress. By using a small load (750 kilos.) the agreement between the two tests becomes much closer; a load of 3000 kilos. is considered to be far too great.

R. Hadfield, in an appendix, strongly recommends the operation of the Brinell test with variable load so as to give the same size depression (1 mm. diameter), and gives curves and tables showing the relationship of hardness to tensile and compression properties.—F. C. Th.

*Iron alloys; Magnetic analysis as a means of studying the structure of* —. K. Honda. Iron and Steel Inst., Sep., 1918. [Advance copy.] 43 pages.

MAGNETIC analysis is practically free from any time lag, and indicates the existence of critical points in steel at 790° C., 900°—910° C., and 1410°—1420° C., in cobalt at 1150° C., in nickel at 380° C., in cementite at 215° C., in the double carbide of iron and tungsten (see this J., 1918, 421 A) at 400° C., and in magnetite at 580° or 680° C. These changes allow changes in the structure of steel at high temperatures to be investigated. Thus cementite heated to 800° C. or higher partially decomposes and gives free iron, which, with undecomposed carbide, gives the A1 point. About 90% of the Fe<sub>3</sub>C is thus decomposed. The double carbide of iron and tungsten is completely decomposed at 859° C. A very full examination of the tungsten steels is given.

—F. C. Th.

*Zirconium in ferrozirconium and steel; Determination of* —. J. D. Ferguson. Eng. and Min. J., 1918, 106, 356.

ONE grm. of ferrozirconium (or 3 grms. of steel) is dissolved in 25 c.c. of a mixture of 10 c.c. of water,

5 c.c. of nitric acid, and 5 c.c. of hydrochloric acid, and evaporated to dryness. The residue is heated with 1 c.c. of concentrated sulphuric acid and 30 c.c. of water and the silica filtered off and washed successively with boiling water, with 1 c.c. of sulphuric acid diluted with 20 c.c. of water, and again with boiling water. If a residue remains after fuming off the silica with hydrofluoric acid, it is fused with an excess of sodium carbonate, extracted with hot water, and the insoluble sodium zirconate fused with potassium bisulphate. The solution of this fusion is added to the main filtrate from the silica. The combined filtrate is diluted to 400 c.c., heated to boiling, and 6 grms. of sodium thiosulphate for each gm. of alloy is added, with constant stirring. The addition of 1 gm. of sodium phosphate in solution precipitates zirconium as a greenish precipitate, and after standing on a hot plate the precipitate is filtered off without suction, and washed. The precipitate is ignited, and weighed as zirconium phosphate. Iron should be reduced to the ferrous condition, and the solution before precipitation should contain approximately 1% of sulphuric acid. If titanium is present, as noted by the pink colour of the zirconia precipitate, the latter is fused first with sodium carbonate and then with potassium bisulphate as above, and the 1% acid solution is treated with hydrogen peroxide to oxidise the titanium, and the zirconia precipitated as phosphate as described.—C. A. K.

*Copper: Effect of cold work upon the tensile properties of pure —.* W. E. Alkins. Inst. of Metals, Sep., 1918. [Advance copy.] 14 pages.

A HOT-ROLLED bar of copper,  $\frac{7}{8}$  in. in diameter, was cold drawn to 0.04 in., the maximum stress being determined during the process. This increased from 15 to 31 tons, a break in the curve occurring at 23 tons per sq. in., corresponding to a diameter of about 0.35 in. It is suggested that the break is due to an allotropic change.—F. C. Th.

*Copper-zinc alloys: an investigation of some of their mechanical properties.* F. Johnson. Inst. of Metals, Sep., 1918. [Advance copy.] 9 pages.

THE test-pieces were cast in a horizontal mould producing castings 6 in. long by  $\frac{3}{4}$  in. square. It was found that the Brinell hardness was uniform from 45 to 70% Cu; no decided maximum was found. The hardness curve of annealed alloys rich in zinc showed slight irregularities at 5% and at 15% Cu, but no sudden fall followed by a sharp rise. Alloys consisting wholly of the  $\beta$  solid solution were stronger than those containing excess of either  $\alpha$  or  $\gamma$ ; the  $\beta$  solution containing 53% Cu possessed an ultimate tensile stress of 30.4 tons per sq. in. and an elongation of 37.5%, i.e., properties superior to those hitherto observed.—W. R. S.

*Phosphorus in bronzes; Rapid method of estimating —.* T. E. Rooney. Inst. of Metals, Sep., 1918. [Advance copy.] 2 pages.

DRILLINGS of the sample (0.5–2 grms.) are digested with 10 c.c. of strong hydrochloric and 20 c.c. of strong, or, if preferred, 60 c.c. of dilute, nitric acid (sp. gr. 1.135). The digestion must be sufficiently long to oxidise the whole of the phosphorus, but actual boiling and prolonged heating would cause precipitation of tin, which is difficult to re-dissolve. The liquid is diluted, if necessary, to 70 c.c., cooled, and 40 c.c. of ammonia (sp. gr. 0.96) added slowly with constant stirring, followed by 35 c.c. of molybdate mixture. The liquid is shaken for a few minutes, allowed to stand for 1–2 hours, and filtered preferably through a pulp filter. The precipitate is washed with water until free from acid, returned to the flask, and dissolved in N/20 caustic

soda; the excess of the latter is titrated with N/20 sulphuric acid in presence of phenolphthalein; 1 c.c. N/20 NaOH = 0.00337% P on 2 grms.—W. R. S.

*Gun-metal; Influence of impurities on the mechanical properties of Admiralty —.* F. Johnson. Inst. of Metals, Sep., 1918. [Advance copy.] 12 pages.

AFTER summing up the published statements on the influence of impurities on gun-metal, the author describes mechanical tests made with synthetic test-pieces ( $\frac{1}{2} \times 4 \times 3$  in.), both chill-cast and annealed. The work was undertaken with the object of ascertaining which element or elements showed promise of utility in the manufacture of gun-metal castings, and which were detrimental. Tentatively the following conclusions, some of which are at variance with those submitted by other observers, are submitted in tabular form:

Impurity (not exceeding 1%)	Effect on mechanical properties	Effect on fluidity and surface appearance of chill castings
Al	Hardening and embrittling	Good
Si		
Mn	Inappreciable	Bad
As	"	Good
Sb	"	Inappreciable
Fe	Strengthening without loss of ductility	Bad
Ni	Toughening; increase of ductility	Inappreciable

—W. R. S.

*Copper-aluminium alloy; A case of disintegration of a —.* R. Seligman and P. Williams. Inst. of Metals, Sep., 1918. [Advance copy.] 3 pages.

A NUMBER of wire frames forming part of a consignment of scrap aluminium wire netting received for re-melting showed unusual signs of disintegration; the surface of the wire was broken up into loosely adhering, elongated grains uniformly inclined to the axes of the wires. The solid core remaining after removal of the loose grains contained Si 0.33, Fe 0.30, and Cu 2.65%; the copper content of the grains was 2.49%. The bulk of the material was found to contain Si 0.24, Fe 0.22, and Cu 0.07%. The disintegrated wire had apparently been severely overdrawn, for the core broke readily when bent at right angles; on annealing its ductility was restored, showing that the brittleness was not due to disintegration.—W. R. S.

*Aluminium sheet; Annealing of cold-rolled —.* R. J. Anderson. Inst. of Metals, Oct. 11, 1918. [Advance copy.] 18 pages. (See also this J., 1918, 245 A, 424 A.)

FULL annealing of cold-rolled aluminium sheet sufficient for draw-press requirements (i.e., Shore hardness 5 for the annealed metal) can be effected in 1 hour at 370°C. and in 10 minutes at 400°C. The temperature necessary falls as the amount of reduction in rolling is increased. Blistering of the sheets during the annealing is no worse with short heating at 400°C. than with long ones at lower temperatures, and is not affected by the oxidising atmosphere of the furnace. Short annealing at a higher temperature, besides being more economical, resulted in a smaller percentage of defectives in the subsequent drawing.—F. C. Th.

*Tungsten; Metallography of —.* Z. Jeffries. Engineering, 1918, 106, 239–242, 269–275, 300–302.

UNLIKE the majority of metals tungsten in which the crystals are drawn out by cold work, giving a fibrous fracture, is ductile and pliable. When, however, as a result of heating, recrystallisation occurs, the metal is extremely brittle and fragile at the



ordinary temperatures. Ductile tungsten is produced as follows. Tungstic oxide produced from the ore by fusion, lixiviation, and acidification of the solution, is dried and after the addition of 0.75% of thoria made into a paste with thorium nitrate solution. The mixture is dried and heated to 1100° C. for at least an hour to coarsen the particles. The fired oxide is then reduced in hydrogen at 1000° C. The coarse powder produced is compressed, baked in hydrogen at 1300° C., and then electrically sintered at 3000° C. for 10–15 mins. The resulting ingot has sp. gr. 18. The working of the metal is done in a swaging machine with high-speed steel dies. Before this treatment it is electrically heated to 1600° C. in hydrogen and reheated from time to time. Four or five dies are used to form a round rod. As the degree of work on the rod increases, the reheating temperature is lowered gradually to 1300° C., or for wires of less than 0.03 in. diameter to 1100° C. High-speed rolls may be substituted for the swaging dies. The sintered ingot, which is brittle as glass, consists of equiaxed crystals, which are drawn out during working, and the resulting wire can be bent or drawn cold. The tensile strength of the sintered ingot is 18,000 lb. per sq. in. and that of the 0.03 in. swaged wire 215,000 lb., with an elongation of 4% on 2 in. and a reduction of area of 28%. Wires less than 0.03 in. are hot drawn through diamonds; the smallest wires are the strongest. The tensile strength of a wire 0.00114 in. diameter is 590,000 lb. per sq. in., with a reduction of area of 65%. Reheating ductile tungsten to the recrystallisation temperature causes the loss of its ductility, this metal thus behaving in exactly the opposite way from others. Molybdenum acts like tungsten in that it is more ductile in its "coldworked" condition than in the equiaxed state. Tantalum when very pure and melted *in vacuo* is one of the most malleable and ductile of metals, and when annealed *in vacuo* at 1600° C. for 5 mins., the drawn wire recrystallises, becoming, unlike tungsten, much more ductile. When annealed in hydrogen at 1600° C., tantalum becomes very brittle. The effect of thoria on the sintered tungsten ingot is to decrease the grain growth at high temperatures, thus rendering the tungsten capable of being worked hot. More than 2% of thoria renders tungsten difficult to work into fine wires, since the crystals in the sintered ingot are relatively very large; 5% of thoria, however, again gives a finely crystalline structure. Tungsten very readily shows the excessively coarse structure found locally in annealed coldworked iron. With 2.5% of thoria the temperature of coarse grain growth is 3200° C., with 1.5% it is 2900° C., and with 0.75% 2600° C. The finest structure is obtained by reheating the tungsten to just above these "germinative" temperatures, the time required increasing as the temperature becomes lower, e.g., 12 mins. at 3200° C. or 30 mins. at 2600° C. In the presence of carbon at high temperature, W<sub>2</sub>C is formed around the crystals, rendering the metal very brittle. With 0.75% of thoria, the amount generally used, a grain size of 6000 per sq. mm. is aimed at. A fine-grained metal is very hard. The lower the temperature of working the tungsten, the more ductile will it be. The recrystallisation temperature after cold work is 1300° C. for a 99.99% reduction, 1500° C. for 90%, and 2200° C. for 24%, whence the necessity for the working at progressively lower temperatures as the wire becomes finer.—F. C. Th.

*Lead-bismuth-silver and lead-gold-silver alloys.*  
M. Goto. J. Coll. Eng., Imp. Univ., Tokyo. 1918, 9, 63–114.

In the presence of bismuth it is more difficult to concentrate by Pattinson's process the silver in

silver-lead alloys. The mother liquor contains much of the bismuth. A ternary eutectic is present, but no compound is formed. In the silver-gold-lead alloys rich in lead, this metal first separates out, the solution becoming richer in gold and silver. A eutectic of lead and a solid solution of gold and silver then freezes until the residue contains no silver. The mother liquor then contains 15% Au and freezes finally at 208° C. (See further J. Chem. Soc., Oct., 1918.)—F. C. Th.

*Non-ferrous metals; Rapid recrystallisation in deformed —.* D. Hanson. Inst. of Metals, Sep., 1918. [Advance copy.] 5 pages.

A BROKEN, tapered, tensile test-piece of aluminium was annealed for 30 mins. at 480° C. In the neighbourhood of the shoulder, where the strain was least, remarkably coarse crystals were developed. At 450° C. a less coarse recrystallisation resulted after 5 mins. reheating. Magnesium, zinc, lead, and copper gave similar results. An alloy of aluminium with 3% Cu and 20% Zn annealed for 5 mins. at 450° C. also gave a well-marked coarse grain between fine-grained regions. A 70/30 brass annealed at 800° C. for 5 mins. showed signs of a similar critical growth but less well marked.

—F. C. Th.

*Solder without tin.* C. W. Hill. Chem. and Met. Eng., 1918, 19, 170.

OF the lead-cadmium alloys that containing 8 to 9% Cd is the strongest, its freezing range extending from 275° to 248° C. The best flux is a mixture of fused chlorides in the following proportions: Sodium chloride 11%, potassium chloride 14%, zinc chloride 65%, ammonium chloride 10%. The solder will not flow well on copper, and must be maintained at a very even temperature not more than 30° C. above its melting point. The addition of tin, unless in considerable quantities, does not appreciably improve this solder. The addition of 5% of zinc to the cadmium-lead solder with 8.5% Cd gives excellent results applicable to a wide variety of work.—F. C. Th.

*Metals; Resistance of — to penetration under impact.* C. A. Edwards. Inst. of Metals, Sep., 1918. [Advance copy.] 28 pages.

THE diameter (d) of the indent made by a 10 mm. ball striking the sample with energy of 63 in.-lb. is related to the Brinell hardness number (H) by the equation  $H = \frac{7455}{d^3}$ . Further if E is the energy of im-

pact the formula  $d = c \sqrt[3]{E}$ , where c is a constant for each metal, is approximately true especially for the harder metals and alloys. Experiments are recorded with balls and cones on lead, tin, aluminium, copper and iron which partly confirm the conclusion of Martel (Commission des Methodes d'Essai des Mat. de Construction, 1895, 3, Sect. A (Métaux), 261–277) that given a constant energy of impact then for each metal the volume of metal displaced is independent of the shape of the striker. There appears, however, to be some other as yet unknown factor involved. Metals such as tin, cadmium, and zinc in which crystal twinning is produced by rapid stresses show an abnormally high hardness under impact compared with the Brinell hardness figure. The Brinell hardness figures of 31 elements are recorded, and these when plotted against the corresponding atomic weights give a curve which is very similar to that given by the atomic volumes and melting point respectively. The hardness is a periodic function of the atomic weight.—F. C. Th.

*Grain growth in metals.* Z. Jeffries. Inst. of Metals, Sep., 1918. [Advance copy.] 32 pages.

In most metals the lowest temperature at which recrystallisation after cold work occurs is 0.35 to 0.45 of the absolute melting point. The recrystallisation temperature is not fixed and for any particular metal will be lowest when a fine-grained sample has been worked as much as possible at a comparatively low temperature. A metal worked at a temperature above that of recrystallisation is composed, after cooling, of equiaxed crystals which will be the larger the higher was the finishing temperature of working. Reheating such a metal above the finishing temperature usually causes grain growth, which may also occur on prolonged heating to a slightly lower temperature. Allotropic changes may also result in grain growth. The velocity of the growth of the crystals is very great during the first stages of recrystallisation, becoming less as the grains grow larger. Abnormal grain growth produced by annealing after cold work at times attains a maximum at a certain temperature. Higher temperatures result in less growth ensuing. The general laws of grain growth are discussed and typical examples are given, including several in metallic tungsten.—F. C. Th.

*Ilsemanite, a blue water-soluble molybdenum compound occurring in Utah.* H. F. Yancey. Chem. and Met. Eng., 1918, 19, 186—190.

A CONSIDERABLE deposit of molybdenum ore has been found near Ouray, Utah. It is a sandstone containing ilsemanite, a blue compound, soluble in water, to which the formula  $\text{MoO}_3 \cdot 4\text{MoO}_3 \cdot 6\text{H}_2\text{O}$  is given. Lead and vanadium are absent. Analysis of an air-dried sample gave: 65.92%  $\text{SiO}_2$ , 4.35%  $\text{Fe}_2\text{O}_3$ , 12.27%  $\text{Al}_2\text{O}_3$ , 0.35%  $\text{TiO}_2$ , 1.36%  $\text{CaO}$ , 0.50%  $\text{MgO}$ , 1.22%  $\text{MoO}_3$ , 11.09% of sulphur as  $\text{SO}_3$ , 1.01% moisture, loss on ignition 4.89%. The dark blue solid obtained from the water-soluble portion contained 23.85% of molybdenum as  $\text{MoO}_3$  and 38.5%  $\text{SO}_3$ . The blue compound was extracted with ether in a hydrochloric acid solution and the molybdenum occurring as  $\text{MoO}_3$  determined by titration with  $\text{N}/100$  permanganate. The total molybdenum was obtained by fuming with sulphuric acid and passing the solution through a Jones "reductor" (see Mellor's "Quantitative Inorganic Analysis," 1913, p. 190). The molybdenum may be extracted from the ore with water, dilute acids, dilute alkali carbonates and alkalis, and by roasting with a small amount of the reagents followed by a water leach. The best results, corresponding to an extraction of about 80% of the molybdenum, were obtained with a cold water leach followed by hot treatment with 15% sulphuric acid or by treatment of the ore with caustic soda. (See also J. Chem. Soc., Oct., 1918.)

—F. C. Th.

*Bismuth tellurides.* Amadori. See VII.

#### PATENTS.

*Hardening, tempering, and case-hardening metal articles; Processes for* —. J. J. W. York, Bensham-on-Tyne, and S. W. H. Taylor, Newcastle-on-Tyne. Eng. Pat. 117,761, Nov. 20, 1917. (Appl. No. 17,078 of 1917.)

$\alpha$ -NAPHTHOL,  $\beta$ -naphthol,  $\alpha$ -anthranol,  $\beta$ -anthranol, phthalic acid, naphthalene mono- or di-sulphonic acids or salts, anthracene mono- or di-sulphonic acids or salts, and borneol are used in conjunction with water, oils, or other fatty substances for the purposes mentioned.—C. A. K.

*Annealing metal wire by electricity; Process of* —. H. Alexander and W. T. Vint, Leeds, and A. Imbery, Halifax. Eng. Pat. 117,822, July 30, 1917. (Appl. No. 2878 of 1917.)

PORTIONS of a continuously travelling wire are heated by causing them to form parts of a regulated electric circuit and are cooled by passing through a bath, the operations being repeated in sequence with any variation of heating required, as many times as desired. The wire travels between guide rolls which form the contacts of the electric circuit.

—C. A. K.

*Heat treatment of metals; Furnaces for* —. H. Rogers and C. M. Waiter, Birmingham. Eng. Pat. 117,903, Aug. 9, 1916. (Appl. No. 11,225 of 1916.)

FOR the heat treatment of metals in a neutral or other atmosphere with after-quenching of the metal without exposure to air, a vertical retort is provided, with a removable lid at the upper end, the lower end dipping into a quenching bath. A portion of the retort is heated externally and the desired atmosphere is obtained by passing a suitable gas through an inlet above the bath. The metal article to be treated is suspended from the lid and is heated by radiation from the heated portion of the retort.—C. A. K.

*Heat treatment furnaces for metals; Annealing, tempering, or* —. J. R. Hoyle and H. S. Burn, Sheffield. Eng. Pat. 118,040, Dec. 4, 1917. (Appl. No. 17,931 of 1917.)

FOR the treatment of metals which require a rapid or special rate of cooling, a fan is provided to supply the air necessary, and may be fixed to draw the hot air from the chamber or to force cold air into the furnace.—C. A. K.

*Metals; Deposition of* — by electrolysis. M. A. Bolton, Oakamoor, and A. Walker, Stoke-on-Trent. Eng. Pat. 117,872, Nov. 8, 1917. (Appl. No. 16,339 of 1917.)

THE cathode is treated with a solution of wax or the like in petrol or similar solvent containing dissolved hydrogen sulphide, whereby a waxy sub-sulphide film is produced sufficient to produce a coloration. This film prevents adhesion between the deposited metal and the cathode, without unduly increasing the electrical resistance.—C. A. K.

*Metals; Electrochemical precipitation of* —. E. R. Holden, Los Angeles, Cal. U.S. Pat. 1,269,565, June 11, 1918. Date of appl., Oct. 10, 1916. Renewed Nov. 5, 1917.

THE electrolyte is agitated, and the electric current introduced at a number of points at different distances from the cathode, the voltage at each point being regulated, in accordance with its distance from the cathode, to maintain any desired density of current throughout the solution.—E. N.

*Crucible furnaces for melting brass and the like.* C. F. Price, Birmingham. Eng. Pat. 118,000, Sep. 19, 1917. (Appl. No. 13,450 of 1917.)

A PORTABLE coke-fired crucible furnace is built so that the outer casing is supported by the grate-bar frame, the outer casing having projecting feet for the support of the internal firebrick lining. The shape of the feet is such as to allow of a circulation of air for the protection of the metal parts.

—C. A. K.



*Crucible melting-furnaces heated by gas or oil.* L. C. Harvey, and The Morgan Crucible Co., Ltd., Battersea. Eng. Pat. 118,037, Nov. 23, 1917. (Appl. No. 17,302 of 1917.)

To facilitate the removal of spilt slag and metal which has solidified in the base of a furnace, a false cover is suspended over the solidified bed, after the removal of the crucible, thus enabling a high temperature to be generated locally sufficient to melt the spilt material. Drain holes are provided in the bottom of the furnace for the escape of the melted mass.—C. A. K.

*Aluminium; Production of* —. R. Welford, London. Eng. Pat. 118,312, Aug. 18, 1917. (Appl. No. 11,865 of 1917.)

ALUMINA-BEARING clay or other aluminous material is dried and mixed with hot hydrochloric acid, filtered, and the filtrate evaporated in a still, the acid being recovered. The separated salts are removed from the still to a crucible or heating chamber, and heated to obtain oxides or hydroxides for subsequent reduction to metal in an electric furnace.—T. H. B.

*Oxides [e.g., alumina]; Reduction of metallic* —. P. R. Hershman, Chicago, Ill. Assignor to The Mineral Products Co., New York. U.S. Pat. 1,273,220, July 23, 1918. Date of appl., Dec. 21, 1914.

ALUMINA is reduced to metal by mixing it with silicon carbide and heating to 2200° C., the gaseous reaction products being quickly and positively removed.—T. H. B.

*Aluminium; Process of annealing* —. L. H. Whitney, Pittsfield, Mass. Assignor to General Electric Co. U.S. Pat. 1,273,706, July 23, 1918. Date of appl., May 7, 1917.

ALUMINIUM containing dissolved silicon is annealed by heating it rapidly to about 370° C. to 450° C., maintaining the temperature until the aluminium is softened, then quickly chilling so as to prevent the formation of appreciable quantities of graphitoid silicon.—T. H. B.

*Aluminum-solder.* D. M. Campbell, Brooklyn, N.Y. U.S. Pat. 1,273,146, July 23, 1918. Date of appl., Aug. 22, 1917.

ALUMINIUM solder having a melting point substantially that of ordinary solder consists of copper, 6 to 8%; aluminium, 7 to 11%; lead, 12 to 15%; tin, 20 to 30%; and zinc, 40 to 50%.—C. A. M.

*Zinc-distilling furnace and condenser; Electric* —. J. Thomson, New York, Assignor to J. Thomson Press Co., Jersey City, N.J. U.S. Pat. 1,265,973, May 14, 1918. Date of appl., Jan. 19, 1916.

THE condenser is provided with a number of transverse plates, and the fume from the furnace passes between the plates and around their ends. Near the outer end of the condenser the plates extend the full width of the condenser except where the upper parts of the ends are cut away to allow the passage of fume. In each of the sections bounded by these latter plates the condensed metal is kept separate, whereas in the sections formed by the earlier plates the condensed metal collects to form a common bath. By suitable regulation of the temperature in the condenser, practically pure zinc is collected in this common bath, and nearly the whole of the cadmium in the condensed metal in the separate sections at the outer end of the condenser. The metal may be drawn from the different sections of the condenser by means of trapped taps.—C. A. K.

*Zinc vapour; Apparatus and method for condensing* —. C. H. Fulton, St. Louis, Mo., Assignor to Metallurgical Laboratories, Inc., Chicago, Ill. U.S. Pat. 1,271,560, July 9, 1918. Date of appl., Jan. 8, 1917.

ZINC vapours from the furnace are conducted through an annular passage between concentric tubular chambers, which may be either heated or cooled. Baffle-plates are provided to act as condensing surfaces.—C. A. K.

*Phosphorus; Process for making alloys of* —. L. A. Jeffs, Salt Lake City, Utah. U.S. Pat. 1,268,849, June 11, 1918. Date of appl., Nov. 13, 1917.

A MIXTURE of a phosphate rock, carbon, a suitable compound of the required metal, e.g., copper, and a flux, is placed in an electric furnace and a sufficiently strong current is passed to maintain a fusion zone in which oxides of phosphorus and the molten metal are liberated. An excess of carbon is used to ensure the reduction of the oxides of phosphorus.—B. V. S.

*Metal coating.* E. W. McMullen, Assignor to Simmons Co., Kenosha, Wis. U.S. Pat. 1,268,987, June 11, 1918. Date of appl., Feb. 11, 1918.

A COATING of zinc is electrolytically deposited on the surface of steel or other ferrous metal articles, the deposit itself being afterwards coated with a cellulose lacquer.—C. A. K.

*Galvanised iron.* J. H. Gravell, New York. U.S. Pat. 1,273,358, July 23, 1918. Date of appl., Mar. 23, 1918.

GALVANISED iron is provided with a superficial coating of zinc phosphate and over the latter an adherent coating of paint.—B. N.

*Alloy; Soft [bearing] metal — and method of making same.* W. L. Rice, Assignor to P. S. Braucher, Reading, Pa. U.S. Pat. 1,269,000, June 11, 1918. Date of appl., June 27, 1916.

AN alloy of lead, tin, antimony, and sodium is prepared by adding caustic soda to a molten mixture of tin and antimony, mixing the molten product with lead, then adding a further quantity of caustic soda.—T. H. B.

*Waste pickle liquor; Method of treating* —. *Process of producing by-products from waste pickle liquor.* (A) A. F. Hoffman and W. M. Parkin, (B) A. F. Hoffman, Pittsburgh, Pa. U.S. Pat. (A) 1,269,441 and (B) 1,269,442, June 11, 1918. Date of appl., Apr. 8, 1916. Renewed Nov. 10, 1917.

(A) SPENT pickle liquor is evaporated and ferrous sulphate crystals are separated and pulverised. The acid mother liquor is used again for pickling. The ferrous sulphate is allowed to oxidise and the product heated in the presence of steam at a temperature between 179° C. and 338° C. to recover ferric oxide and sulphuric acid, the acid vapours evolved being absorbed in waste pickle liquor. (B) Waste pickle liquor is treated with calcium and magnesium compounds to precipitate the soluble iron salts as hydroxides, the sludge being dried and calcined in such a manner as to reduce the calcium sulphate. The alkaline-earth sulphide is removed from the calcined product by leaching with a suitable liquid.—C. A. K.

*Pickling process and bath.* A. F. Hoffman, Pittsburgh, Pa. U.S. Pat. 1,269,443, June 11, 1918. Date of appl., Jan. 23, 1917. Renewed Nov. 10, 1917.

THE article to be pickled is subjected to the action of a pickling bath which is afterwards restored to

an active condition by treatment with an oxidising agent in presence of an oxygenating catalyst (*e.g.*, a manganese compound) at a boiling temperature.—C. A. K.

*Electric welding process.* H. A. Newcomb, Wilkesburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,270,860, July 2, 1918. Date of appl., May 11, 1914.

METALS having different melting points are heated unequally by the passage of an electric current, in somewhat relative proportion to their melting points, and are at the same time pressed together until surface fusion is effected. Iron or steel articles may be welded by fitting a refractory metal plug (*e.g.*, of tungsten type) into recesses formed in the ends of the articles and applying heat and pressure to weld the refractory metal in the recesses.—C. A. K.

*Furnace [for reduction of tungsten]; Electric* —. C. A. Pfanstiehl, Highland Park, Ill., Assignor to Pfanstiehl Co., Inc., North Chicago, Ill. U.S. Pat. 1,271,760, July 9, 1918. Date of appl., Oct. 30, 1916.

A TUBE of refractory material is surrounded by a heating element, and a protecting lining of nickel covering the inner wall of the tube is provided within and insulated from the heating element for protecting the walls of the furnace from the sub-oxides of tungsten. Means are provided for supporting the tungsten trioxide, to be reduced to metallic tungsten, within the metal lining.—B. N.

*Converter lining.* F. E. Hinners, Milwaukee, Wis. U.S. Pat. 1,272,442, July 16, 1918. Date of appl., Oct. 19, 1917.

A LINING is made, shaped, and burned, and is afterwards inserted in the shell with a wet packing between the lining and the shell.—C. A. K.

*Removing enamel from enamelled metal articles.* R. D. Cooke, Assignor to Columbian Enamelling and Stamping Co., Terre Haute, Ind. U.S. Pat. 1,272,917, July 16, 1918. Date of appl., Sep. 5, 1917.

THE enamel is rendered porous by an acid solution and then loosened by an alkali; for example the articles are treated with 10% hydrochloric acid heated to 160° and then with 25% sodium hydroxide at 160°.—J. H. P.

*Platinum and similar metals; Extraction of* — from their sands and ores. R. Thayer, Philadelphia, Pa. U.S. Pat. 1,273,202, July 23, 1918. Date of appl., Aug. 15, 1917.

THE sand or ore is mixed with metallic mercury, with or without an alkali metal, and the mercury distilled off at a temperature below the melting point of platinum or similar metals, the distilled mercury and "volatilised platinum" being collected in water and suitably separated.—T. H. B.

*Cerium and the like; Process of making* —. A. and M. Hirsch, New York, Assignors to Alpha Manufacturing Co. U.S. Pat. 1,273,223, July 23, 1918. Date of appl., Sep. 1, 1917.

FUSED chloride of cerium and similar rare earth metals is electrolysed, and during the electrolysis hot, dried solid portions of the chloride are added to the bath to maintain the temperature at about 850° C. The cathode current density used is sufficient to cause lanthanum and didymium to separate with cerium, and the portion of the anode

above the electrolytic bath is protected by a coating of the electrolyte. A terminal voltage of less than 12 volts is used and an anodic current density of less than 8 amp. per sq. in.—T. H. B.

*Nickel; Method of separating* — from nickel-copper alloys. J. Diavernas, Assignor to United States Nickel Co., New Brunswick, N.J. U.S. Pat. 1,273,465, July 23, 1918. Date of appl., Sep. 8, 1917.

COTTER-NICKEL matte is roasted, then reduced to metal, and treated with neutral or slightly acid copper solution to dissolve the nickel. The solution is treated with metallic nickel to precipitate traces of copper, and the solution of nickel salts drawn off.—T. H. B.

*[Aluminium] alloy.* T. S. Fuller, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,273,762, July 23, 1918. Date of appl., May 24, 1917.

AN aluminium alloy containing about 6–12% Zn, 0.5–2.5% Mg, and 1–3% Fe.

*Electric welding.* S. C. Cary, Brooklyn, N.Y., U.S.A. Eng. Pat. 118,138, Aug. 16, 1917. (Appl. No. 11,511 of 1917.)

*Tin-plates; Manufacture of* —. C. D. Godfrey and A. J. Way, Briton Ferry, Glam. Eng. Pat. 118,377, Dec. 5, 1917. (Appl. No. 18,053 of 1917.)

*Alloys.* The Cooper Co., Assignees of H. S. Cooper, Cleveland, Ohio, U.S.A. Eng. Pat. 113,259, May 14, 1917. (Appl. No. 6846 of 1917.) Under Int. Conv., Oct. 30, 1916.

SEE U.S. Pat. 1,221,769 of 1917; this J., 1917, 556.

*Wires; Process for the manufacture of* — from refractory materials [*e.g.*, tungsten]. A. Lederer, Vienna, Assignor to Westinghouse Lamp Co. U.S. Pat. 1,273,506, July 23, 1918. Date of appl., May 16, 1914.

SEE Eng. Pat. 12,358 of 1914; this J., 1915, 967.

*Process for the recovery of potassium salts from blast-furnace flue dust or the like.* Eng. Pat. 117,991. See VII.

*Method of recovering sulphur dioxide from furnace gases.* U.S. Pat. 1,271,899. See VII.

## XI.—ELECTRO-CHEMISTRY.

### PATENTS.

*Furnaces or other appliances; Supply of electrical energy to* —. W. Travis, and T. H. Watson and Co. (of Sheffield), Ltd., Sheffield. Eng. Pat. 118,233, Feb. 27, 1918. (Appl. No. 3460 of 1918.)

THREE-PHASE current is distributed to electric arc or resistance furnaces, or other similar appliances consuming electrical energy and having unequal resistances, by an improved method so that the loads on the several phases are balanced one with another in the power and power factor. The three transformer secondary windings, grouped in open delta fashion, are arranged so that the two windings, connected to the two top electrodes, are connected to the ends of the transformer winding connected to the furnace hearth or bottom electrode, and each makes a vector angle of 120° therewith. The two first-named transformer windings are identical in voltage and capacity, and the last-named



transformer winding is less than either of the other two, and varies in its relation thereto according to the relative resistance of the furnace hearth to the normal resistance of the electric arc of the furnace between the two upper electrodes and the charge in the furnace. In an alternative method two of the transformer secondary windings are connected in mesh or delta fashion, and are connected at their meeting ends to the hearth or bottom electrode of the furnace. The other secondary winding is split, each portion being connected to the open ends of one of the last-named windings and making a vector angle of  $120^\circ$  therewith, and also connected to one of the two upper electrodes. From the above arrangements, when the hearth is non-conductive, the current passing from one top electrode to the second top electrode must traverse all three transformer secondary windings in series. In a further arrangement, the secondary windings are connected by the "Scott" method, the two ends of one transformer winding being connected to the upper electrodes, whilst the ends of the other transformer winding are connected respectively to the middle point of the first-named winding and to the hearth or lower electrode.—B. N.

*Furnaces; Electric* —. H. Wade, London. From Booth-Hall Co., Chicago, Ill., U.S.A. Eng. Pat. 118,357, Sep. 27, 1917. (Appl. No. 13,956 of 1917.)

THE furnace is formed of a heating chamber, the hearth of which is conductive when hot, and is provided with a wall terminal embedded in the hearth underneath a co-acting main electrode of opposite polarity, automatically adjustable. An auxiliary electrode, adjustable by hand, of the same polarity as the wall terminal, is adapted to give the furnace a preliminary heating. One terminal of the secondary circuit of a transformer is connected to the main electrode, and the second terminal with both the wall terminal and the adjustable auxiliary electrode. Several wall terminals, with refractory facings which are conductive when hot, may be arranged in the hearth with main electrodes directly above them, and the furnace is supplied with two-phase or three-phase current, the secondary circuits on one side being connected with the main electrodes, and on the other side with the wall terminals and the auxiliary electrode. The secondary circuits are provided with connections whereby one conductor from the auxiliary electrode is used in common on one side of the circuits, and a conductor is also included in the circuit between the wall terminals, the conductor having a switch which is opened when the auxiliary electrode is withdrawn.—B. N.

*Furnace; Electric* —. W. E. Moore, Pittsburgh, Pa. U.S. Pat. 1,272,077, July 9, 1918. Date of appl., Sep. 11, 1917.

THE furnace is provided with three arcing electrodes, an electrically-conducting shell, and a bottom composed of a mixture of refractory material increasing in electrical conductivity towards the lower face. Transformer secondaries are connected to the electrodes in Y formation, the neutral point being connected to the conducting shell. Switches are provided for connecting the transformer primaries either in delta or Y formation, and thus varying the voltage of the electrode arcs at will. A circulation of the molten bath is produced.—B. N.

*Electric furnace contact*. F. T. Snyder, Oak Park, Ill. U.S. Pat. 1,271,093, July 2, 1918. Date of appl., Mar. 1, 1917.

A molten contact extending through the bottom of the furnace contains a cooling medium, and is provided with ribs projecting from the inside and

outside so as to increase the surface, whereby the contact has a relatively high conductivity for alternating current and a low conductivity for heat. The end of the contact projecting below the bottom of the furnace is encircled by a strap, and means are provided for clamping conductor ends between the ends of the strap, for conducting current to the contact, and for protecting the strap and conductors against injury.—B. N.

*Furnace; Electric* —. E. F. Collins, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,271,280, July 2, 1918. Date of appl., May 12, 1917.

PRISMATIC electrical conductors are arranged in pairs on the base of the furnace, so as to form recesses with upwardly diverging sides. Replaceable conductive blocks are fitted in the recesses in electrical contact with the conductors, and an adjustable electrode co-operates with each block to support an electric arc.—B. N.

*Furnace; [Electric] melting* —. T. F. Bally and F. T. Cope, Assignors to The Electric Furnace Co., Alliance, Ohio. U.S. Pat. 1,272,186, July 9, 1918. Date of appl., Sep. 4, 1917.

THE heating chamber is provided with a hearth located at the bottom of the chamber, and with spaced walls within the chamber, above the hearth. A circular resistance element in the form of a trough is supported on the spaced walls, and heat rays radiating upwards from it are reflected by the roof down on to the hearth, whilst heat rays radiating downwards strike directly on the hearth.—B. N.

*Electrolytic tank for electro-chemical reactions*. E. H. Hamilton, Trail, British Columbia, Assignor to The Consolidated Mining and Smelting Co. of Canada, Ltd., Toronto, Canada. U.S. Pat. 1,269,078, June 11, 1918. Date of appl., Oct. 4, 1917.

AN electrolytic tank is provided with electrodes, and with a hollow baffle, which is interposed between the tank inlet and the adjacent electrode, the inflowing electrolyte being thus directed below the electrodes. The baffle is provided with an inlet and outlet to allow of the circulation of an agent for regulating the temperature, and also with interior strengthening pieces, which resist distortion of the baffle whilst permitting circulation of the fluid.—B. N.

*Electrode*. A. T. Stuart, Assignor to The Toronto Power Co., Ltd., Toronto, Canada. U.S. Pat. 1,269,128, June 11, 1918. Date of appl., July 5, 1917.

THE electrode is composed of current distributors, and a number of metallic strips arranged parallel to each other and with the rear edge of each strip in contact with the distributors, the width of the strip being at an appropriate angle to the surface formed by the front free edges of the strips. The strips form current-conducting paths, and the unobstructed spaces between them permit freedom of movement of the electrolyte and the electrolytic products of the reaction. The lengths of the strips are many times their width or diameter or thickness.—B. N.

*Depolarisers; Process of making battery* —. H. F. French and R. C. Benner, Fremont, Ohio, Assignors to National Carbon Co., Inc. U.S. Pat. 1,269,173, June 11, 1918. Date of appl., Nov. 21, 1916.

COPPER oxide is reduced by an excess of sulphur, and the mass re-oxidised in the presence of the excess of sulphur.—B. N.

**(A) Depolariser; Organic manganese-compound —.**

(B) *Process of making conducting hydrated black manganese dioxide.* (C) *Process of making manganese dioxide [depolariser].* (D) *Poly-acid of manganese depolariser.* (A and D) C. Ellis, (B and C) C. Ellis and A. A. Wells, Montclair, N.J., Assignors to National Carbon Co., Long Island City, N.Y. U.S. Pats. (A) 1,269,913, (B) 1,269,914, (C) 1,269,915, (D) 1,269,916, June 18, 1918. Dates of appl., (A) Oct. 18, 1915, (B) Dec. 2, 1915, Renewed Feb. 8, 1918, (C) Dec. 6, 1916, (D) Jan. 17, 1917.

(A) MANGANESE dioxide is treated with glycerol in the presence of an alkali, and the product of the reaction decomposed, whereby hydrated manganese dioxide is obtained. (B) Manganese sulphate, in a solution containing not more than 0.5 to 1% of free acid, is oxidised by sodium hypochlorite, and the sulphuric acid liberated is progressively neutralised so as to maintain the acidity at the above value. (C) A grey or ash-coloured manganese dioxide depolariser is produced by oxidising a slightly acid boiling solution of manganese chloride containing hydrochloric acid by means of a hypochlorite, and progressively neutralising the acid formed. (D) A depolariser for a Leclanché cell consists of a poly-acid of manganese dioxide substantially free from salts of poly-manganous acid.—B. N.

**Depolariser; Electric battery —.** M. E. Holmes, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,272,952, July 16, 1918. Date of appl., Jan. 7, 1916.

A DEPOLARISER for primary batteries contains molybdenum trioxide.—B. N.

**Electrolytic apparatus.** A. J. MacDougall and G. N. Middleton, Assignors to The Toronto Power Co., Ltd., Toronto, Canada. U.S. Pat. 1,269,566, June 11, 1918. Date of appl., July 5, 1917.

AN electrolytic cell is provided with electrodes and with an insulated "off-take," the diameter of which bears such proportion to its length that the electrolyte may circulate with negligible shunt-current losses, and so that the evolved gases during their ascent through the "off-take" convey with them portions of the electrolyte. Gas-separating means are provided, through which the evolved gases and portions of the electrolyte are caused to circulate for their separation, and the separated electrolyte, the temperature of which is regulated by suitable means, is returned through an insulated arrangement to the cell.—B. N.

**Electrolyte-feeding device.** F. G. Wheeler, Assignor to Bleach Process Co., Appleton, Wis. U.S. Pat. 1,269,666, June 18, 1918. Date of appl., June 18, 1917.

THE tank in which the level of liquid is to be maintained is connected with an upper supply tank by a sealed conduit provided with an air-trap portion at an intermediate point in its length, the air-trap consisting of a pressure chamber having an overflow port communicating with the conduit, and a lower feed-port communicating with the liquid of the supply tank. The lower end of the conduit is immersed in the liquid in the lower tank, so as to include a body of gas extending from the top of the trap to the bottom of the immersed open end of the conduit, the gas being under a pressure substantially equal to the depth of immersion of the open end in the electrolyte. The pressure of liquid in the supply tank is maintained constant at the outlet, and in amount sufficient to force liquid therefrom up to the overflow level of the trap.

—B. N.

**Electrolysis; Apparatus for effecting —.** A. Dohmen, Cologne, Germany, Assignor to A. F. Krause, Buffalo, N.Y. U.S. Pat. 1,272,397, July 16, 1918. Date of appl., Dec. 1, 1914.

AN electrolytic cell of the filter-press type comprises frameless, sheet nickel bi-polar electrodes, less than 1 mm. in thickness, alternating with diaphragm-supporting members in the form of frames. Rigid parallel flanges are provided within the frame opening, and a diaphragm is held within the opening between the parallel flanges in crimped connection, thus dividing the interior of the frame into two compartments. Two transverse gas passages are arranged through the top of the frame, and these passages are respectively connected with the compartments on each side of the diaphragm by enlarged chambers extending laterally; the electrodes are similarly provided with holes registering with these passages.—B. N.

**Electrolysing apparatus.** H. C. L. Gougnard, Paris, Assignor to The Linde Air Products Co., Cleveland, Ohio. U.S. Pat. 1,273,357, July 23, 1918. Date of appl., Sep. 4, 1915.

AN apparatus of the filter-press type comprises a series of electrolysing elements clamped together, and supported by lugs on parallel insulated rods on which the elements may be shifted lengthwise. Each element is composed of a diaphragm held between two insulating frames forming cells on opposite sides of the diaphragm, and a metal plate electrode applied to one of the frames, over the cell parallel with the diaphragm. The several elements are provided with registering openings forming an electrolyte conduit communicating with all the cells, and gas conduits each of which communicates with alternate cells through passages in the frames.—B. N.

**Electrolytic for electrolytic cells, such as lightning-arresters, condensers, and the like.** L. W. Chubb, Edgewood Park, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,270,784, July 2, 1918. Date of appl., Oct. 2, 1914.

A FILM-FORMING electrolyte is composed of a solution of boric acid and ammonium borate, the number of gram-molecules of boric acid present being 45—98%, preferably substantially 97%, of the total gram-molecules of soluble material.—B. N.

**Electrolytes for electrical etching; Composition for —.** J. H. Weeks, Rutledge, Pa., Assignor to J. S. Weeks, Delaware Co., Pa., and R. M. Weeks, Philadelphia. U.S. Pat. 1,273,432, July 23, 1918. Date of appl., Apr. 4, 1917.

THE electrolyte is composed of one or more of the oxalates of ammonium, barium, calcium, cadmium, magnesium, iron, potassium, sodium, or strontium, dissolved in water, together with a sulphate, a chloride, and an acid.—B. N.

**Electrical furnace.** H. Nathusius, Friedenshütte, Germany. U.S. Pat. 1,265,485, May 7, 1918. Date of appl., Oct. 5, 1914. Renewed Mar. 30, 1918. See Eng. Pat. 13,951 of 1914; this J., 1916, 547.

[*Electrolytic*] **process of making combustible gas.** U.S. Pat. 1,273,050. See IIa.

**Method of electrolytic production of perchloric acid.** U.S. Pat. 1,271,633. See VII.

**Preparation of potassium persulphate by electrolysis.** Ger. Pat. 306,194. See VII.



## XII.—FATS; OILS; WAXES.

*Oil seeds from Sierra Leone; New* —. Bull. Imp. Inst., 1918, 16, 35–40.

*Pentadesma butyracea*, "butter or tallow tree," is a large tree, which occurs sparsely in West Africa, but which could be planted if a demand for the seeds arose. The kernels are large, reddish-brown, and irregular in shape; the samples arrived with 29% of moisture but the kernels could be dried before shipment so as to yield about 40% of fat. The fat is equivalent to 41.8% on the dry substance, pale yellow in colour and rather soft. The following data were obtained:—Sp. gr. at 100°/15° C., 0.8565; m.pt., 33° C.; acid value, 17.4; saponification value, 191.7; iodine value, 42.8%. The residual meal cannot be used for feeding purposes and contains only a small proportion of nitrogen. *Piassava oil*. A sample, presumably obtained from the fruit of a species of *Raphia*, had a dark orange-red colour and contained a fair proportion of separated stearine; it was darker and more liquid than palm oil. The sample showed:—Sp. gr. at 15°/15° C., 0.919; solidifying point of fatty acids, 41.1° C.; acid value, 6.2; saponification value, 192.7; iodine value, 79.5%; Hehner value, 94.7; insoluble fatty acids, 92.6%; unsaponifiable matter, 2.1%; volatile acids, soluble, 0.3; ditto, insoluble, 0.2. This oil is similar in character to ordinary palm oil and would have the same commercial value. "*Po-Yoak*" nuts and oil. These nuts are almost spherical, 1.3 in. diam., with rough dark brown shell. The kernel has a slightly wrinkled, light brown surface and the flesh is fairly soft, dark reddish-brown, and very oily; it has a peculiar odour resembling that of tung oil. The nuts are probably derived from a species of *Parinarium*. The sample of oil received possessed the same odour; it was pale yellow with a considerable deposit of "stearine." Analysis showed:—Sp. gr. at 15°/15° C., 0.963; solidifying point of fatty acids, 30.6° C.; acid value, 2.6; saponification value, 189.1; iodine value, 156.9%; unsaponifiable matter, 0.84%. The high iodine value of the oil indicates that it belongs to the class of "drying" oils; the skin formed on drying is rather rough and not transparent. The oil could be used for the manufacture of paints and varnishes; it becomes thick like linseed oil on heating at 280° C.

—J. F. B.

*Determination of chlorine in mixtures containing silicates*. Bruhns. See VII.

### PATENTS.

*Oils; Preparing catalytic agents for hydrogenating* —. K. Kimura, Kobe, Japan. Eng. Pat. 118,323, Aug. 22, 1917. (Appl. No. 12,032 of 1917.)

A powdered nickel salt is mixed with a non-combustible non-catalytic substance such as powdered asbestos or pumice, and the mixture dried and heated in a vessel made of non-catalytic material, while ammonia gas or a vaporised ammonium compound, such as ammonium chloride, is passed through it. Catalytic material thus prepared acts at a relatively low temperature.—C. A. M.

*Oily material containing unsaturated bodies; Hydrogenating* —. *Hydrogenation of unsaturated bodies*. M. H. Ittner, Jersey City, N.J. U.S. Pats. (A) 1,271,575 and (B) 1,271,576, July 9, 1918. Dates of appl., May 6, 1914, and Sep. 27, 1915.

(A) MATERIAL containing unsaturated compounds is mixed with a catalyst and forced through an orifice

communicating with a supply of hydrogen or gas containing hydrogen, so as to cause the gas to be drawn into the mixture and intimately mixed therewith. The mixture is then discharged into an enclosed space containing an atmosphere of hydrogen and also more of the mixture of unsaturated substance and catalyst, and may be made to pass well beneath the surface of the mixture in the container. (B) Oily material is mixed with a catalyst and forced, e.g., centrifugally outwards, through an orifice below the surface of the material. This orifice communicates with a supply of hydrogen which thus becomes entrained by the oil and catalyst and intimate admixture effected. On continuing the circulation of the mixture through the apparatus further amounts of hydrogen are entrained until the desired degree of hydrogenation is obtained, whilst the resulting currents are arrested by means of barriers such as wire screens or baffles to prevent the formation of a vortex.

—C. A. M.

*Oils and the like; Apparatus for extracting* —.

R. Wells, Homer, N.Y., Assignor to Cobwell Corporation, Cleveland, Ohio. U.S. Pat. 1,272,744, July 16, 1918. Date of appl., Apr. 23, 1915.

THE apparatus comprises a cylindrical chamber for the material to be treated, provided with a central vertical shaft, driven from outside, and carrying radial arms close to the bottom of the chamber, whilst arms freely suspended independently on pivots at intervals along a radius of the chamber engage the upper portion of the mass as it is agitated by the radial arms.—C. A. M.

*Process for the separation of grease from wool scouring or like effluents*. Eng. Pat. 118,332. See V.

*Treatment of linseed oil fatty acids*. Eng. Pat. 118,228. See XIII.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Varnishes; Hardness and viscosity of* —. R. P. L. Britten. Oil and Colour Chem. Assoc., Sep. 17, 1918.

THE varnish maker's practical "thumb-nail test" is inadequate, as it fails to give quantitative expression to the results obtained. The mechanical hardness testers of Laurie and Baly, Jahns, and Clemens measure toughness rather than hardness. The author described a method which would determine true hardness, and which was based on the principle of the hardness of minerals scale, scratch points of various crystals of known relative hardness being used. A method embodying the use of gelatin points of differing water content and registering results obtained in degrees of water was also suggested. Of the various types of instruments for determining viscosities of the widely differing types of varnishes, those in which force as against time, was the factor determined, are recommended. The author has elaborated a type of viscometer having for its principle the determination of the time taken for a glass or metal ball suspended on one arm of a balance of high moment of inertia to fall a definite distance through the liquid the viscosity of which has to be determined. The times taken under similar conditions in air and in water serve for the standardisation of the instrument ("retardation for water"). Viscosity is calculated as time taken in the liquid

less time taken in air, divided by the retardation for water. As equilibrium is readjusted after immersion of the ball, a correction is made for specific gravity. In this way values were obtained for four characteristic oils as follows:—linseed oil, 7.7; olive oil, 10.5; rape oil, 12.1; castor oil, 200.

—A. de W.

*New oil seeds from Sierra Leone. See XII.*

#### PATENTS.

*Linseed oil fatty acids; Treatment of* —. Barry, Ostlere and Shepherd, Ltd., and J. Barry, Kirkcaldy. Eng. Pat. 118,228, Feb. 5, 1918. (Appl. No. 2057 of 1918.)

LINSEED oil fatty acids are rendered readily oxidisable, and thus suitable for the manufacture of linoleum and paints, by subjecting them to the action of superheated steam, preferably after heating in a steam-jacketed pan. The superheated steam decomposes any metallic compounds present, expels volatile deleterious substances, and acts as an oxidation catalyst.—C. A. M.

*Impregnated material; Manufacture of* —. J. Rosen, Assignor to Soc. Anon. des Combustibles Industriels, Paris. U.S. Pat. 1,273,673, July 23, 1918. Date of appl., Jan. 11, 1915. Renewed May 29, 1918.

THE basic material is dehydrated, and coated first with a layer of tar oils, then with talc, and then with successive layers of tar oils having different degrees of oxidation from that of the first coating.—C. A. M.

*Treatment of textile fabrics, wood, leather, and other materials.* Eng. Pat. 117,640. See V.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Vulcanised rubber; Porosity of* —. Remarks on the nature of the vulcanisation process. G. van Iterson. Communications of the Netherlands Govt. Inst. for Advising the Rubber Trade and the Rubber Industry. Int. Assoc. for Rubber Cultivation in the Netherland Indies, 1916 (in Dutch; 1918 in English), Part VII., 224–261.

RUBBER articles vulcanised in direct steam without precautions being taken to prevent condensation on their surface become superficially porous. Every rubber mixing, however, may be made to yield an internally porous product by under-vulcanising in direct steam and then releasing the steam pressure suddenly. Internal porosity is also frequently observed in vulcanite, but can be prevented by raising the temperature very slowly at the commencement and releasing the steam pressure very gradually at the end, a period of  $1\frac{1}{2}$  hours being none too long for each of these operations. The internal porosity is due to the presence of moisture in the rubber-sulphur mixture, even normally masticated rubber sometimes retaining 0.2 to 0.3% of moisture; this explanation of the origin of the porosity is confirmed by experience with oil bath vulcanisation (this J., 1918, 215 A), and by experiments in which the development of water vapour bubbles was prevented by an external pressure of steam or even of air. Under-vulcanised rubber is much more liable than well-vulcanised rubber to the development of porosity, because when the external pressure of the rubber is suddenly released, the temperature being still above 100° C., the under-vulcanised rubber is in a very soft, plastic condition, with low tensile strength, and the vapour pressure of the included

water causes the formation of cavities which still persist when, on cooling, the plasticity gradually becomes replaced by elasticity. In vulcanising bulky articles porosity is likely to occur because there may be a lag in the vulcanisation of the interior, which will also remain warm and plastic longer than the exterior after the release of the steam pressure. It is also possible that during the earlier stages of vulcanisation the vapour pressure may, for various reasons, locally exceed the pressure predominant in the vulcanisation apparatus, with the result that porosity may be developed which may then be rendered permanent by the further progress of the vulcanisation process. In a review of the work of the Institute and of other investigators on the nature of the vulcanisation process the following views are expressed. The sulphur becomes definitely "bound" by the rubber during vulcanisation, but the limit of combination probably is not reached at the proportions 32 : 68, corresponding with the composition  $C_{10}H_{16}S_2$ ; no final evidence as to the reversibility of vulcanisation is yet forthcoming. The nature of the curve representing the variation in the rate of vulcanisation with alteration in the amount of sulphur in the mixing is largely dependent on the latter; with less than 10% of sulphur the curve is almost rectilinear, whereas above this percentage the curve takes the S shape characteristic of an autocatalytic process. In the initial stages of vulcanisation of mixtures containing a high percentage of sulphur the rate of fixation of the sulphur is dependent on the limited solubility of the latter in rubber; as the rubber becomes vulcanised its solvent power gradually increases, and therefore assists the autocatalytic effect in causing the gradual steepening of the vulcanisation coefficient—time curve (see also Skellon, this J., 1915, 671); the initial rate of combination is therefore not proportional to the concentration of sulphur when the latter is high, viz. above 10%. The continuity of the changes induced by vulcanisation, whether by sulphur or by sulphur chloride, together with the considerable effect produced by quite small percentages of "combined" sulphur or sulphur chloride, leads the author to favour the view that a colloidal process plays an important part, and that either the vulcanising agent or a chemical compound of this with part of the rubber becomes colloidal bound with the remaining rubber, forming a complex which is insoluble in the ordinary rubber solvents. Consideration is given to the possibility that the various allotropic modifications of sulphur may be involved in the vulcanisation process, and attention is again drawn to the fact that the common organic accelerators of vulcanisation are also effective catalysts in the inter-transformation of  $S_8$  and  $S_2$  (compare this J., 1917, 787), but no definite conclusion is attained.—D. F. T.

*Mottling of [vulcanised] Para rubber; Nature of* —. H. Rimpel. Gummi-Zeit., 1916, 31, 144. Chem.-Zeit., 1918, 42, Rep., 119.

THE sample examined was free from substitute, and yielded only about 3% of ash. The lightest patches contained twice as much free sulphur as the darkest. The formation of the patches is explained as follows:—After vulcanisation the sulphur which has not combined chemically with the rubber passes on cooling into the amorphous or the rhombohedral form, except at the surface, where octahedral crystals quickly separate. Sulphur wanders from the interior of the sample to the surface, and the sample "sulphurs up." At the same time, however, there occurs conversion of the less stable into the more stable form within the sample, the rubber acting as a solvent, and sulphur wanders from certain parts of the solution and accumulates round other centres.—E. W. J.



## PATENTS.

*Rubber; Process of treating* — F. A. Fahrenwald, Cleveland, Ohio. U.S. Pat. 1,269,168, June 11, 1918. Date of appl., Oct. 11, 1916.

"SULPHURISED" rubber is reclaimed by heating it with tellurium, with or without pressure.

—E. W. L.

*Vulcanisation of rubber and similar materials.* D. F. Twiss, Sutton Coldfield, Assignor to The Dunlop Rubber Co., Ltd., London. U.S. Pat. 1,271,810, July 9, 1918. Date of appl., Jan. 4, 1918.

SEE Eng. Pat. 110,059 of 1916; this J., 1917, 1185.

## XV.—LEATHER; BONE; HORN; GLUE.

*Utilisation of nitrogenous refuse [leather and horn waste, etc.].* Donath and Ulrich. See XVI.

## PATENTS.

*Treatment of textile fabrics, wood, leather, and other materials.* Eng. Pat. 117,640. See V.

*Manufacture of metallopyrophosphoric acids or their salts associated with boron compounds or containing boron, and application of the products in tanning.* Eng. Pat. 117,693. See VII.

*Method of recovering fertilising material from waste liquids.* U.S. Pat. 1,269,189. See XVI.

## XVI.—SOILS; FERTILISERS.

*Nitrification in natural soils and its importance from an ecological point of view.* H. Hesselmann. Skogswärdsföreningens Tidskrift, 1917, 15, 312—446. Bull. Agric. Intell., 1918, 9, 602—665.

A STUDY of the nitrogen cycle in forest-soils. The author distinguishes between "sweet" humiferous soils, well aerated by the action of worms and insects, and "strong" humiferous soils, formed of dead decomposed or decomposing vegetation, the former being characteristic of deciduous forests and, to a lesser degree, of pine forests on soils rich in inorganic salts, the latter, on the contrary, being found in pine forests of the ordinary type, and often forming a series of layers, with varying stages of decomposition, which can be clearly separated from the mineral subsoil. The sources of nitrogen in forest soils are the dead, decomposed covering, to the nitrogen of which is added the atmospheric nitrogen fixed by leguminosae, alders, and other plants; the soil fungi and bacteria which can fix nitrogen, and which obtain their energy for this fixation by decomposing the organic matter in the soil; the ammonia and nitric acid in rain. In naturally wooded land the second factor is the most important one. The processes of nitrate formation in forest soils were investigated by the examination of the soil for nitrifying bacteria, the determination of the nitrifying capacity of the soil, and determination of the nitrogen content of the trees and plants at different seasons, the following conclusions being drawn. The "sweet" humus of beech woods contains as many nitrifying as denitrifying bacteria and has considerable nitrifying power, and the herbs and grasses also help considerably to increase the stock of nitrogen in

such a type of soil. The humus of pine forests with a mossy covering is characterised on the other hand by the absence of nitrifying and denitrifying bacteria, and no potassium nitrate can be found in the tissues of the plants covering the soil. With regard to the acidity of the dark humic soils of pine woods, the author observes that, as colloids are coagulated by the addition of inorganic salts, the humus lying on a subsoil very rich in soluble inorganic salts is less likely to give an acid reaction, because the humus particles are flocculated, thus allowing the penetration of roots, worms, and insects, all of which favour the aeration of the soil. Many natural soils have the power of nitrification, and it is characteristic that humus formation in such soils takes place under the influence of electrolytes, the formation of "sweet" humus being facilitated by the action of worms and insects. In many places nitrification is so rapid that there is a considerable accumulation of nitrogen in the covering vegetation, as in dense beech, elm, oak, ash, and alder forests. Nitrification is not only largely influenced by the origin of the soil, but also by the climate, and as the degree of nitrification constitutes a powerful factor determining the composition of the plant associations of a given soil, the factors of the soil formation may have a decisive influence, in certain cases, on that composition. Thus the same forest trees grow more rapidly on soils where nitrification takes place than where there is no nitrification, and a suitable forest system should make it possible to thin numerous forests so as to favour soil nitrification, thus assuring a higher yield of wood, although a good yield of pine and spruce wood can be obtained on soils where there is no nitrate formation. In the latter case rate of growth is governed by the rate of ammonification, and the formation of the soil might well be influenced by a suitable system of forest management.—W. G.

*Phosphates; Influence of nitrifying bacteria on* — C. G. Hopkins and A. L. Whiting. Univ. of Illinois Agric. Exp. Stat., Bull. 190 (June, 1916), 395-406. Bull. Agric. Intell., 1918, 9, 790—791.

EXPERIMENTS showed that during the conversion of ammonia into nitrite by nitrite bacteria, there was also an action on any insoluble phosphate present, a portion being rendered soluble. About 1 lb. of phosphorus and 2 lb. of calcium were dissolved for each pound of nitrogen oxidised. Nitrate bacteria and ammonia-producing bacteria did not convert insoluble phosphate into soluble compounds, but certain acid-producing bacteria did so. The amounts of calcium and phosphorus rendered soluble in this manner by biochemical action exceed the requirements of a crop.—J. H. J.

*Nitrogen fixation and losses of nitrate in the soil; Influence of plant residues on* — H. B. Hutchinson. J. Agric. Sci., 1918, 9, 92—111.

LABORATORY, pot, and field experiments have been conducted to determine the effect of carbohydrates (sugar and starch) and plant residues, as sources of energy, on nitrogen fixation by *Azotobacter*. In the laboratory experiments, under suitable conditions, an increment of upwards of 6 mgrms. of nitrogen per grm. of plant residues occurred, and in pot experiments gains of 9 mgrms. per grm. of substance were obtained. In the field experiments definite increases of crops, equal to 20—54%, were obtained by the application of sugar (at the rate of 1 ton per acre per annum) when the soil conditions were favourable. The difference between the action of plant residues and sugar being largely one of degree, it is reasonable to suppose that

such substances as stubble, leaves, etc., may also contribute indirectly to the reserves of soil nitrogen. For the successful operation of nitrogen fixation the suitable general soil conditions are, in addition to the supply of some source of energy, a suitable temperature, the presence of phosphates, and a supply of a base such as calcium carbonate. Even under favourable conditions, however, there occurs a period during which adverse processes come into play, and these should be allowed to run to completion before a crop is introduced. Under unfavourable conditions, and particularly during periods of low temperature, these adverse changes may persist without any subsequent soil gains. Thus in the field experiments the only cases where crop increases were obtained were when the sugar was applied in the early autumn for the next year's crop of barley.—W. G.

*Nitrification; Influence of polsherd on — in the Indian alluvium.* J. N. Sen. J. Agric. Sci., 1918, 9, 32—42.

The results of pot and lysimeter experiments in the field indicate that the admixture of 30% of potsherds with the soil causes a marked increase in the total amount of nitrates found in the percolation water.—W. G.

*Soil; Protozoa and the phenomena of reduction in —.* C. A. H. von Wolzogen Kühr, jun. Arch. Suikerind. Ned. Ind., 1917, 1125—1182. Bull. Agric. Intell., 1918, 9, 788—790.

QUANTITATIVE determinations of reduction phenomena in soil include the "reduction-index," i.e., the number of c.c. of  $N/10$  permanganate required to oxidise the ferrous iron liberated from 100 grms. of dried soil in a "sulphurous solution," and the "ferro-index," a similar result obtained in an "acetic solution." Soils may be divided into three classes: good soils, showing little reduction and containing few protozoa, less than 50 per grm.; bad soils, with a high reduction-index and many protozoa, over 50 per grm.; very bad soils, with a very high reduction-index and few protozoa. As protozoa are aerobic, their number increases during the first stages of reduction when they feed on the anaerobes; as reduction continues, the air decreases and the development of the protozoa is checked; finally, the activity of the anaerobes increases to such an extent that the protozoa decrease and die. Butyric fermentation is especially fatal to protozoa. Sulphate reduction is not so injurious, as the hydrogen sulphide produced combines with the iron present. The examination of a large number of soils from sugar-cane plantations in Java showed that there was a relation between the water content, the reduction-index, the ferro-index, and the number of protozoa.

—J. H. J.

*"Soil sicknesses"; Researches on certain —.* N. L. Sölmgén, A. Knetemann, and K. T. Wieringa. Verslag. van Landbouwk. Onderzoek. der Rykslandbouwraproefstat., 1917, 21, 121—165. Bull. Agric. Intell., 1918, 9, 659—662.

AN investigation of the soil conditions favouring the appearance of "haverzlekte" (oat disease) and "Hooghalensche ziekte" (Hooghalen disease) which have appeared recently in the Netherlands and Germany. The first is attributed to an excess of bases in the soil, and the second to excessive soil acidity. The direct and immediate cause of the appearance of these "soil sicknesses" has not yet been determined, but a close relationship has been established between their appearance and the free humic acid content of the soil, thus enabling the soil to be rendered healthy by means of suitable

treatment. Two methods for the determination of the content of free and combined humic acid and the lime requirement of a soil are described, namely, (a) by means of cultures of *Azotobacter*, and (b) by the aid of a diffusion method on agar plates stained by indicators. In method (a) 50 c.c. of distilled water, 2% of mannitol, 0.2% of dipotassium phosphate, and 5 grms. of soil are placed in each of two conical flasks, the mixture is inoculated with *Azotobacter* and incubated at 26° C. A control flask receives in addition some calcium carbonate. The soil is said to be basic or acid according as the bacteria do or do not grow in the flask without calcium carbonate, and it was found that soils causing the oat disease did not favour the growth of the bacteria, whilst soils affected with Hooghalen disease did favour their growth. To determine the lime requirement of the soil two species of *Azotobacter* were used, *A. vinelandii* and *A. chroococcum*, the former being able to grow at a slightly higher degree of acidity than the latter. The lime requirement of a soil was taken as that amount which just suffices for the growth of *A. vinelandii*, but is insufficient for *A. chroococcum*. The results by this method agree quite well with those obtained by the diffusion method. This latter method is based on the fact that acids and bases, even when difficultly soluble, diffuse in a hydrogel. In a layer of agar coloured by litmus, small cavities are made without piercing the layer, and it is found that acids or alkalis placed in these cavities develop a surrounding zone of acid or alkaline reaction in the agar, and this effect is perceptible down to a dilution of  $N/5000$ . In the determination of the free humic acid content of a soil, 10-grm. portions of soil are heated for three hours at 100° C. with increasing quantities (5 mgrms. at a time) of calcium carbonate, and their reaction is then tested on the agar plate, the results for the soil being expressed in mgrms. of calcium carbonate on the soil or on its loss on ignition. For the determination of total humic acid content (free and combined), the soil is boiled with  $N/10$  hydrochloric acid, filtered, and washed free of chlorine, and then the total humic acid is titrated by the agar plate method. For the determination of excess of alkali in a soil, increasing amounts of hydrochloric acid instead of calcium carbonate are added to 10-grm. portions of the soil and the process continued as above. From the results of a large number of analyses the authors find that for any soil, whether sand or peat, acid, neutral or alkaline, the total humic acid per grm. of loss on ignition is always constant and equivalent to about 80 mgrms. of calcium carbonate. They have determined the ratio of free humic acid : combined humic acid, and state that if this ratio is greater than 0.6 the soil is "hyper-acid"; if it is between 0 and 0.5 the soil is healthy, the best percentage of free humic acid being 25% of the total. The content of free humic acid is increased by the application of such salts as potassium chloride, magnesium chloride, and ammonium sulphate, and decreased by the addition of lime, magnesium or sodium carbonates, and sodium nitrate.—W. G.

*Bacterio-toxins in the soil; Non-persistence of —.* H. B. Hutchinson and A. C. Thaysen. J. Agric. Sci., 1918, 9, 43—62.

SEVEN English soils were examined in order to ascertain whether partial sterilisation effects are due to the destruction of bacterio-toxins in the soils. The test organism used was *B. prodigiosus* and the untreated extracts made by means of 0.8% salt solution varied largely in their suitability for the growth of this organism. Treatment of the extracts by heat invariably led to bacterial decreases, contrary to the results of Greig Smith



(Proc. Linn. Soc. N.S.W., 1910, 25, 808—822; 1911, 26, 679—699; Cent. Bakt. Par. ii., 1911, 30, 154—156), who stated that the toxins are destroyed by heat. Extracts of soils treated with antiseptics (which are not supposed to destroy toxins) were on the whole more favourable for growth than those of untreated soils and were found to contain more organic nitrogen than the latter. Unsuitable extracts could be converted into favourable media by the addition of minute quantities (6 parts nitrogen per million) of peptone. Similar tests with a common soil organism, *B. fluorescens liquefaciens*, gave no evidence of toxicity. The decreases in bacterial numbers in poor, untreated soil extracts are symptomatic of starvation. These results are opposed to Greig Smith's views (*loc. cit.*) and only one soil, an acid heath soil, gave extracts at all similar to those reported by him. The value of the extract from this soil was distinctly increased by heating it to 94° C. for one hour, which process at the same time caused the precipitation of acid iron and alumina compounds removed from the soil by the action of the saline solution. The "toxicity" of this soil can be effectively removed within twenty-four hours by treatment with calcium carbonate. Although it is possible, under well-defined conditions, to induce the formation of bacterio-toxins in culture solutions, there is no evidence to show that these are likely to possess importance in the phenomena of partial sterilisation of soil.—W. G.

*Saline soils and their improvement.* K. K. Gedroic. Reprint, pp. 1—16.

THE author describes the treatment necessary for the improvement of soils containing excess of neutral salts, such as the so-called white alkali land, and those containing soda, such as the black alkali land.—T. H. P.

*Soil; Absorptive power and zeolitic bases of —.* K. K. Gedroic. Reprint, pp. 1—55.

EXPERIMENTS have been made on the displacement of the zeolitic bases of a soil by other bases, this being effected by repeated treatment of the soil with solutions of one or more chlorides of N/5, N/1, and 4N concentrations.—T. H. P.

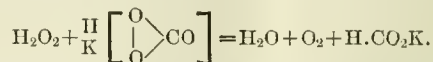
*Salt requirements for young and for mature buckwheat plants in solution cultures; Comparative study of —.* J. W. Shive and W. H. Martin. J. Agric. Res., 1918, 14, 151—175.

THE culture solutions used were thirty-six different sets of salt proportions of the three salts potassium phosphate, calcium nitrate, and magnesium sulphate, the total initial osmotic concentration in each case being equal to 1.75 atmos. In a period of four weeks directly following germination the highest yield of buckwheat tops and roots was obtained in solutions containing the following salt proportions:  $\text{KH}_2\text{PO}_4$  0.0144 m.,  $\text{Ca}(\text{NO}_3)_2$  0.0052 m., and  $\text{MgSO}_4$  0.0200 m. During the second four weeks (including the period of seed production and ripening) the highest yield was obtained in a solution containing  $\text{KH}_2\text{PO}_4$  0.0108 m.,  $\text{Ca}(\text{NO}_3)_2$  0.0130 m.,  $\text{MgSO}_4$  0.0100 m. The amounts of water lost by transpiration in each of the two periods of differing development indicate in a general way the yields of tops. There is no definite correlation between the yields of tops and of seeds, such as there is between the yields of tops and of roots.—W. G.

*Carbon dioxide; Reduction of — by hydrogen peroxide as the basis of assimilation by plants.* H. Wislicenus. Ber., 1918, 51, 942—965.

HYDROGEN peroxide is always at the disposal of the green plant, and the author regards this as the

agent which causes the first stage in the reduction of carbonic acid, namely to formic acid. The carbonate anions are supposed to have peroxide structures, and consequently the reaction is a mutual de-oxidation of two peroxides and requires no expenditure of energy. The process can be tested, for example, with solutions of potassium bicarbonate and hydrogen peroxide, in which case the following equation is given:



(See further, J. Chem. Soc., 1918, i., 472.)—J. C. W.

*Plant; Direct influence of the sap of the wild on the cultivated —, and the action of acid solutions, directly absorbed, on the plant.* C. Campbell. Atti R. Accad. Lincei, 1918, 27, I., 57—61.

ACCORDING to Comes, the greater resistance normally exhibited by wild plants when compared with cultivated ones is due to the greater acidity of the juices of the former. In this connection the author has made some preliminary experiments to ascertain the influence exerted on a cultivated stock by a wild graft and the effect on plants of the direct absorption of acid solutions.—T. H. P.

*Plants; Influence of organic substances on the development of —.* H. G. Ciamician and C. Ravenna. Atti R. Accad. Lincei, 1918, 27, I., 38—42.

FURTHER experiments have been made to ascertain the effect of a number of organic compounds on the germination of the seeds and the development of the plants of beans, maize, tobacco, beetroot, and lupins. (See J. Chem. Soc., 1918, i. 473.)—T. H. P.

*Nitrogenous refuse [leather and horn waste, etc.]; Utilisation of —.* E. Donath and G. Ulrich. Oesterr. Chem.-Zeit., 1917, 20, 165—168. Z. angew. Chem., 1918, 31, Ref., 218.

REFUSE, rich in nitrogenous matter, is heated with waste sulphuric acid; a portion of the nitrogen is thus converted into ammonium sulphate and the remainder is obtained in the form of a highly nitrogenous charcoal. Products which are practically dry, and rich in ammonium sulphate, are obtained by heating for several hours at over 240° C. Chamber acid or waste sulphuric acid may be used. If larger proportions of acid are employed, pasty products are obtained from which fertiliser mixtures may be prepared by combining them with ground limestone or phosphate. The yield of ammonium sulphate may be increased by carrying out the destruction in the presence of certain catalytic agents, preferably at 250°—270° C.—J. F. B.

#### PATENTS.

*Superphosphates and other artificial manures; Apparatus for the manufacture of —.* Simon-Carves, Ltd., and J. H. Brown, Manchester. Eng. Pat. 118,055, Jan. 31, 1918. (Appl. No. 1793 of 1918.)

IS a pit or den, for use in the manufacture of superphosphates and other artificial manures, of the kind requiring rotary scrapers carried by a vertical shaft, which cut away the material in slices and deliver it to a central passage through which the shaft passes, the opening in the bottom of the den is provided with a removable or hinged grid adapted to form a bearing for supporting and steadying the scraper shaft, thus maintaining it central. The grid may carry a shoot also adapted to form a bearing for the scraper shaft.—W. G.

*Fertilising material; Method of recovering — from tannery waste liquids.* V. H. Kadish, Assignor to H. L. Kadish, Milwaukee, Wis. U.S. Pat. 1,269,189, June 11, 1918. Date of appl., May 31, 1917.

The waste liquid is treated with a quantity of acid sufficient to neutralise it and to precipitate the fertilising material and the latter is then separated.

—W. P. S.

*Fertiliser; Preparation of new —.* G. Borghe-sani and G. Stampa, Rome. U.S. Pat. 1,272,001, July 9, 1918. Date of appl., Apr. 7, 1917.

See Eng. Pat. 113,493 of 1917; this J., 1918, 217 A. The pyrites ash may be omitted.

## XVII.—SUGARS ; STARCHES ; GUMS.

*Law of action of sucrose [invertase]; hypothesis of an intermediate compound.* Colin and Chaudun. See XVIII.

*Utilisation of rice and its by-products.* See XIXA.

### PATENTS.

*Carbons; Decolorising — and process of producing the same.* H. M. Shilstone, New Orleans, U.S.A. Eng. Pat. 116,253, July 26, 1917. (Appl. No. 10,759 of 1917.) Under Int. Conv., May 31, 1917.

RICE hulls, rice straw, or rice chaff, or other cereal fibrous material rich in silica, is carbonised and then heated with an alkaline solution to extract any resinous or other soluble matters. The material may first be heated gradually, to vaporise volatile resins, the temperature being subsequently raised for carbonisation; the ground product is then boiled with alkali, preferably with 20% of its weight of sodium hydroxide, and after washing with water it may be extracted with acid. The use of carbons prepared by this process for the decolorisation of, and removal of tannins from, saccharine liquids, e.g., sugar cane juice, is also claimed.—J. H. L.

*Carbon; Process of manufacturing decolorising —.* The Ensuiko Seito Takushoku Kabushiki Kaisha, Y. Okada, and H. Miyake, Shioeisho, Japan. Eng. Pat. 117,828, June 27, 1917. (Appl. No. 9248 of 1917.)

*Fatsia papyrifera*, cane megasse, or any grass found in fresh or sea water, is boiled with a solution of calcium hydroxide, and after the latter has been removed by washing, the material is carbonised, preferably in an iron retort at 750°—800°C. The product may subsequently be extracted with acid.—J. H. L.

*Adhesive [from starch].* C. R. Robinson, Assignor to Perkins Glue Co. U.S. Pat. 1,267,699, May 28, 1918. Date of appl., Mar. 13, 1916.

Dry starch is treated with sodium bisulphate (1%) and sodium hydroxide (6%), and then mixed with trisodium phosphate (1%) and water (6%), with or without ammonia. The mixture is cooked to render the starch soluble, and then neutralised and treated with cuprammonium sulphate (1%), with or without casein. Adhesive or coating preparations are claimed consisting of soluble starch, water, and a waterproofing material, with or without casein.

—J. H. L.

*Glue; Process of manufacturing vegetable — [from starch].* V. G. Bloede, Catonsville, Md. U.S. Pat. 1,269,678, June 18, 1918. Date of appl., Oct. 10, 1917.

A CLEAR viscous liquid is prepared from starch by the action of a caustic alkali and water, and the alkali is then completely or partly neutralised by injection of acid in vapour form.—J. H. L.

*Starch; Method of producing dustless —.* A. W. H. Lenders, Cedar Rapids, Iowa. U.S. Pat. 1,272,682, July 16, 1918. Date of appl., Mar. 30, 1914.

STARCH or other friable material is freed from dust by subjecting it, during its descent through a relatively short, unobstructed chamber, to a series of jets of air at various angles, the separated dust being carried by a current of air away from the field of the jets and the region of delivery of the treated starch.—J. H. L.

*Fuel from waste material and making and utilising the same.* Eng. Pat. 117,921. See IIA.

## XVIII.—FERMENTATION INDUSTRIES.

*Wine; Fixed organic acids, especially lactic acid, in [Tuscan].* G. de Astis. Annali Chim. Appl., 1918, 9, 155—241.

MOESLINGER's method of determining lactic acid in wine (precipitation as barium lactate) usually gives results from 0.1 to 0.2 grm. per litre too high, owing to the influence of tartaric, malic, citric, or succinic acids, but in the case of wines from grapes grown on calcareous soils, or which from their treatment contain a considerable amount of calcium malate, the error may be as high as 0.75 grm. per litre. The addition of sulphur dioxide (5 to 8 grms. per litre) to Tuscan grape juice retarded for a few days the beginning of fermentation, but then accelerated its course and increased the yield of alcohol by 0.1 to 0.5 grm. per litre. It reduced the loss of fixed organic acids, but this effect was lessened or inhibited by the presence of grape stalks during the fermentation. The quantity of fixed and volatile acids produced during fermentation of Tuscan wines fluctuates between 20 and 50 c.c. of N/1 acid per litre (1.80 to 4.20 grms. of tartaric acidity), and is composed mainly of lactic acid (5 to 11 c.c. of N/1 acid), succinic acid (11 to 19 c.c.), and volatile acids (8 to 20 c.c.). The loss of acidity in the course of a year during the conversion of must into wine may vary from 14 to 134 c.c. of N/1 acid (1 to 10 grms. as tartaric acid per litre). The addition of citric, malic, or succinic acid to grape must causes an irregular increase in the amounts of glycerol, extract, ash, and alkalinity of ash of the wine, whilst tartaric acid has the opposite effect. In the case of Tuscan wines it is the usual practice to ferment the must in such a way as to reduce the fixed acidity and to increase the volatile acidity by 0.1 to 0.4 grm. (as acetic acid) per litre. In these wines the free and combined tartaric acid varies from 1.5 to 5 grms. per litre, and rarely exceeds the higher limit. Tartaric acid does not contribute to the formation of lactic acid in the wine, but even checks the formation if present in quantities of 0.2% and over. The tartaric acid in Tuscan wines may amount to as much as 86% of the total fixed acidity. The amount of malic acid in Tuscan grape musts varies as much as the tartaric acid, over which it predominates only in unripe grapes. It is exceedingly soluble in wine, and is readily destroyed by fermentation and by bacteria. Free malic acid in wine does not undergo the malolactic fermentation, but in the form of calcium malate



is rapidly transformed into lactic acid both by yeasts and bacteria. Malic acid added to wine in the proportion of 0.5 to 1.5% increases the fixed acidity and the extract, but may totally disappear during the maturing of the wine, without producing lactic acid. The amount of succinic acid in Tuscan wines varies from about 0.65 to 1.20 grm. per litre; it is formed as a metabolic product of the yeast during the fermentation of the must. It exists for the most part in the free state in the wine, though it tends to form soluble salts. When added to grape must it may be transformed by specific bacteria into lactic acid, even in the presence of sulphur dioxide. Citric acid is present in traces in the grape must and is not produced during fermentation. When added to the must or the wine it comes next to tartaric and succinic acids in its resistance to micro-organisms. It remains largely unaltered in wines containing sulphur dioxide, but in the absence of sulphites is destroyed by bacteria. It does not contribute to the production of lactic acid, but checks the production when present in quantities of about 0.2%. The addition of citric acid to grape must increases the solubility of tartaric acid in the wine, and causes an increase in the amounts of glycerol, extract, ash, and alkalinity of the ash. In practice citric acid is added in association with sulphur dioxide to maintain the acidity of the wine during and after fermentation. Tuscan wines contain from 0.5 to 4 grms. of lactic acid per litre, corresponding to 4 to 49% of the fixed acidity. The larger quantities are due to abnormal fermentation of the sugar, or to bacterial fermentation of the malic and succinic acids and probably also of the glycerol. Neutralisation of part of the acidity of the grape must by means of calcium carbonate promotes the rapid production of lactic acid. Wines containing 1% of total acidity rarely undergo lactic fermentation. The method of fermentation used for Tuscan wines accelerates and increases the production of lactic acid by 0.1 to 0.9 grm. per litre. As the wine matures there is a gradual decomposition of the lactic acid, 50% of which may disappear within a year. Wines rich in alcohol (12% or more) are usually poorer in lactic acid than wines of lower alcoholic strength.—C. A. M.

*Sucrase [invertase]; Law of action of —; hypothesis of an intermediate compound.* H. Collin and A. Chaudun. *Comptes rend.*, 1918, 167, 338—341.

THE results obtained in the hydrolysis of sucrose by varying the relative proportions of sucrose and sucrase are in agreement with Brown's hypothesis (this J., 1902, 419) of the formation of a compound between the sugar and the enzyme. (See further J. Chem. Soc., Oct., 1918.)—W. G.

*Utilisation of rice and its by-products.* See XIXa.

*Examination of fruit juices [raspberry and apple].* Härtel and Silling. See XIXa.

#### PATENTS.

*Yeast; Method of treating brewers' — to render it suitable for baking purposes.* H. W. Anderson, Exeter, W. Lamshead, Paignton, W. Lamshead, jun., Exeter, and J. M. Ramsay, Kilmarnock, Ayrshire. Eng. Pat. 117,666, July 24, 1917. (Appl. No. 10,632 of 1917.)

BREWERS' yeast is treated first with an aqueous solution of borax and an alkali carbonate, and subsequently with a solution of alkali carbonate alone. For example, 25 galls. of yeast, mixed with 60 galls. of water, is sifted and left to stand; after removal of the water the yeast is stirred for about 15 mins.

with 50 galls. of water containing 3 lb. of borax and 10 oz. of sodium bicarbonate and allowed to subside. The liquid is then drawn off and the yeast stirred for 20—30 mins. with 60 galls. of water containing 7 oz. of potassium carbonate or an equivalent amount of other alkali carbonate.—J. H. L.

*Yeast; Preparation of —.* L. Roush, Beaver, Pa. U.S. Pat. 1,271,371, July 2, 1918. Date of appl., Apr. 5, 1917.

YEAST is stimulated by growth in a fluid culture mass into which air is caused to penetrate through a porous body; *e.g.*, pressed yeast is invigorated by introducing it into a wort prepared from malt flour, malt combs, and flour, and allowing air to penetrate through a porous body into the wort.

—J. H. L.

*Wort and other liquids; Aeration and carbonisation of — in connection with counter-current surface cooling apparatus.* P. Robinson, Burton-on-Trent. Eng. Pat. 117,911, May 25, 1917. (Appl. No. 7514 of 1917.)

IN apparatus such as those used for cooling and aerating wort or carbonating beer, in which the liquid and gas are together passed through a tubular system subjected to external counter-current cooling, claim is made to two devices, one connected with the inlet and the other with the outlet of the tubular system. The former consists of a small chamber through which the liquid passes and into which air or gas is injected through minute orifices in the ends of one or more tubes projecting into the chamber. The latter consists of a small chamber the lower part of which is filled with the wort issuing from the tubular system, and this liquid supports a float actuating a snift valve which controls the pressure of air or gas in the liquid by allowing it to blow-off whenever the pressure exceeds the desired value.—J. H. L.

*Grain; Process of treating — for beverages.* B. Leibbrandt, Santa Cruz, Cal. U.S. Pat. 1,269,100, June 11, 1918. Date of appl., Nov. 12, 1917.

WHEAT in the form of whole grains is first softened at about 80° F. (27° C.) until germination commences, next moistened and subjected to a "cooking heat" for about 6 hours until it becomes mellow, then dried by gentle heat, and finally roasted.

—J. H. L.

*Sulphite [cellulose] liquor; Process of fermenting —.* A. Lackman, New York. U.S. Pat. 1,269,287, June 11, 1918. Date of appl., Feb. 26, 1912.

THE waste sulphite liquor is brought to the boil and a quantity of sulphuric acid, *e.g.*, about 0.2%, substantially equivalent to the sulphur dioxide and sulphites present, is added to the hot liquor; the sulphur dioxide liberated is removed in the gaseous form, and the acidified liquor is cooled and fermented.—J. F. B.

*Sulphite [cellulose] liquors; Process of obtaining alcohol from —.* R. H. McKee, Ridgefield Park, N.J. U.S. Pat. 1,273,392, July 23, 1918. Date of appl., Aug. 27, 1917.

YEAST is added to sulphite liquor containing an appreciable amount of free sulphurous acid, the solution being aerated substantially throughout the fermentation, thereby maintaining the fermentative action; alcohol vapours are recovered from the exit gases by scrubbing them with unfermented liquor.—J. F. B.

*Distilling liquid mixtures or [alcoholic] solutions; Method of* —. H. O. V. Bergström, Stockholm, Sweden. U.S. Pat. 1,271,654, July 9, 1918. Date of appl., Jan. 8, 1917.

In the recovery of relatively small proportions of alcohol from an aqueous liquid, the latter, suitably preheated, is introduced into a column still at a point above the lower de-alcoholising portion, in which it is boiled by injection of steam. The alcoholic vapours produced, after mounting through the upper dephlegmating portion of the column, yield their heat (in a tubular cooler) to a cooling liquid (water) which, being maintained under a relatively low pressure, becomes vaporised and thus yields a useful quantity of steam.—J. H. L.

*Beverages; Process of stabilising temperance cereal* —. C. S. Ash, Assignor to Gould and Ash, San Francisco, Cal. U.S. Pat. 1,271,991, July 9, 1918. Date of appl., Nov. 15, 1917.

The beverages are rendered chill-proof and capable of undergoing pasteurisation without deterioration of flavour by treating them, at any stage of their manufacture, with tannin and sulphurous acid or its salts, and removing the precipitate formed thereby. (Cp. U.S. Pat. 1,234,255; this J., 1917, 1059.)—J. H. L.

*Vinegar-making; Process of* —. Apparatus for vinegar-making. E. Klein, Peekskill, N.Y. U.S. Pats. (A) 1,272,276 and (B) 1,272,277, July 9, 1918. Date of appl., Jan. 12, 1916.

(A) The vapours escaping with the spent air from an acetifier working on the Schutzenbach principle are condensed by means of the incoming gyle, which has previously been cooled, and the mixture is then subjected to the action of fresh air. (B) An acetifier is divided by a number of false bottoms into a vertical series of compartments, these false bottoms consisting of strips with spaces between. Means are provided for cooling the gyle, and for making air rise through the false bottoms at a temperature automatically maintained below a predetermined maximum.—C. A. M.

*Alcohol; Process for the manufacture of* — from grain in open vats with the employment of saccharifying mucors. H. Boulard, Paris U.S. Pat. 1,266,657, May 21, 1918. Date of appl., Dec. 7, 1916.

See Eng. Pat. 102,945 of 1916; this J., 1918, 525 A.

*Bread-making process.* U.S. Pat. 1,259,754. See XIXA.

## XIXA.—FOODS.

*Wheat, flour, etc.; Determination of the non-digestible residue in* — by means of pancreatin. L. Devillers. J. Pharm. Chim., 1918, 13, 5—12.

FIVE grms. of the wheat (whole grains) is placed in a thick-walled stoppered flask and covered with N/5 sodium borate solution (38.20 grms. per litre) diluted with three times its volume of water. The flask is immersed in water at 55° C. for 12 hrs., the contents then shaken, the grains collected on a sieve, washed with warm water, returned to the flask, and again heated at 55° C. with 25 c.c. of N/5 sodium borate solution, 3 c.c. of N/1 calcium chloride solution, 2.5 c.c. of 1% pancreatin solution (in chloroform water), and sufficient water to make the volume up to 100 c.c. The mixture is stirred until the grains are disintegrated

(this requires from 1 to 2 hrs.), the flask is then closed securely and heated, first at 75° C. for 30 mins., and then in an autoclave at 120° C. for 15 mins. After cooling to 55° C., 2.5 c.c. of the pancreatin solution is added, and the mixture maintained at 55° C. until all the starch has been hydrolysed (iodine test). Ten c.c. of N/1 hydrochloric acid is then added, the heating at 55° C. continued for 1 hr., and the insoluble residue collected, dried, and weighed. Wheat yields from 9.9 to 11.5% of insoluble matter (calculated on the dry substance), flours 4.8 to 8.2%, bread 7.5%, and bran 35.2%.—W. P. S.

*Maize protein; Efficiency of* — in adult human nutrition. H. C. Sherman and J. C. Winters. J. Biol. Chem., 1918, 35, 301—311.

FEEDING experiments with maize on a young woman are described, which show that nitrogenous equilibrium can be maintained over long periods with a protein intake considerably below that of the Chittenden standard, and of which very much the largest part may be maize protein. This indicates that the protein in maize is sufficient to maintain life even in the case of a low protein diet. Ordinarily, therefore, the substitution of maize products for those of wheat is physiologically justified.

—H. W. B.

*Rice and its by-products; Utilisation of* —. Bull. Imp. Inst., 1918, 16, 16—24.

A NUMBER of samples of Burmese rice have been examined, classified, and analysed, all of which would be suitable for starch manufacture and brewing. In these industries the broken grains are employed, and it is not considered advisable to grow special varieties for the purpose. For starch manufacture a rice of medium hardness with low protein content is preferred. The variation in the percentage of starch is not large, and this percentage is not necessarily an index of the manufacturing yield; variations in the fat content have no influence. "Red skin" is objectionable, as it gives a dark solution when the grain is softened by alkali. Yellowness of the grain, generally caused by heating in transit, is also objectionable. Fully matured rice is preferred as being freer from undersized granules. For brewing, rices with a minimum fat-content and maximum percentage of starch give the best results. *Rice straw for paper-making.* A sample of Egyptian rice-straw has been tested, containing moisture, 11.8; ash, 17.6; cellulose, 50%. The length of the ultimate fibres was 0.6—3.0 mm.; mostly 0.9—1.3 mm. On digestion for 4 hours at 140° C. with 14% of caustic soda the yield of dry pulp was 44%; with 10% of soda, 50%; and with 8% of soda, 52%, all expressed as dry pulp on air dry straw. These pulps all bleached easily to a pale cream colour, almost white. On digestion for 12 hours at 140° C. with 20% of lime, the straw yielded 56% of yellow straw pulp suitable for boards and brown papers. The above experiments showed that a good bleaching pulp could be obtained with an unusually small consumption of soda, but this advantage is partly counterbalanced by losses in the recovery of soda from a material containing such a large proportion of silica. *Rice husks.* The sample examined contained on the dry substance 14.7% of ash and 42% of cellulose; the fibres varied in length from 0.5 to 1.5 mm., being mostly 0.5—0.7 mm. When digested for 4½ hours at 140° C. with 16% of caustic soda, the husks yielded 36% of dry pulp on the air dry weight. The pulp contained a large proportion of gelatinous material and could not be bleached satisfactorily. It gave a very weak and brittle paper, and could not be used in paper-making except as a filler in admixture with pulps of longer fibre.—J. F. B.



*Milk; Correlation between the percentage of fat in cow's — and the yield.* E. Roberts. J. Agric. Res., 1918, 14, 67—96.

THE results of a statistical investigation into the relation between quality and yield of cow's milk are given in the case of American cattle. The breeds studied were Jerseys, Guernseys, Ayrshires, Holstein-Friesians, and unclassified cows. It was found that the yield of milk increased with age up to 5 years (cows over 5 were classed with those of 5). The percentage of fat remained constant, except for the Ayrshires, where there was a decrease with age. Breed had an influence on the variability of the two factors: for variability in yield the order was Holsteins, Ayrshires and Guernseys equal, Jerseys; for variability in fat the order was Jerseys, Guernseys, Ayrshires and Holsteins equal. The total yearly yields were: Holsteins, 14,443 lb.; Ayrshires, 9417 lb.; Guernseys, 8644 lb.; Jerseys, 7491 lb.; unclassified, 5824 lb. The average percentages of fat were: Jerseys, 5.39; Guernseys, 5.03; Ayrshires, 3.93; unclassified, 3.90; Holsteins, 3.43.—J. H. J.

*Milk; Effect of heat on the citric acid content of —. Isolation of citric acid from milk.* H. H. Sommer and E. B. Hart. J. Biol. Chem., 1918, 35, 313—318.

MILK contains about 0.2% of citric acid. The content of citric acid is not affected by heating the milk. The anti-scorbutic property of milk, which is lost by heating, cannot therefore be associated with its content of citric acid. (See further J. Chem. Soc., 1918, i., 465.)—H. W. B.

*Chymosin (rennet) and pepsin; Action of —. IV. Action of the enzymes on sodium caseinogenate.* O. Hammarsten. Z. physiol. Chem., 1918, 102, 23—77.

THE differences in the action of two extracts of gastric mucous membrane containing pepsin and rennet on milk and on fibrin or white of egg are exactly paralleled by their action on sodium caseinogenate in alkaline and acid solution respectively. Thus, if one extract shows relatively greater clotting than digesting power when tested on milk and on fibrin, then it is found that its action on sodium caseinogenate is more marked in alkaline than in acid solution. These facts support the hypothesis which regards rennet and pepsin as two distinct enzymes.—H. W. B.

*Fruit juices [raspberry and apple]; Examination of —.* F. Härtel and J. Sölling. Z. Unters. Nahr. Genussm., 1918, 35, 437—442.

SIX samples of raspberry juice, which had been fermented in the presence of the fruit, then expressed, and filtered, yielded the following results on analysis:—Sp. gr., 1.0139 to 1.0171; alcohol, 1.96 to 3.48; total solids, 3.48 to 5.15; acidity, 1.46 to 2.19; sucrose, none; invert sugar, 0.09 to 0.40; ash, 0.44 to 0.71 gm. per 100 c.c.; alkalinity value, 10.50 to 12.17. A raspberry juice expressed directly had sp. gr. 1.0361; alcohol, none; total solids, 8.48; acidity, 1.17; sucrose, 0.034; invert sugar, 5.54; ash, 0.49 gm. per 100 c.c.; alkalinity value, 10.09. Analyses are also given of 24 raspberry juices which had been treated with various preservatives, kept for 1 year, and then examined. Salicylic acid (1 gm. per litre) gave the best results, then followed hydrofluoric acid (0.5 gm. per litre), benzoic acid (1.5 gm. per litre), and formic acid (2.5 gms. per litre). Seven of the samples had decomposed in spite of the presence of preservative, and four of these had been preserved with formic acid. A sample of apple juice was also examined, the first, last, and main runnings being analysed separately to see whether there was any difference in these portions of the juice; no differ-

ence was found. The results for the main runnings were:—Sp. gr., 1.0506; alcohol, none; total solids, 12.48; acidity, 0.66; sucrose, 1.76; invert sugar, 8.02; ash, 0.28.—W. P. S.

*Sudan-grass hay; Composition and digestibility of —.* W. G. Gaessler and A. C. McCandlish. J. Agric. Res., 1918, 14, 176—185.

THE dry matter of Sudan-grass (*Andropogon sorghum*) hay changes slightly in composition from the time of heading until the crop is ripe. The protein and fat content increase in the early stages of ripening and then decrease, whilst the changes in the nitrogen-free extract (carbohydrates) and ash content are in the opposite direction. There is very little change in the fibre content of the dry matter from the time the Sudan grass heads out until it is fully ripe. Either as a green feed or as hay Sudan grass is very palatable, and the hay has a comparatively high apparent digestibility, the coefficients of digestibility, as determined with two heifers a year and a half old, being dry matter 64.9%, protein 47.4%, nitrogen-free extract 67.8%, fibre 70.6%, ether extract 58.4%. Sudan-grass hay supplies energy to cattle much more efficiently than it does protein as compared with hay from timothy or millet.—W. G.

*Rape straw as a paper and fodder\* material.* Heuser and Blasweiler. See V.

*Cephalin.* V. Hydrocephalin of the egg yolk. Levene and West. See XX.

*Histidine and carnosine. Synthesis of carnosine.* Baumann and Ingvaldsen. See XX.

#### Erratum

*Detection of cane sugar in milk.* G. D. Elsdon (see this J., 1918, 558 a). In lines 4 and 5 of abstract read "3N hydrochloric acid," and for "0.1 gm. of resorcinol" read "0.5 gm."

#### PATENTS.

*Dry milk or milk-powder; Impts. in, and apparatus for, the production of —.* R. R. F. Bntler. Devises. Eng. Pat. 117,713, Aug. 23, 1917. (Appl. No. 12,130 of 1917.)

IN apparatus in which milk is dried on heated cylinders, the milk is kept cool, up to the moment when it comes into contact with the heated cylinder, by circulating cold brine through a jacket surrounding the containing tray and also through the feed rollers.—W. P. S.

*Emulsifying apparatus [for milk].* J. T. Davis. Alameda, Cal., Assignor to J. Rothschild, San Francisco, Cal. U.S. Pat. 1,269,399, June 11, 1918. Date of appl., July 13, 1917.

AN apparatus for emulsifying milk consists of two discs having on their adjacent surfaces a series of concentric circular grooves, the grooves of one disc being eccentric to those of the other; means are provided for revolving the discs and pressing them together.—W. P. S.

*Butter substitutes and the like and the preparation of same.* J. J. A. Talbot, Southsea. Eng. Pat. 118,215, Dec. 13, 1917. (Appl. No. 18,546 of 1917.)

WASTE fat and meat are cut up and shredded and slowly melted, with or without the addition of chopped marrow or other bones, at about 42° C., the material being clarified by the addition of salt at intervals. The melted fat is strained from unmelting particles and impurities and poured into a mixing vessel where it is churned with milk and a binding agent such as white of egg or dried blood. The melting process may be accelerated and

coagulation of the fat on the sides of the melting vessel prevented by the use of a wooden plunger or other squeezing device.—C. A. M.

*Milk-food product and method of making the same.* W. P. M. Grelek, Lincoln, Nebr. U.S. Pat. 1,272,035, July 9, 1918. Date of appl., Dec. 26, 1916.

A PASTEURISED acid milk food product is prepared by subjecting soured milk to the action of a jet of steam whereby the casein is precipitated and when shaken is obtained in the form of minute, mutually non-adherent particles. These particles may be mixed with extract of malt, and the excess fluid removed.—W. G.

*Prepared food. Vegetable milk.* A. S. Burdick and C. Nielsen, Assignors to The Abbott Laboratories, Chicago, Ill. U.S. Pats. (A) 1,273,144 and (B) 1,273,145, July 23, 1918. Date of appl., July 13, 1917.

(A) Soy beans are soaked in water, the water drained off, the beans mashed, and the mash extracted with water. The extract is mechanically separated from the residue, filtered, mixed with a cereal flour gruel which has been malted to produce a syrupy liquid containing dextrins and sugar, and salts as contained in body tissues are added in the desired proportions. (B) Grain is soaked in water, the water drained off, the residual grain mashed, the mash extracted with water, and the extract separated from the unextracted residue.—C. A. M.

*Dietetic food; Apparatus for the production of — and for obtaining fat from animal carcasses and refuse.* G. Zwicky, Geneva, Switzerland. Eng. Pat. 117,852, Aug. 7, 1917. (Appl. No. 11,336 of 1917.)

THE material is disintegrated by heating it under pressure in a steam-jacketed vessel provided with a stirrer; the separated fat is then removed from the surface of the mass, and the latter is dried by stirring it, while hot compressed air is forced through it. The air inlets are at the bottom of the vessel, both at the circumference and at the centre. The dried material is discharged through an opening at the bottom of the vessel.—W. P. S.

*Flour; Treating —.* H. Greville, Birkenhead. Eng. Pat. 117,917, June 28, 1917. (Appl. No. 9274 of 1917.)

IN order to give a greater body and improved pile to the bread from weak or medium wheat mixtures, the sulphates or acid sulphates of sodium, potassium, or ammonium either singly or conjointly, or mixed with other substances, are mixed in powdered or granular form with the flour at the rate of 1—2 oz. per sack of 250 lb. of flour, the exact amount to be added to a particular flour being ascertained by trial bakings.—W. G.

*Bread-making process.* F. P. Siebel, Assignor to Siebel Institute of Technology, Chicago. U.S. Pat. 1,269,754, June 18, 1918. Date of appl., Feb. 15, 1918.

A MASH prepared with malt and water is saccharified, the wort is separated, cooled, mixed with yeast, and, when fermentation has commenced, the mixture is added to the dough. A dough for bread making is prepared from "brewers' grains" in place of other flour, and a fermenting wort in place of ordinary sugar.—W. P. S.

*Food; Prepared — and process for making the same.* X. Kuzmler, Hartsdale, N.Y. U.S. Pat. 1,273,072, July 16, 1918. Date of appl., May 7, 1918.

FOOD is cooked with a liquid until practically all the soluble constituents are dissolved out. The solid portion is then removed and dried, and the solution is evaporated to a volume such as will be absorbed by the solid portion, with which it is then mixed, any necessary flavouring being added, and the whole heated at 160°—210° F. (71°—99° C.) for a time sufficient to sterilise it and dry it without overcooking. The product is packed in moisture-proof containers.—W. G.

*Milk product; Desiccated — and process and apparatus for manufacturing it.* C. H. Campbell, New York. Eng. Pat. 117,276, Apr. 5, 1917. (Appl. No. 4967 of 1917.)

SEE U.S. Pat. 1,233,446 of 1917; this J., 1917, 1025.

*Method of treating brewers' yeast to render it suitable for baking purposes.* Eng. Pat. 117,666. See XVIII.

## XIXB.—WATER PURIFICATION; SANITATION.

*Water; Bacteriological examination of —. Detection of indole-forming bacteria by the bile-dextrose and phenol methods.* L. Bourdet. J. Pharm. Chim., 1918, 18, 42—46.

COMPARISON of the bile-dextrose method proposed by Grysez and Pieret (Presse Med., Oct., 1917) with the ordinary method showed that the results obtained do not agree well with each other in many cases. Of fifty samples of water tested by the methods, concordant results were obtained in twenty cases only; in the others, higher results were obtained sometimes by the bile-dextrose method and sometimes by the phenol method.

—W. P. S.

*Copper; Determination of — in insecticides.* G. S. Jamieson. Chem. and Met. Eng., 1918, 19, 185.

FROM 0.2 to 0.5 gm. of the insecticide is moistened with 10 c.c. of water, and 5 c.c. of sulphuric acid (1:3) added. The mixture is then heated, and, when completely decomposed, most of the free acid is neutralised with ammonia. The solution is heated nearly to boiling, and 10 c.c. of sulphurous acid and 5 to 10 c.c. of a 10% solution of thiocyanate added. The precipitate is allowed to settle, filtered off, washed, dissolved in a mixture of 35 c.c. of concentrated hydrochloric acid and 20 c.c. of water, and 7 c.c. of chloroform added. The solution is then titrated with a potassium iodate solution containing 11.784 grms. KIO<sub>3</sub> per litre, each c.c. corresponding to 0.002 gm. of copper. The titration is done in a stoppered bottle which is well shaken after each addition of iodate.—F. C. Th.

## PATENTS.

*[Sewage] water-purifier. Sewage treatment.* G. Moore, Joplin, Mo., Assignor to The Chemical Process Co., New York. U.S. Pats. 1,271,925 and 1,271,926, July 9, 1918. Date of appl., Oct. 31, 1914.

SEWAGE is passed into a container where it is continuously agitated in presence of previously coagulated sewage solids, and also, if desired, in presence of fuel solids. From this container the sewage is passed into a second container, where the solids



are separated in the lower portion and returned to the first container, whilst the liquid with suspended solids passes away at the top into a third container, where it is filtered or otherwise separated from the suspended solids. The latter may be converted into a fuel.—J. H. J.

*Water-softening apparatus. Means for regulating water-softening apparatus. Means for regulating the feed of chemicals in water-softening apparatus.* C. L. Kennicott, Chicago Heights, Ill., Assignor to The Permutit Co., New York. U.S. Pats. (A) 1,272,052, (B) 1,272,053, (C) 1,272,054, July 9, 1918. Date of appl., Mar. 20, 1916.

(A) THE apparatus consists of a vertical drum, filled except at the top and bottom with a base-exchanging material. A float lies on the top of the material and actuates an air outlet valve, whereby the water entering at the top may pass down as a solid column through the material at a uniform rate. An outlet for the softened water is provided at the bottom. (B) A portion of the raw water is by-passed into a bucket which floats in a tank and controls the outlet of a chemical tank so as to discharge the chemicals into the main stream of raw water. (C) Means are provided for supplying displacing fluid to the chemical tank in an amount which is regulated by the position of the bucket. An overflow for the chemical tank is provided and means for mixing the liquid overflowing with the main stream of raw water.—J. H. J.

*Bacteria destruction by pressure; Method of —.* J. R. Witzel, Philadelphia, Pa., Assignor to Food Sterilization Co. U.S. Pat. 1,269,562, June 11, 1918. Date of appl., Jan. 29, 1917.

FLUID containing cell bodies and organisms in suspension is confined in a vessel, which is exhausted to cause an expansion of the cell bodies, and a pressure of about 75,000 to 100,000 lb. per sq. in. is then applied to break down the bodies. The organisms are destroyed and the cell bodies are ruptured or disintegrated, so that the substances are more intimately diffused throughout the containing fluid.—B. N.

*[Sewage] sludge; Apparatus for pressing activated —.* W. Buckley, Chicago, Ill. U.S. Pat. 1,271,425, July 2, 1918. Date of appl., July 20, 1916.

SLUDGE is automatically delivered at intervals from a container into a vessel from which it is forced into a filter-press, the frequency of the deliveries depending upon the speed of filtration.—C. A. M.

*Insecticides.* J. F. Moseley and E. McCardell, Manchester. Eng. Pat. 117,916, June 9, 1917. (Appl. No. 8252 of 1917.)

TETRACHLOROETHANE or other halogen-substituted hydrocarbon is mixed with naphthalene, and the resulting solution is emulsified with soap or an alkali salt of a sulphonated fatty acid. The product is miscible with water; it may be mixed with diatomaceous earth to form a dusting powder. An alkali borate or silicate may be incorporated with the soap before emulsification.—J. H. J.

*Vermin [rats, mice, etc.]; Substance [3-methylxanthine] for the destruction of —.* Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 305,926, Nov. 5, 1915.

3-METHYLBXANTHINE, with or without addition of other substances, can be used for the extermination of vermin without having any harmful effect on human beings and domestic animals.—L. A. C.

*Water; Apparatus for the softening and purification of —.* J. J. Lassen and V. F. Hjort, London. U.S. Pat. 1,265,346, May 7, 1918. Date of appl., Mar. 12, 1917.

SEE Eng. Pat. 106,514 of 1916; this J., 1917, 905.

*Water and other liquids; Treatment of — by ultra-violet rays.* V. Henri, A. Helbronner, and M. von Recklinghausen, Paris, Assignors to The R.U.V. Co. U.S. Pat. 1,266,803, May 21, 1918. Date of appl., Oct. 31, 1910. Renewed Aug. 13, 1917.

SEE Eng. Pat. 25,509 of 1909; this J., 1910, 1470.

*Sterilising liquids [by means of ultra-violet rays].* M. von Recklinghausen, A. Helbronner, and V. Henri, Paris, Assignors to The R.U.V. Co. U.S. Pat. 1,271,246, July 2, 1918. Date of appl., July 15, 1915.

SEE Addition of Apr. 20, 1912, to Fr. Pat. 453,191 of 1912; this J., 1913, 882.

*Process for the separation of grease from wool scouring or like effluents.* Eng. Pat. 118,332. See V.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*[Ocimum] oils; Aromatic — from the Seychelles.* Bull. Imp. Inst., 1918, 46, 32–35.

*Ocimum gratissimum* grows as a weed in Mahé (Seychelles); distillation of entire shoots yielded 0.1% of a light brown oil having a pronounced odour of cloves with a peculiar, not very pleasant secondary aroma. The characters of the oil were as follow:—Sp. gr. at 15° C., 0.995;  $n_D^{20} = -14.1\%$ ;  $n_D^{20} = 1.526$  at 21° C.; soluble in 0.7 vol. of 80% alcohol at 15° C.; phenols, 62%, principally eugenol. This oil from the Seychelles differed completely from an oil stated to be derived from *O. gratissimum* from the Ivory Coast, which contained 41% of phenols, almost entirely thymol. The oil was not sufficiently pleasant in odour to be of use in perfumery, and in view of the small yield of oil the plant would hardly be capable of being utilised to advantage as a source of eugenol. *Ocimum basilicum* (sweet basil) from the Seychelles yielded 0.21% of an oil having the following characters:—Sp. gr. at 15° C., 0.962;  $n_D^{20} = +0.82^\circ$  at 28° C.;  $n_D^{20} = 1.514$  at 21° C.; acid value, 0.8; ester value, 2.5; nearly soluble in 4 vols. of 80% alcohol but not completely soluble even in 10 vols. These characters agree generally with those of Réunion sweet basil oil. The Seychelles oil appeared to contain anethol as well as methylchavicol. The commercial prospects of this oil are not considered encouraging. —J. F. B.

*Ajowan seed and thymol.* Bull. Imp. Inst., 1918, 16, 30–32.

AJOWAN seed is derived from an annual herb, *Carum copticum*, closely related to the ordinary caraway. It is grown mostly in India, and the volatile oil of the seeds is used as the principal source of thymol. Samples of seeds from experimental crops grown in the Seychelles and Montserrat have been examined. The Seychelles seed had a poor appearance, a large proportion being small and immature. The seed yielded approximately 9% of oil containing about 28% of thymol; Indian ajowan seed yields only 3–4% of oil containing 40–55% of thymol. Consequently, although the Indian oil is

richer, the yield of thymol from the Seychelles seed is higher than from Indian seed. The Montserrat seed yielded 3.1% of oil containing 51% of thymol; the oil had sp. gr. 0.925 at 15°/15° C., and  $\alpha_D = +0.90^\circ$ . The seed was harvested six months after sowing and showed a yield of 1128 lb. per acre. The result is considered to be encouraging, but the distillation of the oil should be carried out in the country of origin.—J. F. B.

*Terpenes and essential oils.* CXX. O. Wallach, K. Pelikan, H. Berthold, L. Augspurger, H. Worlitzer, and F. Pohle. *Annalen*, 1917, **414**, 195—233.

The greater part of the work has been already published. The expectation, justified by the accepted formulae of pinol hydrate and dihydrocarveol, that the reduction product of the former will be identical with the menthane-2,8-diol obtained by the addition of water to the latter, is not borne out by experiment: both with the optically active and inactive forms, the products in the two cases are quite dissimilar. When pinol hydrate is reduced by hydrogen and colloidal palladium in the absence of free acid, dehydration occurs to some extent and tetrahydrocarveol is obtained as a by-product. This furnishes a further proof that a hydroxyl group occurs in position 2 in pinol hydrate. Terpinene dihydrobromide is formed by the action of hydrogen bromide in glacial acetic acid on *i* or *l*-dihdropinol hydrate. Dihdropinol hydrate is not identical with menthane-1,4-diol or with menthane-2,4-diol, obtained by reducing sabinene-glycol by Paal's method. The latter diol has b.pt. 135°—140° C. at 9 mm., and when dehydrated by oxalic acid and water yields an alcohol,  $C_{10}H_{17}OH$ , b.pt. 219°—221° C., sp. gr. 0.9250 at 22° C.,  $n_D = 1.4790$ , which is probably  $\Delta^3$ -menthene-2-ol containing a little  $\Delta^1(3)$ -menthene-2-ol. (See also J. Chem. Soc., Oct., 1918.)—C. S.

*Terpenes and essential oils.* CXXI. *Pulegenic acid and the conversion of carvone into pulegenic acid.* O. Wallach and W. von Rechenberg. *Annalen*, 1917, **414**, 233—243.

An improved method of preparing pulegenic acid from pulegone dibromide by the action of aqueous potassium hydroxide is described. The conversion of pulegenic acid into dihydropulegenic (1-methyl-3-isopropyl-cyclopentane-2-carboxylic) acid, which cannot be effected by direct reduction of the acid or of its amide, has been accomplished indirectly through methyl hydrochloropuleginate. The amide of the reduced acid is converted by Hofmann's method into a base, presumably 2-amino-1-methyl-3-isopropyl-cyclopentane. The conversion of the 6-ring in carvone into the 5-ring of pulegenic acid and the formation of this acid from dihydrocarvenolic acid have been effected. (See also J. Chem. Soc., 1918, i., 428.)—C. S.

*Terpenes and essential oils.* CXXII. *Behaviour of the nitrosochloride additive compounds towards reagents which eliminate hydrogen chloride.* O. Wallach, J. Wolff, and M. Walter. *Annalen*, 1918, **414**, 257—270.

An excellent method for converting a nitrosochloride into an oxime is to heat it with a mixture of acetone and pyridine on the water-bath for 10—15 minutes; for example, limonenitrosochloride is thus converted into carvoxime. In the case of certain nitrosochlorides hydrogen chloride is eliminated in different ways by different reagents. Thus  $\alpha$ -terpineolnitrosochloride is converted into 8-hydroxy- $\Delta^6$ -menthene-2-oneoxime by the preceding method, but when heated with anhydrous sodium

acetate and glacial acetic acid it yields a crystalline substance,  $C_{10}H_{17}O_2N$ , m.pt. 139° C., which is regarded as an oxime-oxide.  $\beta$ -Terpineolnitrosochloride does not yield a well-characterised oxime. The crystalline substances obtained by the action of methyl or ethyl alcohol on the nitrosochloride and recrystallised from either of the two alcohols are probably mixtures. (See also J. Chem. Soc., 1918, i., 439.)—C. S.

*Terpenes and essential oils.* CXXIII. *Behaviour of dibrominated hexacyclic ketones in relation to the position of the bromine atoms.* O. Wallach, H. E. Woodman, W. Jessen, A. Hallsteln, and E. Grote. *Annalen*, 1918, **414**, 271—296.

When dibromo derivatives of menthone and of carvomenthone are shaken for 2—3 days at the ordinary temperature with 2% aqueous potassium hydroxide, the products may be monobromides, phenols, aliphatic ketonic acids, or pentacyclic hydroxy-carboxylic acids according to the positions of the bromine atoms. Rules are formulated correlating these positions and the nature of the product. For example, 1,2-dibromomenthone is converted into *m*-cresol, and 1,6-dibromocarvomenthone into  $\epsilon$ -keto- $\beta$ -isopropylheptonic acid. (See also J. Chem. Soc., 1918, i., 440.)—C. S.

*Terpenes and essential oils.* CXXIV. *Method of transforming hexacyclic ketones into cyclopentanones based on the conversion of dibrominated cyclohexanones into pentacyclic  $\alpha$ -hydroxy-carboxylic acids.* O. Wallach, M. Gerhardt, and W. Jessen. *Annalen*, 1918, **414**, 296—366.

The dibrominated cyclohexanones which are converted into hydroxycyclopentanecarboxylic acids by aqueous alkali hydroxide (see preceding abstract) are in general easily obtained by treating the cyclohexanone in glacial acetic acid solution with four atoms of bromine. The first products of the interaction with the alkali to be isolated are substances which have the composition of *o*-diketones but behave like unsaturated ketols. The hydroxycyclopentanecarboxylic acids yield cyclopentanones by oxidation with lead peroxide and sulphuric acid. (See also J. Chem. Soc., 1918, i., 442.)—C. S.

*Terpenes and essential oils.* CXXV. *Compounds of the eucarvone series.* O. Wallach and M. Standacher. *Annalen*, 1918, **414**, 367—375.

By reduction with sodium and alcohol  $\beta$ -dihydroeucarvoxime yields tetrahydroeucarvylamine,  $C_{10}H_{19}NH_2$ , b.pt. 208.5° C., sp. gr. 0.8680 at 21° C.,  $n_D = 1.4665$ , several derivatives of which are described. By bromination in glacial acetic acid tetrahydroeucarvone yields, at 0° C., a monobromoderivative,  $C_{10}H_{17}OBr$ , m.pt. 32° C., or, without cooling, a dibromo-derivative,  $C_{10}H_{16}OBr_2$ , m.pt. 68° C. When treated with aqueous potassium hydroxide the former is converted into hydroxy-tetrahydroeucarvone,  $HO.C_{10}H_{17}O$ , b.pt. 98° C. at 12.5 mm., sp. gr. 0.9810 at 20° C.,  $n_D = 1.4626$ , and the latter into a saturated acid,  $C_{10}H_{16}O_2$ , m.pt. 91.5°—92.5° C., the constitution of which has not yet been determined. (See also J. Chem. Soc., 1918, i., 444.)—C. S.

*Elemicin and isoelemicin; Synthesis of —.* F. Mauthner. *Annalen*, 1917, **414**, 250—255.

2,6-DIMETHOXYPIENYL allyl ether,  $C_9H_{12}(OCH_3)_2.O.C_3H_5$ , a colourless oil, b.pt. 140°—141° C. at 14 mm., prepared from allyl bromide and pyrogallol 2,6-dimethyl ether in boiling acetone in the presence of powdered potassium carbonate, is con-



verted at 220° C. into 2,6-dimethoxy-4-allylphenol,  $\text{HO.C}_6\text{H}_2(\text{OCH}_3)_2\text{CH}_2\text{CH}:\text{CH}_2$ , b.pt. 168°–169° C. at 11 mm., the methylation of which yields 3,4,5-trimethoxyallylbenzene, identical with elemicin. The position of the allyl group is proved by oxidising the substance to gallic acid trimethyl ether, and of the double linking by decomposing the ozonide with water, whereby homogallaldehyde trimethyl ether is produced. Synthetic elemicin is converted into isoelemicin (3,4,5-trimethoxypropenylbenzene) by warming with alcoholic potassium hydroxide; the ozonide of the latter yields gallaldehyde trimethyl ether on treatment with water.—C. S.

*Ginger; Pungent principles of* —. II. H. Nomura. Sci. Rep. Tohoku Imp. Univ., 1918, 7, 67–77.

THE pungent principles of Japanese ginger on distillation yield, in addition to zingerone (this J., 1917, 1062), a substance, shogaol,  $\text{C}_{15}\text{H}_{20}\text{O}_5$ , b.pt. 231°–238° C. at 15.5 mm., and sp. gr. 1.0448 at 25° C.,  $n_D^{25}=1.52467$ ; this contains a methoxyl radicle and also a hydroxyl group, the presence of the latter being demonstrated by the conversion of shogaol into monomethyl, monoethyl and monoacetyl derivatives. The presence of a ketonic group is also probable and reduction with hydrogen and platinum black yields a dihydro-derivative, thus proving shogaol to contain one double bond. (See also J. Chem. Soc., 1918, i., 446.)—D. F. T.

*Phenolic ketones; Synthesis of some — and their tastes.* H. Nomura and F. Nozawa. Sci. Rep. Tohoku Imp. Univ., 1918, 7, 79–92.

IN order to decide on what constitutional factors the pungent taste of zingerone (4-hydroxy-3-methoxyphenylethyl methyl ketone) depends, an examination has been made of a number of phenylethyl methyl ketones containing hydroxyl or methoxyl groups substituted into the aromatic nucleus and also of 4-hydroxy-3-methoxyphenylethyl phenyl ketone. The results indicate that of the substances thus tested, those having a free hydroxyl group in the para position to the ketonic side chain, especially those derived from vanillin, have a pungent taste similar to zingerone. The degree of pungency is considerably influenced by the position of the hydroxyl group relative to the ketonic side chain, and is increased by the presence of a meta-methoxyl group. The influence of the simple hydrocarbon radicle attached to the ketonic group is little greater than that of the position of the hydroxyl radicle because 4-hydroxy-3-methoxyphenylethyl phenyl ketone resembles zingerone in taste. (See also J. Chem. Soc., 1918, i. 438.)

—D. F. T.

*Cephalin. V. Hydrocephalin of the egg-yolk.* P. A. Levene and C. J. West. J. Biol. Chem., 1918, 35, 285–290.

HYDROCEPHALIN has been isolated in an almost pure state from the preparation of hydrolecithin in which it was detected as an impurity (see this J., 1918, 136A). (See further J. Chem. Soc., 1918, i., 421.)—H. W. B.

*Histidine and carnosine. Synthesis of carnosine.* L. Baumann and T. Ingvaldsen. J. Biol. Chem., 1918, 35, 263–276.

CARNOSINE is shown to be  $\beta$ -alanylhistidine by synthesis from histidine and  $\beta$ -iodopropionyl chloride as well as by deamination, followed by hydrolysis, when a 70% yield of histidine is obtained. The isomeric histidyl- $\beta$ -alanine could

not be prepared. Carnosine is not hydrolysed by extracts of dog's liver or muscle. (See further J. Chem. Soc., 1918, i., 454.)—H. W. B.

*Foaming; Inhibition of* — [by isoamyl isovalerate]. C. H. Fiske. J. Biol. Chem., 1918, 35, 411–413.

ISOAMYL ISOVALERATE is recommended as a foam inhibitor in analytical operations, and two methods are described for its preparation: (1) by heating a mixture of isoamyl alcohol and isovaleric acid in the presence of concentrated sulphuric acid, and (2) by oxidising isoamyl alcohol with potassium bichromate and sulphuric acid. (See further J. Chem. Soc., Oct., 1918.)—H. W. B.

*Aldehydes; Reduction of* — to corresponding alcohols. I. Reduction of heptole aldehyde (ananthol). P. A. Levene and F. A. Taylor. J. Biol. Chem., 1918, 35, 281–283.

THE reduction is effected by an emulsion of metallic sodium in toluene. The calculated quantity of sodium is suspended in dry toluene, heat applied until the sodium melts, the liquid stirred vigorously in order to form a fine emulsion, and then cooled quickly in ice, the stirring being continued. The aldehyde, dissolved in glacial acetic acid and toluene, is then added slowly, and when the reduction is finished, water is added to dissolve the sodium acetate, and the toluene layer separated and fractionally distilled. The yield of heptole alcohol from ananthol was 58% of that theoretically obtainable.—H. W. B.

#### PATENTS.

*Ethylidene diacetate; Manufacture of* —. Soc. Chim. des Usines du Rhône, anc. Gilliard, P. Monnet, et Cartier, Paris. Eng. Pat. 112,766, Oct. 29, 1917. (Appl. No. 15,756 of 1917.) Under Int. Conv., Dec. 9, 1916.

ACETYLENE is converted into ethylidene diacetate by passing the gas into a solution of 40 parts of mercuric oxide and 20 parts of methylene sulphate in 1100 parts of glacial acetic acid at 50°–70° C., the current of gas being so regulated that absorption is complete and being continued until 200 parts have been fixed. Other alkyl sulphates may be used in place of methylene sulphate.—D. F. T.

*Alkylating reagents; Preparation of certain* —. J. C. Irvine and W. N. Haworth, St. Andrews. Eng. Pat. 117,824. (Appl. Nos. 5337, Apr. 20, and 12,885, Sep. 8, 1917.)

By the action of sulphuryl chloride on methyl or ethyl alcohol a mixture of the corresponding dialkyl sulphate and alkyl hydrogen sulphate is obtained. The reactions may be represented  $2\text{R.OH} + \text{SO}_2\text{Cl}_2 = \text{HO.SO}_2\text{OR} + \text{RCl} + \text{HCl}$  and  $2\text{R.OH} + \text{SO}_2\text{Cl}_2 = \text{SO}_2(\text{OR})_2 + 2\text{HCl}$ . The sulphuryl chloride may be prepared separately and used in the crude or rectified condition, small quantities being added to the alcohol as long as the desired reaction occurs, but care is necessary to avoid exceeding 5% above the semimolecular proportion of the chloride. It is advantageous to retain in the reaction system the alkyl chloride produced, because this tends, by mass action, to reduce the formation of alkyl hydrogen sulphate. Instead of using pre-formed sulphuryl chloride, equimolecular quantities of chlorine and sulphur dioxide may be passed into the alcohol, or the gases may be introduced alternately, care then being taken to avoid the chlorine being present in an excess of the molecular proportion of the sulphur dioxide. On distillation of the final reaction product under reduced pressure the

alkyl hydrogen sulphate undergoes decomposition into sulphuric acid and dialkyl sulphate, so that the latter is obtained in good yield.—D. F. T.

*Photochemical [chlorinating] apparatus.* W. O. Snelling, Pittsburgh, Pa. U.S. Pat. 1,271,790, July 9, 1918. Date of appl., July 1, 1914. Renewed Jan. 3, 1918.

The apparatus consists of a reaction chamber, the side walls of which are impervious to light, carrying a series of parallel plates pervious to light which are so arranged as to afford a continuous passageway for gases (chlorine and substances to be chlorinated) around the plates from the inlet at one end of the chamber to the outlet at the other. The arrangement is such that light, directed on the apparatus, will act with greatest actinic effect on the gases nearer the outlet and with less actinic effect on the gases nearer the inlet, there being a means for regulating the temperature of the reacting gases passing through the passageway.—W. G.

*Protoid silver; Preparation of* —. Y. Nukada, Tokyo, Japan. U.S. Pat. 1,272,987, July 16, 1918. Date of appl., Sep. 21, 1917.

THIRTY pounds of "natio" is added to 12 gallons of water and the whole heated for six hours at 100° C., the liquid then being filtered and evaporated on a water bath to a volume of 9 cub. m. After standing 12 hours the solution is filtered and to it is added 175 grms. of silver nitrate.—W. G.

*Caffeine; Process of recovering* —. L. Fontanelli, Ferrara, Italy. U.S. Pat. 1,273,054, July 16, 1918. Date of appl., Aug. 27, 1917.

Soot produced in the roasting of coffee is heated in a restricted current of gas to vaporise the caffeine, and the gas is then cooled to condense the caffeine.—W. F. F.

*Mercury compound; Preparation of an organic* —. R. Lüders, Berlin-Steglitz. Ger. Pat. 306,198, May 5, 1916.

$\alpha$ -AMINO BUTYRIC acid in slightly alkaline solution is treated with mercuric oxide. The resulting compound is easily soluble in water and shows a faintly acid reaction; part of the combined mercury is not precipitable by the usual reagents.—J. F. B.

*Mercury preparations for therapeutic purposes; Process of producing* —. H. Bachtala, Gratz, Austria, Assignor to Dr. Bayer es Tarsa, Budapest. U.S. Pat. 1,271,846, July 9, 1918. Date of appl., Aug. 18, 1915.

SEE Ger. Pats. 267,411 and 267,412, and Eng. Pat. 11,302 of 1913; this J., 1914, 220, 767.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Ammonium persulphate; Effect of the iron content of* — on its photographic reducing power. S. E. Sheppard. Communication No. 60 from Eastman Kodak Research Lab. Brit. J. Phot., 1918, 65, 314–315.

The reaction between silver and ammonium persulphate is very slow, the reactions between silver sulphate and ammonium persulphate and between silver persulphate and metallic silver being much more rapid. The reducing action of a pure persulphate bath is therefore very slow in starting,

but the solution can be activated by the addition of small quantities of silver solvents such as silver sulphate or ferric sulphate. The variation in the initial activities of different samples of persulphate is due to the variable iron content; if this rises above 1 to 2 parts of iron per 1000 of persulphate, the resulting solution is too active to be under proper control. The fact that the reaction product (silver sulphate) increases the activity of the persulphate solution explains the greater proportional effect of the reducer on high densities.—B. V. S.

### PATENTS.

*Coloured photographic image and method of producing the same.* Hess-Ives Corporation, Assignees of F. E. Ives, Philadelphia, Pa. Eng. Pat. 113,617, Feb. 19, 1918. (Appl. No. 2910 of 1918.) Under Int. Conv., Feb. 20, 1917.

A RED image is obtained by copper-toning a black silver image in, for instance, a copper-ferricyanide-citrate toning bath, removing the silver salt, which is formed in the toning, by treatment with thio-sulphate, and then treating with a solution of a dye or dyes, e.g., Fuchsian and Auramine, to which copper acts as a mordant.—B. V. S.

*Natural colour cinematography.* L. F. Douglass, San Rafael, Cal., U.S.A. Eng. Pat. 117,864, Oct. 19, 1917. (Appl. No. 15,226 of 1917.)

ALTERNATING screens of two complementary colours, e.g., red and blue-green, are used in taking the negative, from which a black and white positive is taken in the usual way. The positive is then stained or toned in suitable baths in two processes, one of the alternating sets of pictures being masked, e.g., by coating with a spirit-soluble varnish, while the other set is being treated.—B. V. S.

*Colour photography.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,268,847, June 11, 1918. Date of appl., Jan. 15, 1917.

A TWO-COLOUR mosaic taking-screen is employed, the two components being respectively complementary to two of the primary colours. This is provided with a sensitive film sensitised to these two primary colours, which in turn is placed in contact with a second film sensitive to the third primary colour and stained to prevent action of the first two primary colours. Exposure is made through the mosaic screen. In printing from the two negatives thus obtained, two positives are made from the one negative and one from the other and the three positives are combined.—B. V. S.

*Photographs; Method of and article for making* —. J. G. Capstaff, Assignor to Eastman Kodak Co., Rochester, N.Y. U.S. Pat. 1,273,457, July 23, 1918. Date of appl., Jan. 6, 1917.

A SENSITISED gelatin film is applied to a transparent support first provided with a separating opaque silver screen obtained by a preliminary sensitive coating which has been exposed, developed, etc., in the usual way before applying the second film. The support may be provided with a sensitised coating on the other side also, the opaque screen then acting as a protection to each film during exposure of the other. After exposure through a colour-sensation record and development, etc., the film is treated with a bleaching-hardening bath and a suitable dye bath. The back film is bleached and hardened along with the exposed parts of the upper film, so that only the unexposed parts of the latter are stained.—B. V. S.



*Tellurium toning-bath and process of toning photographic elements.* E. R. Bullock, Assignor to Eastman Kodak Co., Rochester, N.Y. U.S. Pat. 1,273,797, July 23, 1918. Date of appl., Apr. 5, 1916.

A SEPIA tone is produced on a silver image by treatment in a bath containing tellurium chloride, hydrochloric acid, sodium chloride, and potassium bromide. The resulting image may be intensified and the tone modified by further treatment in an aqueous solution of sodium sulphide.—B. V. S.

*Treatment of textile fabrics, wood, leather, and other materials.* Eng. Pat. 117,640. See V.

## XXII.—EXPLOSIVES; MATCHES.

*Smokeless powder; Detection of acidity in —.* A. Angeli. Atti R. Accad. Lincei, 1918, 27, I., 164–166.

ALL the known methods of determining the stability of smokeless powder exhibit disadvantages and the author proposes to replace them by the use of the indicator, dimethylaminoazobenzene. The latter forms red salts, which become fixed to the surface of the explosive like a dyestuff to a fibre. A few c.c. of distilled water containing three or four drops of a 0.2% alcoholic solution of the indicator is poured on to about 0.5 gm. of the powder, the whole being well stirred. Powder of good quality turns lemon-yellow, whereas if more or less acidity is present, it becomes more or less intensely red; in either case, the supernatant liquid remains completely colourless. Not all explosives which are acid show low stability when subjected to the heat test, but experiment demonstrates that they inflame far more readily than normal explosives when kept in large amount at a relatively high temperature, and they also burn irregularly and incompletely, and in the firing test exhibit abnormalities in the pressure and range. A dilute solution of the above indicator may be used for revealing the effect of light on smokeless powder; the parts of the latter which have been exposed are turned red by the solution, whereas the other parts become yellow.—T. H. P.

*Source of error in Bunsen's method for determination of chlorates, peroxides, etc.* Rupp. See VII.

### PATENTS.

*Nitro-compounds [trinitrotoluene]; Method of separating — from liquids.* F. M. G. Johnson, Montreal, Canada. U.S. Pat. 1,271,578, July 9, 1918. Date of appl., Sep. 18, 1917.

SPENT acid containing trinitrotoluene is confined at pressures below atmospheric until the solid particles are floated to the surface by adhering gas bubbles. The cleared acid is drawn off from below the floating mass, and the latter is then heated sufficiently to melt the trinitrotoluene.—T. St.

*Method of producing ammonium perchlorate.* U.S. Pat. 1,273,477. See VII.

## XXIII.—ANALYSIS.

*Analytical results; Calculation of —.* J. Grossfeld. Chem.-Zeit., 1918, 42, 389–391.

FACTORS are given in tabular form for calculating results from analytical data. The tables include factors for many elements, inorganic and organic compounds and acids, sugars, alkaloids, etc. (See also J. Chem. Soc., Oct., 1918.)—W. P. S.

*Colorimeter; New form of —.* J. C. Bock and S. R. Benedict. J. Biol. Chem., 1918, 35, 227–230.

IN the new instrument, the prisms employed in the Duboscq colorimeter are replaced by mirrors, and there is only one plunger, a cell of known dimensions being substituted for the other. (See also J. Chem. Soc., Oct., 1918.)—H. W. B.

*Gases; Apparatus for measurement of — in volumetric analysis.* H. Fincke. Chem.-Zeit., 1918, 42, 415.

A 200–250 c.c. graduated cylinder is closed by a rubber stopper carrying a siphon with arms of equal length, one of which reaches nearly to the bottom of the cylinder and the other nearly to the bottom of a taller and wider cylinder, and a tube bent at an angle of 45° and connected to a reaction flask containing a weighed quantity of the sample sufficient to yield about 100–150 c.c. of gas. The acid or other reagent is contained in a flask which is connected to the reaction flask by means of a tube bent at right angles. The graduated cylinder and the siphon are filled with a saturated solution of common salt. The flask containing the reagent is inverted and the latter then flows into the reaction flask, the gas evolved expelling a corresponding volume of salt solution from the first into the second graduated cylinder. In this apparatus the use of india-rubber tubing and stopcocks is avoided. (See also J. Chem. Soc., Oct., 1918.)—A. B. S.

*Gas analysis; Use of the interferometer in —.* F. M. Seibert and W. C. Harpster. U.S. Bureau of Mines, Tech. Paper 185, 1918. 18 pages.

IN special work requiring analysis of gases at frequent intervals, the Rayleigh interferometer, as adapted to gas analysis by Haber and Lowe (this J., 1906, 802), has partly supplanted the older analytical methods. It is suitable for analysing binary gas mixtures such as occur in mines, in the production of oxygen from liquid air, and in the commercial production of hydrogen. For accurate determinations the gas chambers consist of a cylindrical metal tube 1 metre long, and divided lengthwise by a metal partition, the upper half containing the gas to be tested, while the lower section is filled with a gas used for comparison. Parallel rays from a Nernst lamp pass through the object glass of a collimator and travel in three parts to an observation telescope, producing an image of the slit with diffraction bands. The lower half of the pencil of rays is divided into two parts by the metallic partition between the gas chambers, each part also passing through a compensator plate, the upper part of which is fixed and the lower part capable of movement by means of a micrometer screw. Thus the optical path of the gas in the lower section of the gas chamber can be varied. When both chambers are filled with the same gas, the interference effect produced by the lower half of the pencil of rays is similar to that produced by the upper half, but if a difference in the gas composition in the two sections exists, the lower band image is moved laterally and can be brought back to a zero position by manipulation of the compensating plate. The measure of the movement of the compensator is a measure of the refractive difference between the two gases, which must be dry and at the same temperature. In mine gases a decrease in the amount of oxygen and a corresponding increase in the amount of nitrogen as compared with atmospheric air would show imperfect ventilation. Among other technical uses the instrument may be used to detect contamination of hydrogen with

heavy gases, and the degree of carburisation of illuminating gas carburetted with benzene. (See also J. Chem. Soc., Oct., 1918.)—C. A. K.

*Volatile substances; Manipulation of —.* III.  
A. Stock. Ber., 1918, 51, 983–989.

THE following pieces of apparatus are fully described. I. An improved form of gas holder for substances which can be condensed by liquid air (see Ber., 1917, 50, 1006). II. (a) An arrangement for filling a tube with a gas or mixture, and sealing it, without exposure to the air, and (b) a "vacuum tube opener" for breaking the capillary of a sealed tube and leading off the gases without exposure to the air. III. A simple apparatus for analysing a gaseous mixture by measuring the pressure of the gas left uncondensed at different temperatures. (See further, J. Chem. Soc., Oct., 1918.)—J. C. W.

[Acidity:] *A new physico-chemical volumetric method [for determining —].* R. Dubrisay. Ann. Chim., 1918, 9, 25–18.

THE method is based on a determination of the number of drops formed when a definite volume of vaseline oil mixed with stearic acid is allowed to flow slowly into a given volume of the liquid, of which it is required to determine the acidity, before and after the addition of increasing amounts of standard alkali. It is found that there is a sharp rise in the number of drops formed when the liquid is just neutralised.—W. G.

*Tungsten and vanadium; Determination of — in the presence of titanium.* G. Fenner. Chem.-Zeit., 1918, 42, 403.

WHEN a mineral, etc., containing tungsten and titanium is fused with sodium peroxide, the melt then treated with water, boiled for a few mins., and filtered, the titanium remains insoluble in the iron precipitate whilst the tungsten is completely soluble and is found in the filtrate. Vanadium, if present in small quantity (less than 0.5%), remains almost entirely insoluble, but in larger quantity a portion is found in the filtrate. To determine vanadium in the presence of titanium, the mineral is treated with hydrofluoric acid and sulphuric acid to remove silica, the residue is then fused with sodium pyrosulphate, the melt dissolved in dilute sulphuric acid, the solution filtered, and the filtrate treated with hydrogen peroxide until a distinct yellow coloration has developed, a large excess of peroxide being avoided. Ammonium fluoride or hydrofluoric acid is then added, whereby the yellow colour of the titanium compound is destroyed, whilst the red-brown coloration of the vanadium compound remains unaffected. Phosphoric acid is added to remove colour due to iron salts, and the vanadium is determined colorimetrically by comparison with a standard prepared with the same quantities of reagents. (See also J. Chem. Soc., Oct., 1918.)—W. P. S.

*Mercury; A new method of estimating — by means of zinc filings.* M. François. Ann. Chim., 1918, 10, 84–100.

A MORE detailed account of work already published (see this J., 1918, 445 A).—W. G.

*Quick determination of incombustible matter in coal and rock dust mixtures in mines.* Fieldner and others. See 11A.

*Laboratory determination of explosibility of coal dust and air mixtures.* Clement and Lawrence. See 11A.

*Determination of ash of coal.* Mastbaum. See 11A.

*Apparatus for measuring the loss [of heat] up the chimney and the elements constituting this loss.* Chopin. See 11A.

*Modification of benzol method of water determination in mineral oils.* Fuchs. See 11A.

*Differentiation of natural and artificial silks.* Herzog. See V.

*Determination of chlorine in mixtures containing silicates.* Brühns. See VII.

*Source of error in Bunsen's method for determination of chlorates, peroxides, etc.* Rupp. See VII.

*Estimation of nitrates and nitrites.* Strecker. See VII.

*Quantitative analysis of vanadium compounds with the aid of carbon tetrachloride.* Jannasch and Harwood. See VII.

*Neutralisation of hydrogen peroxide with sodium borate.* Cambe and Diacono. See VII.

*Silicon hydrides. V. Decomposition of silicon hydrides by water. Action of hydrogen bromide on monosilane.* Stock and Somieski. See VII.

*Determination of zirconium in ferrozirconium and steel.* Ferguson. See X.

*Rapid method of estimating phosphorus in bronzes.* Rooney. See X.

*Hardness and viscosity of varnishes.* Britten. See XIII.

*Researches on certain "soil sicknesses."* Sölmgen and others. See XVI.

*Determination of the non-digestible residue in wheat, etc., by means of pancreatin.* Devillers. See XIXA.

*Bacteriological examination of water. Detection of indole-forming bacteria by the bile-dextrose and phenol methods.* Bourdet. See XIXB.

*Determination of copper in insecticides.* Jamieson. See XIXB.

*Inhibition of foaming [by isoamyl isovalerate].* Fliske. See XX.

*Detection of acidity in smokeless powder.* Angeli. See XXII.

PATENTS.

*Vapours in gases; Process for the quantitative estimation of —.* H. Simmonds and M. D. Davis, Winnipeg, Canada. U.S. Pat. 1,272,922, July 16, 1918. Date of appl., Oct. 15, 1917.

THE process consists in filling to the same pressure two flasks connected by a manometer, the one with the gas to be analysed and the other with an inert



gas free from the vapour in question, and breaking inside each flask a sealed glass bulb, containing a liquid of the same composition as the vapour to be estimated in quantity larger than that required to saturate with the vapour, at that temperature, the gas in the flask in which it is broken. The original concentration of the vapour in the gas is calculated from the differential pressure which develops between the two flasks.—W. G.

*Impact testing machines.* W. & T. Avery, Ltd., and E. A. Allcut, Birmingham. Eng. Pat. 118,175, Sep. 22, 1917. (Appl. No. 13,635 of 1917.)

*Testing gases; Method and apparatus for* —. F. Haber and R. Leiser, Berlin-Dahlem, Germany. U.S. Pat. 1,269,599, June 18, 1918. Date of appl., Apr. 30, 1914.

SEE Eng. Pat. 9344 of 1914; this J., 1915, 926.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Bigot. Tunnel furnaces. 15,255. Sep. 19. (Fr., Nov. 8, 1917.)

Gal. Furnaces. 15,265. Sep. 19. (Fr., Jan. 12.)

Garbutt. Rotary furnaces. 15,683. Sep. 26.

Gloss. Drying-plant. 15,031. Sep. 16. (Fr., Apr. 17, 1917.)

Imperial Trust for Encouragement of Scientific and Industrial Research, and Truscott. Apparatus for separating materials of varying specific gravities. 15,397. Sep. 21.

Kestner. Apparatus for enabling extracts, infusions, or decoctions to be obtained in a continuous manner. 15,397. Sep. 21.

Mead. Heating-furnaces. 15,074. Sep. 17.

Sarron and Simon. Producing changes of physical state or endothermic chemical reactions. 15,542. Sep. 24.

Smallwood. Apparatus for cooling liquids or subjecting them to the action of air or gas. 15,281. Sep. 20.

Soc. l'Air Liquide. Apparatus for effecting exothermic chemical syntheses. 15,514. Sep. 24. (Fr., Feb. 20.)

Walsh. Obtaining hot air for heating or drying. 15,140. Sep. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

3866 (1917). Bontemps and Vis. Apparatus for the continuous separation of solids from liquids. (118,855.) Sep. 25.

13,021 and 13,022 (1917). Nicholas. Separation of liquids from solids. (110,349 and 110,350.) Sep. 25.

14,217 (1917). Urquhart. Evaporating and distilling machinery. (119,106.) Oct. 2.

14,251 (1917). Bowler and others. See XIX.

18,093 (1917). Clarke. Drying apparatus. (119,156.) Oct. 2.

18,459 (1917). Candlot. Ball mill. (115,223.) Sep. 25.

6614 (1918). Alexander. Apparatus or kilns for drying granular, pulverulent, or pasty materials. (119,198.) Oct. 2.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING

#### APPLICATIONS.

Alchevsky. Artificial fuel. 15,774. Sep. 27.

Beilby. Carbonisation of coal, shale, peat, etc. 15,599. Sep. 25.

Benton. Treatment of shales, cannel coal, and organic refuse to recover oils, fats, ammonia, etc. 15,276. Sep. 20.

Bost. Wood distillation. 15,432. Sep. 23.

Bourne. Producing working mixture for internal-combustion engines. 15,134. Sep. 17.

Brown, and Simon-Carves, Ltd. Gas washer. 15,208. Sep. 19.

Colman and Harman. Manufacture of coal gas. 15,448. Sep. 23.

Gray. Gas producers. 15,041. Sep. 16.

Jackson (Naaml. Vennootsch. Nederlandsche Lichte Olie Maatsch.). Converting higher molecular carbo-hydrates into lower ones. 15,057. Sep. 16.

Knowles. Composition for treating coal, etc., for manufacture of water-gas. 15,635. Sep. 26.

Leamon. Treatment of gases containing hydrogen sulphide. 15,568. Sep. 25. (U.S., Nov. 8, 1917.)

Oswald. Retorts. 15,039. Sep. 16.

Porat. Production of paraffin, waxes, oils, or fats from peat, peat straw, mosses, lichens, algae, grass, straw, etc. 15,357. Sep. 21.

Westinghouse Lamp Co. Treatment of filaments for incandescence lamps. 15,523. Sep. 24. (U.S., Oct. 15, 1917.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

8461 (1917). Lyman and Rambush. Ammonia-recovery producer-gas process and apparatus. (119,049.) Oct. 2.

12,802 (1917). Payne. See III.

12,809 (1917). Axtell. Liquid fuels. (109,806.) Oct. 2.

13,714 (1917). Edgeworth. Treatment of peat for fuel. (118,903.) Sep. 25.

18,478 (1917). Blair, Campbell, and McLean, Ross, and Corner. Mechanical retorts for destructive distillation of sawdust, shavings, small wood, peat, beans, nuts, etc. (118,960.) Sep. 25.

18,689 (1917). Sieurin. Transforming anthracite, coke, etc., into a product suitable for manufacture of carbon electrodes. (119,164.) Oct. 2.

4622 (1918). Bouillon. Treatment of peat. (118,993.) Sep. 25.

8258 (1918). Lyman and Rambush. Gas producers. (119,207.) Oct. 2.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Benton. 15,275. See XII. 15,276. See II.

Fryer, and Rowntree and Co. Combination of pitch, etc. 15,809. Sep. 28.

Jackson. 15,057. See II.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,802 (1917). Payne. Treatment of hydrocarbon oils. (119,066.) Oct. 2.

14,166 (1917). Koppers Co. See XIII.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Broadhurst, Lamble, Peachey, and United Alkali Co. Manufacture of substitute for celluloid or leather, etc. 15,244. Sep. 19.

Farquharson. Apparatus for coating papers, fabrics, etc., with waterproofing or other materials. 15,169. Sep. 18.

Grant. Waterproofing vegetable fibres, etc. 15,296. Sep. 20.

Gray and White. Installation for bleaching paper pulp, etc. 15,221. Sep. 19.

Schauffelberger. Manufacture of cellulose, etc. 15,772. Sep. 27.

Short, Short, and Tully. Compositions for treating textile fabric to render it waterproof and gas-tight. 15,168. Sep. 18.

Zdanowich. Manufacture of cellulose acetates. 15,163. Sep. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,208 (1917). Dreyfus. Manufacture of non-inflammable celluloid. (118,891.) Sep. 25.

8750 (1918). Aktiebolaget Cellulosa. Production of soda cellulose. (116,288.) Sep. 25.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Fish, and Jackson and Bro. Open-bleach kiers, etc. 15,146. Sep. 18.

Royle Dyeing Co. Dyeing machine for tubular knitted fabrics, etc. 15,420. Sep. 23.

#### COMPLETE SPECIFICATION ACCEPTED.

1225 (1918). Bright and Bros., and Evans. Machines for boiling, bleaching, dyeing, sizing, or similarly treating yarns or fabrics. (119,173.) Oct. 2.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Auden. Extraction of potash from alkaline, etc., minerals and manufacture of residual products into cement, china, etc. 15,431. Sep. 27.

Blockey, and Walker and Sons. Manufacture of chlorine liquors and salts. 15,786. Sep. 28.

Calvert. Production of cyanide and cyanogen compounds. 15,757. Sep. 27.

Gregory. Recovery of tin compounds. 15,389. Sep. 21.

Lamb. 15,496. *See* XV.

Leamon. 15,568. *See* II.

Lindblad. Production of cyanides and nitrides. 15,113. Sep. 17. (Sweden, June 8, 1917.)

Lunden and Thorssell. Production of nitrates by bacteria. 15,667 and 15,669. Sep. 26.

Lunden and Thorssell. Production of nitrates from ammonia, ammonium compounds, and organic nitrogen compounds. 15,668. Sep. 26.

Lunden and Thorssell. Production of calcium nitrate. 15,670. Sep. 26.

Lunden and Thorssell. Oxidation of ammonia, ammonium compounds, or organic nitrogen compounds. 15,671. Sep. 26.

Norsk Hydro-Elektrisk Kvaestofaktieselskab. Manufacture of concentrated nitric acid. 15,241. Sep. 19. (Norway, Oct. 30, 1917.)

Pierce. Manufacture of strontium peroxide. 15,691. Sep. 26.

Ross. 15,367. *See* XX.

Soc. Gros et Bouchardy. 15,540. *See* XVI.

Soc. Ind. de Produits Chimiques. Transforming free or combined ammonia mixed with inert gases into ammonium sulphate or concentrated or pure ammonia gas. 15,756 and 15,764. Sep. 27. (Fr., May 27 and June 22.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

8461 (1917). Lynn and Rambush. *See* II.

13,234 (1917). Mower and Williams. *See* XVI.

13,519 (1917). Kenyon and Williams. Stepped brick for the walls of acid plants, etc. (119,087.) Oct. 2.

16,636 (1917). Blingham. *See* X.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Auden. 15,431. *See* VII.

Bigot. 15,255. *See* I.

Hughes. Apparatus for annealing glass. 15,364. Sep. 21.

McLean. Bricks for furnaces, etc. 15,018. Sep. 16.

Webster. Kilns for burning clay products, glazed ware, and pottery. 15,590. Sep. 25.

#### COMPLETE SPECIFICATION ACCEPTED.

13,906 (1917). Mason and Couper. Manufacture of refractory bricks, blocks, tuyeres, etc., used in connection with furnaces for smelting metals, etc. (119,101.) Oct. 2.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Auden. 15,431. *See* VII.

Grant. Production of roofing materials. 15,297 and 15,464. Sep. 20 and 23.

Olson. Process of making waterproof cement. 15,712. Sep. 26.

O'Reilly. Composition for insulating blocks, roofing tiles, etc. 15,755. Sep. 27.

#### COMPLETE SPECIFICATION ACCEPTED.

17,783 (1917). Quick. Composition and manufacture of bricks, slabs, etc. (119,152.) Oct. 2.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Aarts and Aarts. Process for sulphatising ores, etc. 15,830. Sep. 28. (Holland, Oct. 10, 1917.)

Adam and Stevenson. Detinning of tinned iron. 15,323. Sep. 20.

Alpha Products Co. Making castings of rare earth metals. 15,122. Sep. 17. (U.S., Sep. 17, 1917.)

Angel. Treatment and reduction of refractory sulphide, etc., ores. 15,035. Sep. 16.

Armstrong. Metallurgy of iron and steel. 15,744. Sep. 27.

Arnold. High speed steel. 15,554. Sep. 25.

Barelay and MacKenzie. Treatment of metals. 15,285. Sep. 20.

Bibby and Boving. Electric blast furnaces. 15,345. Sep. 20.

Cammell, Laird, and Co., Carter, and Williams. Furnaces for melting metals, etc. 15,551. Sep. 25.

Ferguson. Apparatus for concentrating ores, etc. 15,598. Sep. 25.

Mate and Poulson. Production of malleable iron. 15,806. Sep. 28.

Ross. 15,367. *See* XX.

Shotton. Manufacture of iron for malleable iron castings. 15,720. Sep. 27.

Vautin. Recovery of metals from slags. 15,027. Sep. 16.

Williams. Alloy. 15,555. Sep. 25.

#### COMPLETE SPECIFICATIONS ACCEPTED.

8563 (1917). Webster, Edser, and Wood. Concentration of tin and wolfram ores. (119,050.) Oct. 2.

13,547 (1917). Jackson (Potthoff). Electro-plating apparatus. (119,091.) Oct. 2.

13,906 (1917). Mason and Couper. *See* VIII.

14,132 (1917). Eustis. Treatment of iron ore. (112,615.) Sep. 25.

14,915 (1917). Bertoya. Manufacture of tungsten metal powder. (119,117.) Oct. 2.



16,636 (1917). Bingham. Mechanical furnaces for roasting blende, pyrites, etc. (118,940.) Sep. 25.  
 17,095 (1917). British Thomson-Houston Co. (General Electric Co.). Alloys. (118,947.) Sep. 25.  
 2823 (1918). Bamfield. Case-hardening. (118,983.) Sep. 25.  
 7074 (1918). Shepherd, and Brazil, Straker, and Co. Electro-deposition of iron. (119,200.) Oct. 2.

# XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

Bibby and Boving. 15,345. *See* X.  
 British Thomson-Houston Co. (General Electric Co.). Electric furnaces. 15,320. Sep. 20.  
 Clark, and Hart Accumulator Co. Electric accumulators or secondary batteries. 15,277. Sep. 20.  
 Crosby. Electric furnaces. 15,600. Sep. 25.  
 Etchells, Greaves, and Watson and Co. Electric furnaces. 15,548. Sep. 25.  
 Fawcett and Hoyle. Electric furnaces. 15,302. Sep. 20.  
 Lawton. Electric furnaces. 15,812. Sep. 28.  
 Snyder. Electric furnace and method of operating same. 15,586. Sep. 25.  
 Stewart. Electrodes or plates for accumulators of the alkaline type. 15,587. Sep. 25.

## COMPLETE SPECIFICATIONS ACCEPTED.

13,547 (1917). Jackson (Potthoff). *See* X.  
 13,853 (1917). La Roche. Electric accumulators. (119,100.) Oct. 2.  
 17,377 (1917). Smith. Electric storage batteries. (116,256.) Sep. 25.  
 18,689 (1917). Sieurin. *See* II.  
 7074 (1918). Shepherd and others. *See* X.  
 9285 (1918). Kilburn (Norske Aktieselskab for Elektrokemisk Industri). Manufacture of carbon electrodes. (119,018.) Sep. 25.

# XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Benton. Distillation of fatty acids, recovered grease, coal tar, etc. 15,275. Sep. 20.  
 Benton. 15,276. *See* II.  
 Hannin. Creosote soap. 15,091. Sep. 17.  
 Porat. 15,357. *See* II.

## COMPLETE SPECIFICATION ACCEPTED.

8929 (1917). De Bruyn. *See* XIX.

# XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATIONS.

Duke. Pigments, and production of same. 15,533. Sep. 24.  
 Hutchins. Manufacture of lampblack. 15,444. Sep. 23.  
 Hutchison. Non-inflammable paint. 15,750. Sep. 27.

## COMPLETE SPECIFICATION ACCEPTED.

14,166 (1917). Koppers Co. Manufacture of resins from benzol plant residues. (112,419.) Oct. 2.

# XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

Banbury. Machines for treating rubber. 15,084—15,087. (U.S., Nov. 18, 1916, and Mar. 24, 1917.)  
 Peachey. Rubber composition. 15,362. Sep. 21.

# XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

Blockey, and Walker and Sons. 15,786. *See* VII.  
 Broadhurst and others. 15,244. *See* V.  
 Lamb. Detanning chrome leather for manufacture of glue, etc., and recovery of chrome compounds. 15,496. Sep. 24.  
 Potter. Leather substitute for soles of boots, etc. 15,083. Sep. 17.  
 Schlakmann. Dressing, fleshing, and unhairing skins. 15,702. Sep. 26.

## COMPLETE SPECIFICATION ACCEPTED.

9144 (1918). Tada. Preparation of gelatin. (119,210.) Oct. 2.

# XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

Fitzgerald. Manufacture of nitrogenous manures, etc. 15,312. Sep. 20.  
 Soc. Gros et Bouchardy. Production of cyanamide. 15,540. Sep. 24. (Fr., Nov. 3, 1917.)

## COMPLETE SPECIFICATION ACCEPTED.

13,334 (1917). Mower and Williams. Apparatus for use in the manufacture of superphosphate. (119,074.) Oct. 2.

# XVII.—SUGARS; STARCHES; GUMS.

## APPLICATIONS.

Goodwin. 15,507. *See* XIX.  
 Greville. 15,423. *See* XVIII.

# XVIII.—FERMENTATION INDUSTRIES.

## APPLICATION.

Greville. Brewing, and manufacture of brewing sugar, etc. 15,423. Sep. 23.

# XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Goodwin. Treatment of semolina. 15,507. Sep. 24.  
 Linden. Treating sludge. 15,328. Sep. 20.  
 Nielsen. Apparatus for concentrating milk, etc. 15,561. Sep. 25.  
 Wollaston. Softening water. 15,210. Sep. 19.

## COMPLETE SPECIFICATIONS ACCEPTED.

8929 (1917). De Bruyn, Ltd., and De Bruyn. Means for imparting a definite flavour to edible oils and fats. (119,052.) Oct. 2.  
 12,354 (1917). Senior. Whey extract for use as food. (118,874.) Sep. 25.  
 14,251 (1917). Bowler, Paterson, and Levy. Drying food and other products. (118,911.) Sep. 25.  
 6502 (1918). Spencer and Kemp. Dried eggs in powder form. (119,007.) Sep. 25.

# XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATION.

Ross. Manufacture and use of tungsten and its compounds for detection, prevention, and treatment of disease in animals. 15,367. Sep. 26.

# XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## APPLICATION.

Christensen. Manufacture of photographic plates and pictures. 15,194. Sep. 18.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Solvents; Recovery of — from air-vapour mixtures.* B. L. Knoedler and C. A. Dodge. J. Ind. Eng. Chem., 1918, 10, 593—595.

A PLANT is described for the recovery of mixed vapours of methyl alcohol, ethyl alcohol, acetone, and camphor which are evaporated in drying the collodion coating of gas mantles. The mantles are passed continuously through a heated drying chamber supplied with a current of air under slight pressure. The air-vapour mixture from the drying chamber is conducted to the recovery plant, which consists of a large surface condenser acting as a pre-cooler, a group of scrubbing towers, receivers cooled by brine coils, and a column still. The vapours, after passing through the pre-cooler, enter the "bell-and-seal" scrubbing towers where they bubble through water at 5° C.; the solvent vapours are thus absorbed and the cooled air is returned to the pre-cooler where it serves to cool vapours just entering the plant. The air may then be discharged into the atmosphere or used over again in the drying chamber. The water in the scrubbing towers is used repeatedly until it has absorbed about 12% of solvent vapours, and is then distilled. The camphor remains in the sections of the still where the temperature ranges between 95° and 75° C., and is removed periodically. About 80% of the vapours passing through the plant are recovered, and the estimated cost is 0.26 dollar per gall. of recovered solvent without making any allowance for the camphor.—W. P. S.

**PATENTS.**

*Continuous-rectification apparatus; Cooling plates for —.* E. Barbet et Fils et Cie., Paris. Eng. Pat. 110,910, Oct. 25, 1917. (Appl. No. 15,525 of 1917.) Under Int. Conv., Oct. 31, 1916. Addition to Eng. Pat. 105,549.

IN the cooling plates described in Eng. Pat. 105,549 (this J., 1918, 197A), the perforated plates are made in the form of a ring provided on the periphery with some larger perforations which serve to facilitate the starting of the apparatus. —W. H. C.

*Condenser; Annular —.* S. Hayek, Charlottenburg. Ger. Pat. 304,602, Apr. 16, 1916.

THE condenser comprises two concentric cylinders mounted within a casing. The inner cylinder has horizontal corrugations, the ridges of which extend nearly to the enclosing cylinder. There are gaps in the ridges at diametrically opposite places, the gaps in one ridge being displaced 90° from those in adjacent ridges. Cooling water is supplied to the outer casing, and flows thence to the central space within the inner cylinder.

*[Grinding] mills; Edge runners, pan and like —.* J. E. Savage, and Brinjes and Goodwin, Ltd., London. Eng. Pat. 118,465, Sep. 3, 1917. (Appl. No. 12,620 of 1917.)

THE grinding pan is supported on and driven by a vertical shaft, and its periphery is turned outwards and downwards so as to dip into an annular water seal on the enclosing casing. Dust from the pan is thereby prevented from reaching the gearing in the bottom of the casing. The two rollers in the pan are overhung from pivots supported entirely outside the pan, and each pivot is connected by bracing to a fixed parallel pivot so that the rollers rest freely on the bottom of the pan and may oscillate about the fixed pivot. A sleeve is interposed between each roller and its

spindle projecting outwards from the pan, and a second sleeve projects oppositely into the annular space between the first sleeve and the spindle. The second sleeve is braced to the parallel fixed pivot. Washers are provided between the sleeves at both ends to exclude dust and take up wear. The inner end of the sleeve and spindle projecting through the roller are covered by a detachable cap. The apparatus is intended more especially for use in the manufacture of explosives.—W. F. F.

*Grinders; Breaker-plate for —.* M. F. Williams, Assignor to Williams Patent Crusher and Pulverizer Co., St. Louis, Mo. U.S. Pat. 1,274,126, July 30, 1918. Date of appl., Feb. 12, 1918.

IN a grinding machine having rotary hammers mounted above an arcuate cage or grating, a breaker-plate is placed between the end of the sloping bottom of the feed hopper and the adjacent end of the cage. The front of the plate has a smooth upper face forming a continuation of the bottom of the feed hopper, and flush therewith, and a lower corrugated face to grind the material in conjunction with the rotary hammers. An intermediate step between the smooth and corrugated faces serves as a fulcrum for preliminary breaking of the feed by the hammers, prior to secondary reduction on the lower face and final reduction in the cage.—J. H. P.

*Ball mill.* D. H. Fairchild, Denver, Colo. U.S. Pat. 1,275,184, Aug. 6, 1918. Date of appl., May 31, 1917.

A BALL or pebble mill is provided with gratings spaced inwardly from the outer heads of the mill and with a central feed opening at one end and a central discharge opening at each end. Means are provided between each grating and the corresponding head for elevating the pulverised material to the adjacent discharge opening.—A. B. S.

*Agitating liquids, powders, granular substances, and the like; Device or apparatus for —.* F. Moore, Birmingham. Eng. Pat. 118,567, Mar. 19, 1918. (Appl. No. 4800 of 1918.)

A SQUARE wooden framework is mounted transversely on a horizontal shaft and has its sides projecting outwards at each corner of the square to form four shelves. Receptacles containing the substance to be agitated are strapped on the shelves, and the shaft and framework are rotated by a hand-wheel.—W. F. F.

*Filter.* A. J. Moisant, Assignor to General Research Laboratories. U.S. Pat. 1,270,042, June 18, 1918. Date of appl., Oct. 27, 1916.

TWO long vertical filter bags, one inside the other, and each supported by a wire frame, are mounted within a casing, the lower ends of the bags being fastened to the upper end of an outlet pipe which extends downwards to and through the bottom of the casing. Enclosing the filter bags is a non-perforated cylinder, closed at the top and open at the bottom; the lower end of the cylinder is some distance above the bottom of the casing but below the top of the outlet pipe. Around this cylinder is a foraminous cylinder. The space between the filter bags may be filled with a suitable filtering medium. The liquid to be filtered is fed into the casing at the top, flows through the foraminous cylinder, which retains coarse particles, rises in the space between the outer filter bag and the enclosing cylinder, and flows through the filter bags to the outlet pipe.



*Oxidising apparatus.* M. J. Chaplin, Seattle, Wash., Assignor to R. A. Brown, New York. U.S. Pat. 1,272,009, July 9, 1918. Date of appl., Mar. 15, 1917.

An annular retort is formed by an outer cylindrical wall and an inner conical wall; the latter is connected at the top with a chimney and the space enclosed by it forms the furnace. Surrounding the lower part of the chimney is a rotary annular hopper provided with a horizontal foraminous partition. Adjustable blades extend into the hopper and feed the material through the partition to delivery spouts extending into the retort. Means are provided for removing slag from the conical wall and the floor of the retort and directing it towards a discharge opening.

*Kiln.* M. M. Minter, Assignor to P. J. Brown, Albany, Ga. U.S. Pat. 1,272,495, July 16, 1918. Date of appl., Apr. 10, 1918.

In a series of progressively operated kilns, each kiln is connected with a flue through which gaseous fuel is supplied and also with a common exit flue. The kilns are connected with each other by flues which are also connected between each pair of kilns with the gas supply flue.—W. H. C.

*Fire-resistant composition and process of making same.* J. A. Zibell, Tappan, N.Y. U.S. Pat. 1,273,307, July 23, 1918. Date of appl., Mar. 9, 1918.

"RIPENED" milk is made into an emulsion and mixed with hydrated lime, sodium chloride, and a suitable pigment.—W. H. C.

*Heating and drying apparatus.* J. B. Mnse, Cameron, N.C. U.S. Pat. 1,273,396, July 23, 1918. Date of appl., May 18, 1917.

A FURNACE provided with a casing having double walls spaced apart is arranged at one end of a drying chamber. Heat-distributing tubes, which serve also as a brace for the top of the inner casing, extend longitudinally from the space between the walls of the casing into the drying chamber.—W. H. C.

*Dry-kiln.* C. A. Lohman, Seattle, Wash. U.S. Pat. 1,274,911, Aug. 6, 1918. Date of appl., July 18, 1917.

A DRYING kiln is arranged immediately over a heating chamber, with a communicating opening at one side of the dividing floor. A vertical flue at the other side communicates with the drying chamber and with the lower part of the heating chamber. The openings on both sides are provided with dampers, and a circulation is maintained through both chambers. Steam heating pipes are arranged horizontally in the heating chamber just above the flue opening. A perforated horizontal air-supply flue extends through the heating chamber, and a discharge flue is provided for the drying chamber.—W. F. F.

*Dehydrator; Continuous suction — with a fixed or horizontally moving filter base.* A. Gerlach, Nordhausen. Ger. Pat. 306,259, Nov. 18, 1916.

The filter base proper is protected by a superposed framework which holds and supports a strong subsidiary layer of filtering material; the filter base is thereby relieved of the pressure and movement of the wet material as this is moved along by an archimedean screw, and the ease with which the subsidiary filter-frame can be removed facilitates repairs or cleaning.—D. F. T.

*Steam plate dryers.* O. Kraushaar, Meuselwitz. Ger. Pat. 306,505, May 23, 1917.

By leading away the condensed water from one or more plates through a water trap and connecting the condensed water pipe with an air separator so that the air may be released intermittently or continuously, together with any occasional excess of steam, it is possible to obtain a considerable increase in the velocity of the flow of the steam and therefore also of the heat transference.

—D. F. T.

*Drying apparatus.* Kraftwerk Stettin Ges.m.b.H., Stettin. Ger. Pat. 306,515, Aug. 30, 1916.

Drying is effected by means of a closed steam coil or the like into which the vapours evolved from the material being dried are introduced by means of a steam jet pump. The drying chamber is closed, so that admixture of air with the vapours withdrawn therefrom is avoided.

*Heating device for rotary drying cylinders, heated internally and externally.* C. Runge, Willershausen. Ger. Pat. 306,748, Jan. 26, 1916.

THE drying cylinder is provided with wooden diaphragms at intervals along its length, and the heating chamber around it is subdivided by transverse walls, each section being connected by an adjustable inlet with the duct which supplies the drying medium throughout the length of the cylinder. By adjusting the heating valve of each section, the apparatus may be used for drying a variety of fruits.—D. F. T.

*Centrifugal drying machine.* T. A. Bryson, Troy, N.Y., Assignor to Tolhurst Machine Works. U.S. Pat. 1,273,817, July 30, 1918. Date of appl., Nov. 28, 1914.

THE machine consists of a rotating bowl with an outer cylindrical wall for holding a relatively dense and impervious layer separated by the centrifugal action. At intervals around the wall enclosed recesses are formed by perforated walls projecting from the cylindrical wall through the impervious layer. Liquid from within this layer passes through the perforations into the recesses.

—J. H. P.

*Centrifugal machine.* W. H. Uhland Ges.m.b.H., Leipzig-Gohlis. Ger. Pat. 306,670, Jan. 30, 1916.

THE machine comprises two concentric cones, of different taper, mounted on a common horizontal axis, the inner one rotating at a greater velocity than the outer. The separated solid is driven by centrifugal force into an annular chamber at the larger end of the cones. In this chamber is a screw which forces the separated material inwards to discharge openings in the end wall.

*Separating entrained liquids from gases; Apparatus for —.* J. P. Fisher, Bartlesville, Okla. U.S. Pat. 1,273,595, July 23, 1918. Date of appl., June 8, 1916.

THE gas is diverted through a by-pass main in which is an open tray having the shape of the pipe and provided with vanes which cause the gas to take a spiral path. An opening is left below the vanes for the passage of any liquid which separates.—W. H. C.

*Refrigerating machine; Rotary —.* C. Orr, Fort Wayne, Ind., Assignor to General Electric Co. U.S. Pat. 1,273,653, July 23, 1918. Date of appl., Sep. 15, 1916.

A CLOSED condenser within which is a reservoir for the refrigerating liquid is connected with a closed evaporator by a hollow shaft. The refrigerating

liquid flows from the reservoir to the evaporator through a pipe passing through the hollow shaft, and the flow is automatically stopped when the pressure in the condenser reaches a certain limit. The vapour from the evaporator passes through an oil separator into the hollow shaft and thence to the condenser. The separator is supported on the outlet pipe within the evaporator and is provided with a spout from which the separated oil can flow out only when the separator is tilted from its normal position.—W. H. C.

[Ammonia] condenser [for refrigerating apparatus]. L. Block, Mamaronock, N.Y. U.S. Pat. 1,271,268, July 2, 1918. Date of appl., May 20, 1915.

COMPRESSED ammonia gas passes through a pipe, provided with an injector nozzle, to the lowest turn of a coil over which water flows downwards. The gas passes upwards through the coil and is liquefied, the liquid ammonia leaving through a pipe at the top. A branch pipe leads from the outlet pipe to the injector nozzle on the gas inlet pipe, so that part of the liquefied ammonia is drawn into the coil with the entering gas, whereby the efficiency of the condenser is increased.

Solid constituents from an emulsion; Electro-osmotic process of separating —. A. Gerlach, Nordhausen. Ger. Pat. 305,217, Oct. 1, 1916.

SOLID constituents are separated from an emulsion, and subsequently dried by a process of electro-osmosis, effected by means of a roller-shaped anode rotating between surrounding cathodes, which are constructed in sections to which the current is separately conducted. One of these sections, connected with a feed hopper, approaches nearer to the anode in the direction of rotation of the latter, and those sections which effect the subsequent dehydration are pressed by means of springs against the layer of partly dehydrated material on the anode.—C. A. M.

Feed pipe for hot gases for rotary drums and vessels. R. Bergmans, Berlin-Wilmersdorf. Ger. Pat. 306,506, July 31, 1917.

THE conical nozzle of the pipe supplying the heated gas fits into an opening in the end of the rotating drum or vessel and contains a concentric cone, by the adjustment of which the flow of gas can be regulated. The cone is hollow and is perforated near its apex; its interior is connected through the hollow supporting rod and a piece of flexible tubing with one arm of a glass U-tube, the other arm of which is connected with the space outside the nozzle; the position of the cone is then adjusted so that a liquid in the U-tube stands at approximately the same level in each limb.—D. F. T.

Fires; [Apparatus for] extinguishing of —. C. H. Meigs and F. A. Epps, New York. Eng. Pat. 118,534, Dec. 7, 1917. (Appl. No. 18,148 of 1917.)

Expansive force; Utilising an — [for pumping liquids]. H. A. Humphrey, London, and W. J. Rusdell, Dudley, Assignors to Humphrey Gas Pump Co. U.S. Pat. 1,271,712, July 9, 1918. Date of appl., May 16, 1912.

SEE Eng. Pat. 28,187 of 1911; this J., 1912, 860.

Drying china or other clay, fullers' earth, whitening, and the like; Method of and apparatus for —. W. J. Gee, London. U.S. Pat. 1,274,158, July 30, 1918. Date of appl., Apr. 6, 1915.

SEE Eng. Pat. 8892 of 1914; this J., 1915, 702.

Centrifugal separators. Eng. Pat. 118,262. See XXII.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

Coal; Deterioration in the heating value of — during storage. H. C. Porter and F. K. Ovlitz. U.S. Bureau of Mines, Bull. 136, 1917. 34 pages.

TESTS were made on New River and Pocahontas coals (both semi-bituminous), on Pittsburgh coal (bituminous—used for carbonisation), and on Wyoming coal (sub-bituminous). The coal was exposed to atmospheric influence by storage in piles and in bins. The quantities varied from about 300 lb. to 120 tons. A wide range of climatic conditions was secured by carrying out the experiments at several stations, including the Isthmus of Panama. Except in the case of the sub-bituminous coal the maximum loss of calorific value during exposed storage was 1.2% in the first year, and 2.6% after two years. The sub-bituminous variety deteriorated 2 to 3% in the first year and 5.5% after three years. This coal was stored in bins. Where the bin was uncovered the lumps on the surface became pulverised to a depth of 8 to 12 inches. It would seem that this powdery layer acts as a protection to the bulk of the coal, which will not then be attacked unless there be atmospheric access through the walls or floor of the bin. Further experiments showed that submersion under water effectually prevents loss in calorific value during storage of coal. It is, however, concluded that submersion under water is an unjustifiable expense, and that the loss in calorific value due to storage in exposed conditions has been commonly over-estimated. In making the calculations a formula was employed whereby the calorific value of the actual coal substance was determined, allowance being made for the content of ash, sulphur, and moisture.

—H. J.

Gas-producer; Coke and coal as fuel for —. D. J. Stadler. Stahl u. Eisen, 1918, 38, 765—769.

MARKGRAF (Stahl u. Eisen, 1916, 36, 1245) found that gas from a coke producer was less effective than that from a coal producer in open-hearth steel furnaces. The author has collected data on the composition of gas from producers fired with coke and coal respectively, and shows by calculations that in general when working under similar conditions, gas from coal is more efficient in use than is that from coke, since less air is required for its combustion, and higher temperatures may be attained.—F. C. Th.

Bitumens; Device for determining the dropping point (melting point) of fats, waxes, paraffin wax, etc., and especially —. F. Dupré. Chem.-Zeit., 1918, 42, 398.

A METAL cup with a small central opening in the bottom is filled with the bitumen or the like, the latter being first melted if necessary. The cup fits into the lower end of a metal tube surrounding the lower end of a thermometer, the bulb of which is just above the top of the metal cup. The thermometer with attached metal tube and cup is mounted by means of a cork, inside a test-tube. This is placed in a beaker of water or sulphuric acid, which is heated at first strongly to within 10°–20° C. of the melting point and then so that the temperature rises about 1° per minute. The results obtained with tallow and paraffin wax agreed well with those obtained by Ubbelohde's apparatus (this J., 1905, 941), over which the new apparatus has several advantages, especially for bitumens, etc., of higher melting point.



*Formation of ammonia by heating coke with calcium hydride.* Glaser. See VII.

*Optical dispersion of oils from an analytical point of view.* Fryer and Weston. See XII.

#### PATENTS.

*Fuel product and process of making same.* O. F. Werner, San Jose, Cal. U.S. Pat. 1,274,480, Aug. 6, 1918. Date of appl., Aug. 30, 1917.

LAMPLACK 70 parts, a comminuted combustible material 30 parts, and lime dust 2 parts, are heated, mixed with an asphalt binder 5 parts, compressed, and cooled.—W. F. F.

*Coal dust and wood charcoal powder; Treatment of — to prevent absorption of water and to hinder spontaneous combustion.* N. K. H. Ekelund, Jönköping, Sweden. Ger. Pat. 306,727, Feb. 18, 1917. Under Int. Conv., Apr. 14 and Aug. 5, 1916.

THE material is well dried and mixed with finely divided peat which has been dried at a temperature of at least 100° C.—L. A. C.

*Lignite, peat, etc.; Treatment of — to produce a non-hygroscopic fuel.* Bertzit Ges. m. b. H., Munich. Ger. Pat. 306,880, Dec. 6, 1913.

THE material is first dried at a moderate temperature and then heated with exclusion of air to between 180° and 250° C. until evolution of water, carbon dioxide, and nitrogenous gases is complete. Esters and imides contained in the material are thus decomposed and the bituminous portion of the fuel swells and fills up the pores, preventing the absorption of water.—L. A. C.

*Coke-ovens.* E. Oré, Caen, France. Eng. Pat. 118,470, Sep. 7, 1917. (Appl. No. 12,866 of 1917.)

A COKE-OVEN having a number of carbonising chambers adjoining one another and separated by partitions, is provided with a number of heating flues in the partitions and end walls, each in the form of an inverted U. Each limb of the flue is provided with a gas burner at the bottom, the burners on either side being used alternately. The gas reaches the burners through refractory conduits extending from a common supply conduit at the front of the oven and provided with separate regulating valves. The air supply for each burner passes from a regenerator below the oven through a similar refractory conduit provided with a regulating damper, to the refractory block forming the burner. The hot combustion gases pass up one side of the flue and down the other side to the corresponding burner, which has its gas-supply passage closed but its air-supply passage open, so that the hot gases may pass through this passage to the complementary regenerator below the oven to heat it. When the alternate burners are brought into action the hot gases pass through the flues in the opposite direction without modifying the heating. The proportions of gas and air supplied to each burner may be adjusted independently by observing, through openings in the front of the oven, the nature of the combustion products discharged through each inoperative burner. The same type of flue may be used in an oven having no regenerator, the air being heated by contact with the hot walls, or in an oven in which the air is not preheated. In these cases only one limb of the inverted U is provided with a burner.—W. F. F.

*Coke-oven; Regenerative —.* El. Coppée, Brussels. U.S. Pat. 1,267,380, May 28, 1918. Date of appl., July 28, 1914.

A NUMBER of regenerators for a battery of coke-ovens are connected in parallel to a common supply flue and a common collecting flue. A pair of flues are provided between the top of each regenerator and the bottom of the corresponding oven, through which the hot gases are drawn in series from the regenerator to the uptake, and means are provided to reverse the action of the regenerators as required.—W. F. F.

*Coke-oven; Reversing regenerative —.* C. Still, Recklinghausen. Ger. Pat. 306,214, May 10, 1916.

THE oven has vertical heating flues to which air is supplied at the base and combustible gas at the base and through lateral openings at different heights. Any suitable gas may be used for the burners at the base of the flues and is supplied to one half of the flues during one period and to the other half of the flues when the draught is reversed. The gas supplied to the lateral openings in the flues should have a composition such that it has little tendency to pyrogenic decomposition, or it should be admixed with gases which will hinder or prevent such decomposition. It is supplied continuously, irrespective of the reversal of draught.

*Coal and other materials; Distillation of — for the manufacture of motor gas and utilisation of residual products.* H. K. Hiller, London. Eng. Pat. 118,522, Nov. 20, 1917. (Appl. No. 17,054 of 1917.)

COAL, lignite, wood, peat, shale, or other similar material, is distilled in a horizontal or vertical retort at about 500° C., the vapour condensed, and the residual gas stripped by oil washing and then compressed in a water-cooled compressor to a pressure of about 15 atmospheres. The whole or part of this gas is returned to the retort under pressure, and liberated just above the material in a horizontal retort, or at the temperature zone of 500° C. in a vertical retort. The gas is cooled by expansion and cools and mixes with the vapour liberated in the retort so that cracking of the vapour is prevented. It is claimed that a gas consisting mainly of methane, suitable for use in internal combustion engines, and free from vapours deleterious to balloon fabric is thus obtained.—W. F. F.

*Oils from coal-gas; Extraction and fractional distillation of —.* D. Marbais, Charleroi, and C. Deguide, Ixelles, Belgium. Eng. Pat. 118,730, Nov. 1, 1917. (Appl. No. 15,923 of 1917.)

COAL-GAS is passed up a tower or series of towers filled with suitable material, e.g., wood or iron shavings, etc., down which coal tar oil or anthracene oil, distilling between 230° and 250° C., is passed to absorb the benzene, etc. On leaving the bottom of the tower, the oil passes into a receiver whence it is forced by means of a pump up through a sheaf of tubes contained in a casing and then to the top of a tower similar to the first. As it trickles down the tower, the oil is debenzolised by a current of steam which enters at the bottom and passes out at the top by a pipe which leads to the casing in which it preheats the ascending oil. The steam, charged with benzene and other vapours, on leaving the casing at the bottom at about 95° C., passes into a "draining device or collector," and thence up a condenser and tower, the temperature at the top of which is about 70° C. Condensed liquid, consisting of water and "heavy oil," i.e.,

solvent naphtha and naphthalene, flows downwards into the collecting device, whence it passes into a separator; uncondensed vapour passes through a similar tower the temperature at the top of which is about 70° C. In this tower the "toluened oil or spirit," i.e., a mixture of benzene, toluene, and xylenes, separates out together with water and is drawn off at the bottom. Remaining uncondensed vapour is passed through a descending coil refrigerator, the condensed liquid consisting of benzol and water.—L. A. C.

*Liquefied gas; Method of producing and separating* — L. Wolf, Chicago, Ill. U.S. Pat. 1,274,970, Aug. 6, 1918. Date of appl., May 31, 1916.

HYDROCARBON gas such as distillation gas or natural gas is passed through a heat-insulated chamber, and cooled by the introduction of gaseous constituents of the mixture at such a temperature that the easily liquefiable constituents are liquefied. The gas is agitated in passing through the chamber, the liquid is separated, and the residual gas then liquefied.—W. F. F.

*Motor fuel oil; Method and apparatus for producing — by distilling petroleum.* C. B. Forward, Urbana, Ohio, U.S.A. Eng. Pat. 117,372, Oct. 27, 1917. (Appl. No. 15,654 of 1917.)

The apparatus is a modification of that described in U.S. Pat. 1,189,083 (this J., 1916, 883). Petroleum oil is forced through two preheater coils contained in casings through which superheated steam is passed. The steam enters the second casing at about 650° to 800° F. (about 340° to 430° C.), leaving at about 325° to 650° F. (about 160° to 340° C.), at which temperature it enters the first casing. On leaving the second preheater coil, the oil is mixed with superheated steam and sprayed through an atomiser into a separating chamber in which the heavier portions fall to the bottom as a liquid, while the lighter portions, mixed with steam, pass off at the top into a clarifier, and then, through a reducing valve, into a condenser. The pressure within the separator is about 100 to 200 lb. per sq. in., and the temperature about 400° to 600° F. (about 205° to 315° C.).—L. A. C.

*Heavy hydrocarbons; Conversion of — into lighter hydrocarbons.* S. Maxim, Streatham, and A. W. Crosse, Exors. of H. S. Maxim, London. Eng. Pat. 118,122, Aug. 18, 1916. (Appl. No. 11,764 of 1916.)

PARAFFIN oil is converted into a light volatile spirit and a heavy oil suitable for lubricating purposes by agitating and heating the oil under pressure, and at the same time subjecting it to electrochemical action. The process is carried out in a long steel cylinder, mounted on hollow trunnion shafts midway of its length. The interior surfaces are all heavily coated with copper. The cylinder is heated by a gas burner situated beneath, and the gas supply to the burner is regulated by a device placed on one of the hollow trunnion shafts, and actuated by the changes of pressure within the cylinder. The other trunnion shaft carries a pressure gauge. The cylinder is first charged to about a quarter of its capacity with paraffin oil, to which is added a small quantity of an electrolyte (e.g., an aqueous solution of common salt or dilute sulphuric acid) together with small pieces of zinc, or V-shaped copper-zinc couples. The air inside the cylinder is pumped out, and, if desired, hydrogen gas, which may be under high pressure, is admitted. The cylinder is then rotated over the

heater until the internal pressure is raised to about 300 to 500 lb. per sq. in., which is kept constant for several hours.—T. St.

*Liquid hydrocarbons; Apparatus for obtaining* — E. H. Sheets, Muskogee, Okla. U.S. Pat. 1,273,191, July 23, 1918. Date of appl., June 27, 1916.

GASES and vapours are compressed, expanded, and commingled with an oil in a mixing tank under heat and pressure, to cause combination of the gases and vapours with the liquid oil. The gases and vapours are derived partly from the oil supply tank and partly from other sources. The mixing tank is fitted with several vertical tubes, each having a bend to form return tubes which are connected with a header, whereby the heavier substances are returned to the tank and the lighter substances carried away from the tank.—T. St.

*Crude-oil still.* T. E. Murray, New York. U.S. Pat. 1,273,523, July 23, 1918. Date of appl., Dec. 14, 1916.

A HORIZONTAL cylindrical still is provided with scraping blades carried by hubs pivoted at the ends of radial arms attached to a longitudinal rotary shaft mounted eccentrically within the still. The hubs also carry levers which are connected to the radial arms by helical springs. Solid material on the bottom of the still is moved by the scrapers towards a delivery opening in one of the still heads.—T. St.

*Crude petroleum; Apparatus for reducing* — C. B. Forward, Urbana, Ohio. U.S. Pat. 1,274,405, Aug. 6, 1918. Date of appl., Sep. 25, 1916.

OIL and superheated steam are forced together into the first of a series of separator chambers fitted with means to maintain a predetermined vapour pressure therein. From the top of each chamber is a passage for vapour and at the bottom is a passage for the residuum so arranged as to discharge into the next chamber. Superheated vapour is added to the residuum in the passage, and an atomiser is provided at the discharge end of the passage.—L. A. C.

*Petroleum oils; Process of treating [cracking] — Apparatus for treating petroleum oils.* W. F. M. McCarty, Baltimore, Md. U.S. Pats. (A) 1,274,912 and (B) 1,274,913, Aug. 6, 1918. Date of appl., Aug. 17, 1916.

(A) PETROLEUM oil having a boiling point higher than that of gasoline is subjected to the action of superheated steam in a closed container, at a pressure of 150 lb. per sq. in., in the presence of an aqueous solution of an aluminium compound. The mixture is then allowed to settle and the aqueous solution and suspended matter withdrawn. The oil is vaporised at 400° C. and then treated at 600° C. with hydrogen and a catalytic agent, and the resulting product condensed. (B) Oil and hydrogen are passed downwards through a vertical cracking tube, and the products pass into a collecting chamber connected to the lower end and provided with draw-off pipes at top and bottom. A shaft carrying a fan extends through the lower part of this chamber and the cracking tube, and operates an agitating device in the cracking tube. Means are provided to reciprocate and rotate the shaft. The cracking tube is enclosed in a shell containing a heating tube opening into the shell at one end and connected to a flue at the other end. The shell contains a heating device, and combustion products pass through the heating tube.—W. F. F.



[*Petroleum*] oil; *Process of distilling* —. J. E. Biggins, Port Arthur, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,274,976, Aug. 6, 1918. Date of appl., Oct. 23, 1917.

IN the cracking distillation of petroleum oils, the oil is supplied at one end of a cylindrical, inclined, unobstructed chamber in which a cracking temperature and a pressure greater than atmospheric are established; air is supplied at several points along the length of the chamber to maintain a limited amount of combustion therein and keep up the heat. Vapours and gases are removed at the further end, tar and carbon are settled out from the vapours and gases, the mixture is cooled to condense any high boiling oils therein, the condensed oil is returned to the feed end of the chamber, and the residual mixture of vapours and gases is cooled to recover the cracked products.

—J. F. B.

*Fuel for internal combustion engines. Explosions-Turbine-Studien-Ges.m.b.H.*, Berlin-Wilmersdorf. Ger. Pat. 3/6,283, Feb. 21, 1914. Addition to Ger. Pat. 298,309.

To the mixture described in the chief patent is added a substance capable of forming an emulsion with water and of higher sp. gr. than water. A fuel of approximately the same sp. gr. as water has the following composition: 500 parts of tar-oil, 500 of benzol, 200 of pentachloroethane, and 100 parts of soap solution.—L. A. C.

*Lubricating oil; [Hydrogenated naphthalene as] substitute for* —. Tetralin Ges.m.b.H., Berlin. Ger. Pat. 306,836, Mar. 7, 1916.

HYDROGENATED naphthalene, e.g., tetrahydronaphthalene, decahydronaphthalene, etc., may be used for lubrication, either alone or mixed with other lubricants.—L. A. C.

*Coke; Apparatus for discharging and quenching* —. S. N. Wellington, Assignor to Coalite, Ltd., London. U.S. Pat. 1,274,826, Aug. 6, 1918. Date of appl., Apr. 17, 1916.

SEE Eng. Pat. 16,108 of 1915; this J., 1916, 958.

*Burning pitch; Apparatus for* —. G. H. H. Bölling, Christiania, Norway. U.S. Pat. 1,272,198, July 9, 1918. Date of appl., Mar. 26, 1917.

SEE Eng. Pat. 101,444 of 1916; this J., 1916, 1100.

*Apparatus for redissolving separated constituents of coal tar oils or other liquids.* Ger. Pat. 306,219. See III.

*Nitrated product and process of making same.* U.S. Pat. 1,273,568. See XXII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Recovery of solvents from air-vapour mixtures.* Knoedler and Dodge. See I.

### PATENTS.

*Arc-light electrodes; Homogeneous — containing luminiferous substances.* Rütgerswerke A.-G., Berlin. Ger. Pat. 304,461, Jan. 1, 1915.

IN the operation of electric arc lamps with electrodes containing luminiferous substances, beads

of the fused material may form and solidify on the electrodes and hinder or prevent the production of an arc. According to the present patent the electrodes are provided with openings, channels, or the like, which serve to retain the fused material under the action of capillarity and prevent formation of beads of fused material at the crater and so ensure complete utilisation and consumption of the luminiferous substances.

*Arc-light electrodes containing luminiferous substances.* Rütgerswerke Akt.-Ges., Berlin. Ger. Pat. 306,658, Sep. 14, 1915. Addition to Ger. Pat. 304,461 (see preceding abstract).

THE upper electrode is provided with a channel in the form of a circle, the diameter of which is equal to or greater than the diameter of a second lower electrode, so that should fused material be dislodged from the upper electrode it will not fall into the crater of the lower electrode.—L. A. C.

*Incandescence gas mantles; Manufacture of* —. Deutsche Gasglühlicht Akt.-Ges. (Auer Ges.), Berlin. Ger. Pat. 306,621, Dec. 3, 1916.

FOR washing the material used in the preparation of incandescence mantles, water purified by means of an aluminate-silicate (permutite), i.e., separated from salts of all but the alkali metals, may be employed in place of distilled water.—L. A. C.

*Treatment of coal dust and wood charcoal powder to prevent absorption of water and hinder spontaneous combustion.* Ger. Pat. 306,727. See IIA.

## III.—TAR AND TAR PRODUCTS.

*Phthalic anhydride from crude phthalic acid; Determination of* —. C. R. Downs and C. G. Stupp. J. Ind. Eng. Chem., 1918, 10, 596–598.

A WEIGHED quantity of the sample is placed in a small glass capsule, and a glass tube, about 5.5 in. long, containing 1.5 grms. of cotton wool previously boiled with 10% sodium hydroxide solution, washed, and dried, is inserted in the top of the capsule to within 0.25 in. of the bottom of the latter. A current of air (3 bubbles per sec.) is drawn through the apparatus, the air entering at the space between the tube and the inner wall of the capsule, and the latter is then dipped in a bath of Rose metal at 220° C. The heating is continued for 45 mins., by which time the phthalic acid is decomposed and the sublimed anhydride collected in the cotton wool tower. The weight of the residue in the capsule is determined; the cotton wool is removed from the tube, boiled for 30 mins. with 45 c.c. of N/10 sodium hydroxide solution and 50 c.c. of water (the tube itself may be rinsed out with the alkali solution), an excess of N/10 acid then added, the boiling continued for 15 mins., and the mixture titrated with sodium hydroxide solution, using phenolphthalein as indicator. Each c.c. of N/10 sodium hydroxide solution is equivalent to 0.0074 gm. of phthalic anhydride.—W. P. S.

*Device for determining the dropping point (melting point) of fats, waxes, paraffin wax, etc., and especially bitumens.* Dupré. See IIA.

*Optical dispersion of oils from an analytical point of view.* Fryer and Weston. See XII.

## PATENTS.

*Coal-tar oils and other liquids; Apparatus for re-dissolving separated constituents of —.* Dampfessel- und Gasometerfabrik A.-G. vorm. A. Wilke und Co., Brunswick. Ger. Pat. 306,219, Apr. 1, 1917.

The receptacle containing the tar oil or the like is provided near the bottom with a heating coil below which is an arrangement for conveying material deposited from the oil to a trough at one side. The trough is fitted with means for heating or stirring and the material therein, after being re-liquefied or dissolved may be either returned to the container or led away to be used, for example, as fuel.—L. A. C.

*Phenol and allied products; Manufacture of —.* R. N. Wallach, Wappingers Falls, N.Y. U.S. Pat. 1,274,961, Aug. 6, 1918. Date of appl., Jan. 12, 1918.

A CONCENTRATED aqueous solution of benzenesulphonic acid at about 100° C. is added to fused caustic alkali at about 300° C.—L. A. C.

*Pyridine bases; Methods and apparatus for recovering —.* F. B. Dodge, New York, and F. H. Rhodes, Philadelphia, Pa., Assignors to The Barrett Co. U.S. Pats. (A) 1,274,998, (B) 1,274,999, and (C) 1,275,000, Aug. 6, 1918. Date of appl., Jan. 17, 1918.

The crude saturated acid solution of ammonium sulphate from the saturator in the direct or semi-direct ammonia recovery process is saturated with ammonia and the liberated pyridine bases recovered (A) by distilling the liquor so obtained, introducing ammonia into the distillate, and separating the pyridine bases by gravitation, or (B) by agitating the liquor with an oil to dissolve the pyridine bases. (C) A suitable apparatus for process (A) consists of a storage tank for the saturator liquor, portions of which are run as required into a still fitted with means for the introduction of ammonia. Connected with the still are a condenser and receiver for the condensed liquid into which ammonia may also be passed. Storage tanks connected with the receiver are provided for the condensed liquid and the separated pyridine bases. A storage tank with a filter interposed is also provided for the liquor from the still.—L. A. C.

*4-Sulpho-1-aminobenzene-2-carboxylic acid; Preparation of —.* Farbenfabr. vorm. F. Bayer und Co. Ger. Pat. 307,284, Oct. 26, 1915. Addition to Ger. Pat. 296,941.

ACCORDING to the chief patent, sulphuric chlorhydrin and anthranilic acid, preferably in molecular proportions, were allowed to interact in an inert organic solvent. As an improvement, the organic solvent is replaced by sulphuric acid monohydrate, which, under the prescribed conditions, does not sulphonate the anthranilic acid.—L. A. C.

*[Tar oils;] Process of manufacture of an agglutinant for coatings [from —].* J. Rosen, Paris. U.S. Pat. 1,273,927, July 30, 1918. Date of appl., July 30, 1914. Renewed May 29, 1918.

SEE Fr. Pat. 474,929 of 1914; this J., 1915, 1084.

*Sulphonation of aromatic hydrocarbons; Process for the —.* H. Bull, Philadelphia, U.S.A. Eng. Pat. 118,727, Oct. 23, 1917. (Appl. No. 15,421 of 1917).

SEE U.S. Pat. 1,247,499 of 1917; this J., 1918, 52 A.

*p-Amino-N-methylformanilide; Process for the manufacture of —.* G. T. Morgan, London. U.S. Pat. 1,273,901, July 30, 1918. Date of appl., Jan. 31, 1917.

SEE Eng. Pat. 111,321 of 1916; this J., 1918, 5 A.

*Extraction and fractional distillation of oils from coal gas.* Eng. Pat. 118,730. See 11A.

*[Hydrogenated naphthalene as] substitute for lubricating oil.* Ger. Pat. 306,836. See 11A.

*Solid preparations containing cresols and mixtures of cresols and manufacture thereof by means of salts of fatty acids.* Eng. Pat. 118,667. See XIXb.

*Nitrated product and process of making same.* U.S. Pat. 1,273,568. See XXII.

## IV.—COLOURING MATTERS AND DYES.

*Colours; Method of standardising the fastness of — to light.* A. Robson. J. Soc. Dyers and Col., 1918, 34, 185—186.

THE method is based on the measurement of the quantity of actinic light required to produce a visible change in the tone or depth of the test-sample by noting the number of consecutively exposed standard strips of a quickly fading colour which are completely bleached under the same conditions. A dyestuff fulfilling the desired conditions for a standard is Eosine GGF (Cassella), which quickly fades to a clear white. Strips of coarse white filter paper are steeped in a 0.1% solution of Eosine, dried without rinsing, and cut into pieces 3" × ½". The samples to be tested are dyed in known proportions on cotton yarn which is wound on a cardboard strip adapted to hold 12 samples lengthwise. A stout cardboard envelope is arranged to cover one half of the row of patterns and a strip of standard Eosine paper, numbered 1, is clipped on the card. The whole is then exposed under a sheet of glass. The set of patterns is examined daily, and when the Eosine strip No. 1 has faded to a clear white, it is replaced by another, No. 2, and so on until the visible change in the test patterns is recorded. In this way a definite measure of the quantity of actinic light is obtained independent of the variations in its quality from day to day and, if necessary, the whole set may be laid aside at any time and the exposure resumed at the point where it was interrupted. It is possible also to subdivide the results into fractions of the standard to an approximate degree. The principal difficulty lies in determining the exact end-point at which the Eosine paper is completely bleached.—J. F. B.

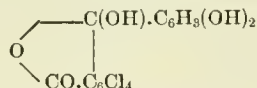
*Scarlet S3R (B) and Ponceau 3R (By); Structure of —.* H. W. Stiegler. J. Ind. Eng. Chem., 1918, 10, 600.

By reduction with stannous chloride it was found that the components of the monoazo dyestuff, Scarlet S3R (Badische) are amino-R-salt (1,2-aminonaphthol-3,6-disodium sulphonate) and pseudocumidine (1,2,4-trimethyl-5-aminobenzene). The components of Ponceau 3R (Bayer) are amino-Bayer acid (1,2-aminonaphthol-8-sulphonic acid) and gamma acid (2,8-aminonaphthol-6-sulphonic acid). Scarlet S3R is thus identical with the Ponceau 3R classified as No. S3 in Schultz's tables and the constitution given by Schultz for Bayer's Ponceau 3R is incorrect.—W. P. S.



*o*-2'.4'-Dihydroxybenzoyltetrachlorobenzoic acid and 2,3,4-trichloro-6-hydroxyxanthone-1-carboxylic acid and some of their derivatives. W. R. Orndorff and W. A. Adamson. J. Amer. Chem. Soc., 1918, 40, 1235—1256.

THE first-named substance is best obtained by heating resorcinol (4 parts) and tetrachlorophthalic acid (5 parts) for 12—15 hours at a temperature not exceeding 131° C. (vapour of boiling xylene). It forms colourless crystals containing 2H<sub>2</sub>O, and the dried substance at its m.pt., 227° C., decomposes into tetrachlorofluorescein and tetrachlorophthalic anhydride. On bromination in glacial acetic acid it yields *o*-3'.5'.5'-dibromo-2'.4'-dihydroxybenzoyltetrachlorobenzoic acid, yellow needles, m.pt. 211°—212° C. The triacetate of the unbrominated acid is converted by 5% alcoholic potassium hydroxide and subsequent acidification into 2,3,4-trichloro-6-hydroxyxanthone-1-carboxylic acid, colourless needles containing 2H<sub>2</sub>O, m.pt. 279°—280° C. (anhydrous). The preceding results, and particularly the fact that the triacetates are not decomposed by boiling alcohol, are interpreted by ascribing to *o*-2'.4'-dihydroxybenzoyltetrachlorobenzoic acid the constitution



(See also J. Chem. Soc., 1918, i., 435.)—C. S.

*Colour reaction of mercury fulminate with phenylhydrazine.* Langhans. See XXII.

#### PATENTS.

*Azo dyes; Gray* —. C. Hagemann, Leverkusen, Germany, Assignor to Synthetic Patents Co., Inc., New York. U.S. Pat. 1,270,290, June 25, 1918. Date of appl., Nov. 27, 1914. Renewed Apr. 16, 1918.

TRISAZO dyestuffs are claimed which dye cotton reddish-grey to greenish-grey shades, fast to light, and on reduction with stannous chloride and hydrochloric acid yield an aromatic aminosulphonic acid, a diamine, and an orthodiaminohydroxynaphthalenesulphonic acid. Special claim is made for the dyestuff which, on reduction, yields 4,5-dichloroaniline-2-sulphonic acid, 2 mols. of 1,4-naphthylene-diaminesulphonic acid, and 1,2-diamino-8-naphthol-6-sulphonic acid.

*Azo dyestuffs capable of being chromed; Manufacture of* —. O. Imray, London. From Soc. of Chem. Ind. in Basle, Switzerland. Eng. Pat. 118,448, Aug. 28, 1917. (Appl. No. 12,348 of 1917.) Addition to Eng. Pat. 16,916, Dec. 1, 1915 (this J., 1917, 78).

SEE U.S. Pat. 1,270,325 of 1918; this J., 1918, 573 A.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### PATENTS.

*Fabric stretching and drying apparatus.* J. W. Shelton, Assignor to Tucson Steam Laundry Co., Tucson, Ariz. U.S. Pat. 1,269,751, June 18, 1918. Date of appl., Dec. 7, 1917.

THE apparatus comprises a perforated drum, a post arranged centrally within the drum, means for stretching a piece of fabric about the drum, a fan journaled around the post within the drum for forcing currents of air outward through the perforations, and means for heating the air currents.—J. F. B.

*Paper-making machines.* E. Partington, Westwood Park, Worcester. Eng. Pat. 118,511, Nov. 3, 1917. (Appl. No. 16,038 of 1917.)

SOME of the drying cylinders over which the web of paper first passes are maintained at a temperature higher than that of the remainder of the cylinders and sufficient to impart to the damp paper enough heat to cause the water therein to evaporate freely as it passes over the lesser heated cylinders. For instance, in a range of 16 cylinders the first five may be heated so as to raise the temperature of the web to 200° F. (93.5° C.) and the temperature of the succeeding cylinders may fall progressively at the rate of about 10° F. (5.5° C.), so that the temperature of the last is about 90° F. (32.5° C.), thus preventing baking and cockling of the paper.—J. F. B.

*Writing-paper and method of making the same.* S. Kuroki, Assignor to Kimpitsu Seishi Kabushiki Kaisha, Ltd., Tokyo. Reissue 14,499, July 30, 1918, of U.S. Pat. 1,234,045, July 17, 1917. Date of appl., Dec. 7, 1917.

SEE this J., 1917, 1004.

*Paper-stock; Apparatus for the treatment of* —. W. G. Fiske, Purfleet. U.S. Pat. 1,273,841, July 30, 1918. Date of appl., Feb. 24, 1917.

SEE Eng. Pat. 103,376 of 1916; this J., 1917, 288.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Method of standardising the fastness of colours.* Robson. See IV.

#### PATENTS.

*Dyeing machines. Fibre-treating machine. Fabric-dyeing machine.* H. M. Dudley, Philadelphia, Pa. U.S. Pats. (A) 1,274,396, (B) 1,274,523, (C) 1,274,524, (D) 1,274,525, Aug. 6, 1918. Dates of appl., (A) Feb. 15, (B) June 1, (C) June 15, (D) July 25, 1917.

(A) IN the dyeing chamber are arranged a series of superposed removable fibre trays adjacent to each other, each tray comprising a non-perforated side wall and a foraminous bottom formed of transverse grate bars, a second chamber below the trays and a third chamber above the trays each containing a series of "upwardly disposed plates." Means are provided for circulating liquid or air through the apparatus and through a testing apparatus similar to but smaller than the main apparatus. (B) IN a receptacle are arranged a series of dyeing chambers having non-perforated sides, each chamber having a fixed foraminous bottom and a movable foraminous top capable of being adjusted and held at any desired position in relation to the bottom. The wall of each dyeing chamber contains a conduit connecting the portion of the receptacle at each end of the dyeing chamber with a testing device, and liquor is circulated by a pump through the apparatus. (C) A fibre-treating machine comprises a receptacle containing an outer foraminous cylinder with a series of spaced parallel members having projections extending upwards and downwards in the receptacle, and an inner foraminous cylinder, spaced from the outer cylinder, with tubular members connecting the openings in the inner cylinder with a supply of liquids, steam, and air. A series of sliding members is provided to abut upon a fabric wound on the cylinder so as to close all the openings

which are not covered by the fabric, also a flexible foraminous covering to enclose the wound fabric tightly on the cylinder. (b) A dyeing chamber has a removable top and a series of hollow reels capable of being revolved within the chamber on a common axis, each reel being composed of a hollow foraminous cylinder with non-perforate flanges extended outwards. A series of conduits within the hollow reels connect the ends of the reels with the inner faces of the foraminous cylinders and allow of liquid being supplied uniformly over the inner faces of the cylinders. Each reel can be revolved independently or locked against rotation, and the interior of a reel is connected with a testing device revolving with it.

—J. F. B.

*Winding and dyeing machine.* W. A. Ainsworth, Assignor to C. N. Mather, Grand Rapids, Mich. U.S. Pat. 1,274,972, Aug. 6, 1918. Date of appl., July 16, 1917.

Thread or yarn is transferred from one holder in the winding machine to another, passing in close proximity to a member impregnated with colouring material, against which it is periodically pressed, so that the colouring matter is intermittently transferred to the thread in its passage through the machine. The means for applying the colour comprises a cylindrical receptacle for containing the colour liquid over which the yarn is adapted to pass, a number of wicks in the receptacle extending a short distance upwards, a shaft parallel to the receptacle, a number of collars, one for each wick, secured on the shaft, and fingers, under which the yarn is adapted to pass, projecting laterally from each collar.—J. F. B.

*Clothes; Cleaning or washing — by means of benzine or benzol.* B. Steffen, Cöthen. Ger. Pat. 306,257, Mar. 24, 1916.

Benzine or benzol which has been used for cleaning clothes, with or without the addition of fresh material, is added to a mixture of dilute sulphuric acid and sodium carbonate which has been allowed to stand until effervescence has ceased, and then distilled by the direct application of steam. The distillate, after settling for 3 or 4 hours, is used again for cleansing purposes.—A. B. S.

*Dyeing machines.* H. M. Dudley, Philadelphia, U.S.A. Eng. Pat. 111,843, Nov. 10, 1917. (Appl. No. 16,508 of 1917.) Under Int. Conv., Dec. 5, 1916.

See U.S. Pat. 1,258,203 of 1916; this J., 1918, 265 A.

*Manufacture of preparations of calcium halogenides and albumin.* Ger. Pat. 306,978. See XX.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Ammonia; Formation of — by heating coke with calcium hydroxide.* R. Glaser. Feuerungstechnik, 1917, 6, 3—S. Z. angew. Chem., 1918, 31, Ref., 248.

On heating coke with a large excess of calcium hydroxide the carbon completely disappears and a large quantity of hydrogen is produced. The yield of ammonia increases with the temperature, amounting at 1145° C. to 40% of the total obtainable from the nitrogen contained in the coke. On addition of nickel the yield of hydrogen is increased and the yield of ammonia reaches a maximum at 630° C., catalytic action of the nickel caus-

ing decomposition of the ammonia at higher temperatures. At 630° C. methane is also formed if nickel is present.—L. A. C.

*Nitrites; Determination of —.* F. Dienert. Comptes rend., 1918, 167, 366—367.

NITRITES react with hydriodic acid according to the equation:  $\text{NaNO}_2 + 2\text{HI} = \text{NaI} + \text{I} + \text{NO} + \text{H}_2\text{O}$ . For the estimation three flasks containing respectively 50 c.c. of 4% potassium iodide solution, 10 c.c. of N/1 sulphuric acid, and the solution of the nitrite, are connected in the order named and the air is displaced by a current of carbon dioxide. By means of this gas the potassium iodide solution is then driven over into the sulphuric acid, and then the mixture in the second flask into the third containing the nitrite solution. The liquid is then made alkaline by the addition of 10 c.c. of a 20% solution of ammonium carbonate and titrated with N/70 arsenious acid solution, of which 1 c.c. = 0.2 mgrm. of nitrous nitrogen. Care must be taken to avoid the presence of oxygen until the solution is made alkaline.—W. G.

*Magnesium oxide; Influence of the temperature of burning on the rate of hydration of —.* E. D. Campbell. J. Ind. Eng. Chem., 1918, 10, 595—596.

FURTHER investigation (see this J., 1909, 1036) has shown that, when it has been ignited above 1200° C., magnesium oxide requires a long time for complete hydration even when immersed in water. For example, magnesite at 1450° C., was only hydrated to the extent of 70% in 6 years. Probably 20 years, or more, would be required for the complete hydration of materials containing free magnesium oxide if burned at temperatures approaching that used for the production of Portland cement.—W. P. S.

*Basic carbonates of copper.* H. R. Dunncliff and S. Lal. Chem. Soc. Trans., 1918, 113, 718—722.

THE determination of the percentage of copper oxide in thirteen samples of commercial copper carbonate has given results which do not support the statement that the ordinary copper carbonate of commerce has the same composition as malachite,  $\text{CuCO}_3 \cdot \text{Cu(OH)}_2$ . The percentage of CuO actually found in the samples varied from 66.16 to 78.60. As the result of attempts to prepare a basic carbonate of approximately constant composition, it has been found that a substance of the formula  $2\text{CuCO}_3 \cdot 5\text{Cu(OH)}_2$  is obtained when a solution of copper sulphate saturated at 14.5° C. is diluted with an equal quantity of water and mixed with a solution containing 5% of sodium carbonate and 5% of sodium bicarbonate.

—H. M. D.

*Minerals [silicates]; Hydrothermal formation of —.* W. J. Müller and J. Koenigsberger. Z. anorg. Chem., 1918, 104, 1—26.

EXPERIMENTS have been made to investigate what products are formed when mixtures containing  $\text{K}_2\text{O}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ , and water are heated together under pressure for varying times at temperatures from 100° to 440° C., in presence or absence of carbon dioxide. The results obtained in presence of carbon dioxide agree better with the observed order of succession of minerals in clefts and cavities in nature than do those without. Below 300° C. potassium feldspar is not formed. At 310° C. in presence of carbon dioxide only quartz is stable, but at higher temperatures aluminosilicates begin to appear. In absence of carbon dioxide zeolites are formed at 100° C., but apparently their limit of stability is reached below 300° C. The opinion is expressed, however, that in no case was a true



equilibrium obtained in the experiments below 400° C. Besides the minerals already mentioned, leucite, potassium nepheline, and pectolite were identified among the products. (See also J. Chem. Soc., Nov., 1918.)—E. H. R.

*Water hyacinth as a source of potash.* Day.  
See XVI.

*Determination of phosphoric acid as magnesium pyrophosphate.* Balareff. See XXIII.

*Use of manna in the determination of boric acid.* Iles. See XXIII.

#### PATENTS.

*Nitric acid; Process for the manufacture of —.* E. Bergve, Notodden. Assignor to Norsk Hydro-Elektrisk Kvaelfstafaktieselskab, Christiania, Norway. U.S. Pat. 1,273,991, July 30, 1918. Date of appl., May 18, 1918.

HIGHLY concentrated nitric acid is prepared by causing a mixture of nitrogen tetroxide, oxygen, and water or dilute nitric acid to react together at about 70° C. and under a pressure of about 20 atmospheres.—W. E. F. P.

*Tin; Oxidation of —. Production of zinc oxide.* H. Maconochie, London, and D. de Ros, Greenhithe, Kent. Eng. Pats. (A) 118,664 and (B) 118,665, Sep. 1, 1917. (Appl. Nos. 12,594 and 12,595 of 1917.)

(A) PREHEATED air or oxygen is blown into or upon metallic tin which has been previously heated to the temperature at which stannic oxide is formed. Additions of preheated tin to the charge are made from time to time in such quantities that the process is operated continuously by the heat of the reaction alone. The tin oxide fume is condensed. (B) Metallic zinc or zinc-containing material, e.g. a mixture of zinc sulphide (ore) and iron, is treated as above.—W. E. F. P.

*Salt; Apparatus for drying —.* T. W. S. Hutchins, Middlewich. Eng. Pat. 118,712, Oct. 5, 1917. (Appl. No. 14,425 of 1917.)

A NUMBER of superposed, horizontal, cylindrical vessels, connected in series within a single casing, are provided with independent hot-air inlets and outlets communicating with common standpipes. Each cylinder has a central, horizontal shaft upon which paddles, serving as combined agitators and conveyors, are fixed, the shafts being intergeared for rotation at high speed, so that while agitated the charge is propelled along adjacent vessels in opposite directions. Means are also provided for passing into the space around the cylinders the products of combustion from the air-heating furnace.—W. E. F. P.

*Peroxide of hydrogen and barium nitrate; Manufacture of —.* F. Cobellis, Philadelphia, Pa. U.S. Pat. 1,273,824, July 30, 1918. Date of appl., Jan. 18, 1917.

BARIUM dioxide is treated with phosphoric acid to produce hydrogen peroxide and barium phosphate, which latter is then treated with a nitrate to form barium nitrate. The barium nitrate is converted into nitric acid and barium dioxide, the latter being used again in the process.

—W. E. F. P.

*Alunite; Process for recovering the constituents of — in useful forms.* H. J. Detwiller, Allentown, Pa. U.S. Pat. 1,274,145, July 30, 1918. Date of appl., Oct. 9, 1917.

POWDERED alunite is digested in boiling nitric acid and the residual silica is separated by filtration. An alkali is added to the filtrate to precipitate the alumina, which is removed by filtration. Any sulphates present are separated by adding barium carbonate, agitating, boiling, and filtering. The filtrate is made acid with nitric acid, boiled to remove the carbon dioxide, and the potassium nitrate is allowed to crystallise out.—A. B. S.

*Formates and oxalates; Manufacture of —.* A. Lackman, Bradford, Pa. U.S. Pat. 1,274,169, July 30, 1918. Date of appl., July 24, 1917.

SODIUM formate is made by passing granular caustic soda through a heated tubular conduit in a direction contrary to that of a current of preheated gas containing carbon monoxide. Sodium oxalate is made by heating sodium formate under similar conditions but at a higher temperature. The waste gases from both operations are used for heating the apparatus used.—A. B. S.

*Potassium content of cement-kiln dust; Process for increasing the solubility of the —.* R. J. Nestell, Assignor to International Precipitation Co., Los Angeles, Cal. U.S. Pat. 1,274,310, July 30, 1918. Date of appl., Jan. 15, 1917.

THE dust from coal-fired cement kilns is treated with water at 100° C., after which the solution is filtered and evaporated to dryness.—W. E. F. P.

*Hydrofluoric acid and hydrofluosilicic acid or their salts; Manufacture of —.* Rüfigerswerke-A.-G., Berlin. Ger. Pat. 306,567, Dec. 17, 1916.

IN the manufacture of hydrofluoric acid, hydrofluosilicic acid, and their salts by fusion of fluorspar or a mixture of fluorspar and silica with acid alkali sulphates, an inorganic compound, e.g., potassium sulphate, sodium sulphate, an acid alkali phosphate, sodium alum, boric acid, etc., is added to the mixture, whereby the fusion takes place more readily and the reaction temperature is reduced.—L. A. C.

*Ammonium sulphite; Process for oxidising — to ammonium sulphate.* H. Danneel and E. Kuhn, Basel, Assignors to Elektrizitätswerk Lonz, Gampel, Switzerland. U.S. Pat. 1,274,247, July 30, 1918. Date of appl., July 21, 1917.

SEE Eng. Pat. 105,906 of 1917; this J., 1917, 1093.

*Phosphorus; Manufacture of —.* H. O. H. Wenman, Bishop's Castle. U.S. Pat. 1,274,479, Aug. 6, 1918. Date of appl., Dec. 7, 1917.

SEE Eng. Pat. 113,584 of 1917; this J., 1918, 206 A.

#### VIII.—GLASS; CERAMICS.

*Continuous kilns; Operation of —.* R. Seydel. Tonind.-Zeit., 1918, 42, 482-483.

THE chief difficulties in the operation of a continuous kiln are due to the irregular passage of the air and hot gases through it. This is particularly the case with Hoffmann kilns in which the fuel is burned in hollow pillars formed by the bricks to be burned. If sufficient care be taken to obtain a uniform draught in various parts of the kiln, various auxiliary methods, such as boxing

files, are unnecessary. Difficulties arise when the finishing point and the point of collapse of the goods lie too close together; the admixture of fire-clay may help to overcome this objection. In a Hoffmann kiln with a tunnel 240—300 ft. in length, of a suitable width, with rounded ends of the same cross-sectional area as the straight portion, and provided with suitable hot air flues, the fire should travel round at the rate of 33 ft. per 24 hours. Excellent results may be obtained without difficulty in a wide kiln, with 5 or 6 feed holes, 3 ft.—4 ft. 6 in. apart, and the setting and drawing of the goods is easier than in a narrow kiln. It is important that the area of the rounded ends should be sufficiently large; the reduction usually made at this part of the kiln causes many difficulties in working. The discharge of black smoke from the chimney is an indication that fuel is being applied too soon, or that the air is not hot enough for complete combustion. Sometimes the air is too cool because too much is passing through the kiln; on reducing the quantity no smoke is produced.—A. B. S.

*Hydrothermal formation of minerals [silicates].*  
Müller and Koenigsberger. See VII.

#### PATENTS.

*Glass articles; Method and means for discharging molten metal in the manufacture of —.* R. E. McCauley, London. Eng. Pat. 118,581, May 21, 1918. (Appl. No. 8472 of 1918.)

THE direct flow of molten metal from an extension of a glass tank to the discharge aperture is deflected by a transverse L-shaped or semi-cylindrical bridge or dam of refractory material, mounted on a vertical pipe surrounding the aperture, so that the metal is compelled to circulate about the latter before passing through. By this means the customary chilling of part of the discharged glass is avoided. The amount of glass discharged is controlled by a hollow plug as described in Eng. Pat. 113,665.—A. B. S.

*Reinforced sheet glass and method of making same. Means for making reinforced sheet glass.* C. and V. Shuman, Philadelphia, Pa. U.S. Pats. (A) 1,274,205 and (B) 1,274,206, July 30, 1918. Date of appl., Nov. 22, 1916.

(A) Two sheets of glass, each having a thin coating of tightly adherent celluloid, are united thereby to an interposed relatively thick sheet of celluloid, so as to form a composite sheet, sufficient heat and pressure being applied to cause the celluloid surfaces to weld. The exclusion of all air may be secured by maintaining the edges of the sheets under the influence of a vacuum. (B) A shallow sealed compartment with flexible metallic sides contains the sheets to be united and is connected to a vacuum pump. It is placed inside a larger vessel containing fluid. Means are provided for varying the temperature and pressure of the fluid as required.—A. B. S.

*Glass-working.* F. L. O. Wadsworth, Pittsburgh, Pa., Assignor to Ball Brothers Glass Manufacturing Co., Muncie, Ind. U.S. Pat. 1,275,178, Aug. 6, 1918. Date of appl., July 31, 1914.

SMALL quantities of glass are successively segregated from the main mass of molten metal, and the segregated portion is ejected by means of a hot gas under pressure from below the surface of the molten metal first into a receptacle, and then, when this is full, the remainder is returned to the main mass of metal at such a point that it cannot return to the segregation point until after a considerable lapse of time.—A. B. S.

*Dental cements.* W. S. Crowell, Philadelphia, U.S.A. Eng. Pat. 118,701, Sep. 26, 1917. (Appl. No. 13,845 of 1917.)

THE cement or filling is composed of an ordinary dental oxyphosphate of zinc powder, an orthophosphate or other salt of silver, and phosphoric acid. The silver phosphate dissolves in the solution, exhibiting characteristic germicidal, antiseptic, sedative, and coagulative properties. When the mass sets, zinc phosphate and neutral silver phosphate are formed, and further action of the silver on the tissue is inhibited. Alternatively, silver phosphate, carbonate, oxide, or hydroxide may be dissolved in phosphoric acid and the solution mixed with ordinary dental cement powder.

—A. B. S.

*Dental filling of vitreous or porcelain-like structure resembling enamel; Production of a —.* F. Schoenbeck u. Co., Leipzig. Ger. Pat. 306,837, Sep. 6, 1907.

CRYOLITE is fused with silica and calcium oxide, the resulting product then being powdered and worked into a plastic mass with the addition of a phosphoric acid containing dissolved aluminium hydroxide.—D. F. T.

*Glass; Conveyance of molten —.* R. E. McCauley, London. Eng. Pat. 118,585, May 31, 1918. (Appl. No. 9030 of 1918.)

*Potters' slip, powdered substances, and the like; Sifters for treating —.* A. C. Harrison, Stoke-on-Trent. Eng. Pat. 118,800, Mar. 5, 1918. (Appl. No. 3823 of 1918.)

*Glass; Apparatus for making plate —.* J. H. McKelvey, Kirkwood, and C. F. Ryan, St. Louis, Mo. U.S. Pat. 1,274,916, Aug. 6, 1918. Date of appl., June 7, 1915.

SEE Eng. Pat. 109,634 of 1916; this J., 1917, 1129.

*Dental cements.* W. S. Crowell, Philadelphia, U.S.A. Eng. Pat. 118,702, Sep. 26, 1917. (Appl. No. 13,846 of 1917.)

SEE U.S. Pat. 1,244,296 of 1917; this J., 1917, 1275.

*Kiln.* U.S. Pat. 1,272,495. See I.

#### IX.—BUILDING MATERIALS.

*Mortar; New ingredients for —.* H. Kühl. *Toniud.-Zeit.*, 1918, 42, 17—18, 37—38, 53—54.

THE author reports on the use of pure hydrated lime and hydrated cement (Mühlen, Ger. Pats. 294,825, 300,397 and 301,118; this J., 1916, 1109) as ingredients of mortar. Tests of mortar made with hydrated lime (produced by passing slaked lime through an air-separator) showed no expansion. Lime-sand bricks made with this lime are much stronger than those made with ordinary slaked lime. Pure hydrated lime should also meet a widespread want in the plaster trade. The coarser silicious and aluminous particles which are removed from slaked hydraulic lime by an air-separator, yield a good bag lime when ground with quicklime, provided that the additional slaking required by such limes is properly effected. Calcined marl or cement-slurry which has been burned with an excess of lime and then hydrated by steam also produces a non-expanding mortar. The setting time varies with the percentage of lime and the mode of preparation of the material. The samples



examined passed the standard tests and also the boiling and hot water test satisfactorily. Tests of the tensile strength were also satisfactory considering the high percentage of lime. These new ingredients facilitate the preparation of new kinds of dry mortars, e.g. mortar of pure hydrated lime, cement mortars, and various mixtures for reinforced concrete.—A. B. S.

#### PATENTS.

*Plastic material; Manufacture of a fire-resisting — and its combination with and its application to other materials for constructional purposes.* R. Y. Ardagh, Hove. Eng. Pat. 118,768, Jan. 4, 1918. (Appl. No. 216 of 1918.)

SILICATE cotton or slag wool is screened to separate slag dross and other coarse particles, and is then passed over a finer sieve, through which the light wool fibres and minute mineral particles pass, but grains larger than those of a poppy seed or turnip seed are retained. The fibre is then ground in a crushing or rolling mill disintegrator, and the coarser material left on the fine sieve is ground by edge runners until it will pass completely through a fine gauze sieve. The two finely ground materials are mixed in proportions varying from 1:1 to 1:6 according to the density required, and the mixture is added to a sufficient quantity of a solution of water-glass of 70° Tw. (sp. gr. 1.35) to produce a plastic paste. The material may be moulded into sheets or columns or it may be applied as a coating to existing structural materials. The hardening may be facilitated by keeping it in a drying room at 100°–130° F. (38°–54° C.). Sawdust, cork, or other powders may be incorporated with the material.—A. B. S.

*Wood; Process of preserving —.* M. Landau, Berlin. U.S. Pat. 1,274,171, July 30, 1918. Date of appl., Jan. 25, 1916.

THE wood is preserved by introducing into it aqueous solutions of trioxymethylene and inorganic salts soluble in water, such as alkali fluorides.

—A. B. S.

*Process for increasing the solubility of the potassium content of cement-killn dust.* U.S. Pat. 1,274,310. See VII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Iron; Chemical equilibria in the reduction and cementation of —.* R. Schenk. Z. Elektrochem., 1918, 24, 248–255. (See also this J., 1907, 692; 1915, 554; 1916, 115.)

A COMPARISON of the temperature pressure equilibria in the reaction  $C + CO_2 \rightleftharpoons 2CO$  with those involved in the interaction of iron and carbon monoxide, whereby the iron is carburised, shows that in this latter effect elementary carbon resulting from the decomposition of the carbon monoxide is involved. This carbon in the presence of excess of ferrite is transformed into a carbide, and hence its presence is not revealed by solution of the iron in hydrochloric acid. Equilibrium between ferrite-carbide-ferrous oxide and the oxides of carbon occurs at low pressures, the content of carbon monoxide in the gaseous phase, however, slightly exceeding that of the  $C + CO_2 \rightleftharpoons 2CO$  equilibrium. The carbide thus formed is different from cementite,  $Fe_3C$ , which on heating to the  $Ac_1$  point is

decomposed into iron and carbon. Thermodynamical reasoning leads to the conclusion that this carbide contains less carbon than  $Fe_3C$ . In the manganese-carbon system an analogous carbide containing about 3% of carbon, corresponding to  $Mn_3C$  or  $Mn_2C$ , has been detected. The name bunsenite is suggested for this new iron carbide.

—F. C. Th.

*Grey iron; Melting of — in an oil-fired furnace.* K. Abeking. Stahl u. Eisen, 1918, 38, 792–795.

THE author describes the production of grey iron castings of special quality in furnaces of the oil-fired "drum" pattern without a crucible. The first cost of an installation for  $\frac{1}{2}$ -ton melts is given as M.5000–6000. The furnace, which takes up little room and is equally satisfactory for other metals than iron, is much more costly to run than a cupola. The iron can be run in a very fluid condition for thin castings, and less labour is required than for a cupola. The tensile strength of the cast iron ranges from 21.7 to 30 kilos. per sq. mm. The total carbon may vary from 2.45 to 3.26%, and the sulphur from 0.062 to 0.12%. The Brinell hardness of the iron varies from 160 to 180. The charge used consists of 5–10% of steel scrap, 20% of hematite iron, and the rest foundry irons. The silicon loss during melting reaches 15–20%. The average heat lasts 2 hours.—F. C. Th.

*Grey cast iron; Method for the prevention of growth in —.* J. E. Hurst. Iron and Steel Inst., Sep., 1918. [Advance copy.] 5 pages.

GROWTH in cast iron when this is repeatedly heated has previously been shown to be due to the penetration of oxidising gases along the plates of graphitic carbon. If a protecting "case" could be given, so as to prevent the entry of oxidising gases, growth of the interior grey iron would be prevented. An example is given of a grey cast iron pipe subject to a temperature of 800°–900° C., which was heavily coated with rust, having been thus protected by a decarburisation of the skin produced by heating in a strongly oxidising atmosphere; the graphite was thereby replaced by the phosphide eutectic. Possible methods by which graphite might be removed without the production of cavities are by superficial decarburisation followed by the liquation of the phosphide eutectic into the remaining cavities, or by conversion of graphite into austenite solid solution at a temperature above 900° C. The author has used the decarburisation method for protecting dies and moulds from growth, i.e. by annealing at 900°–950° C. in ordinary brown rust (iron oxide) for 72 hours. The results are considerably improved by raising the annealing temperature to 975°–1000° C., but troubles from distortion are then experienced.

—C. A. K.

*Malleable cast iron; Phosphorus in —.* J. H. Teng. Iron and Steel Inst., Sep., 1918. [Advance copy.] 19 pages.

FOLLOWING the suggestion of Turner (this J., 1917, 793) that a poorer class of irons should be employed for making malleable castings, the effect of phosphorus varying from 0.05 to 0.5% was examined. Two series of test bars were prepared: one from a very pure American washed white iron, and the other from locally obtained pig irons, the phosphorus being added in the form of a phosphoric iron (4% P) prepared by adding red phosphorus to molten American washed white iron. The maximum stress, elongation, and reduction in area were lowered with increasing phosphorus, the most marked effect showing at about 0.25% P. When tested for malleability by bending, the bending

angle progressively decreased as the phosphorus increased, with again a marked alteration at about 0.25% P, while the hardness increased gradually, with a sudden rise on the appearance of free iron phosphide. One effect of phosphorus noticed was the larger grain size of the crystals. The approximate limit for commercial purposes is given as 0.2% P, as the ill effects of phosphorus are not appreciable until that figure is exceeded. The presence of a considerable amount of silicon and manganese is of more importance than the absence of sulphur with regard to successful annealing, and iron sulphide found in the iron ore which was used as the annealing medium is attributable to the presence of sulphur compounds in the reducing atmosphere existing in the annealing furnace.

—C. A. K.

*Open-hearth furnace design: Principles of —.* C. H. F. Bagley. Iron and Steel Inst., Sep., 1918. [Advance copy.] 19 pages.

THE author discusses the design of open-hearth furnaces from both scientific and practical points of view, and expresses the view that scope for improvement exists in British practice. The most important requirement is an exceedingly high temperature, as the output and efficiency are functions of the average temperature attainable rather than of the heat units generated, and the view is expressed that the object of regenerating is not so much the economical recovery of heat units, but rather that the highest flame temperature may be reached. With the same object in view the gas and air used for combustion should be dry.

—C. A. K.

*Open-hearth furnaces; Utilisation of waste heat from — for the generation of steam.* T. B. Mackenzie. Iron and Steel Inst., Sep., 1918. [Advance copy.] 24 pages.

FLUE gases from the regenerators of an open-hearth steel furnace are passed through a water-tube boiler for the generation of steam. A water-sealed valve is arranged to send the gases either to the boiler, or direct to the chimney, and a fan discharges the gases from the boiler economiser back into the chimney. Explosion doors are required to prevent accidents when the furnace valves are thrown over. From a series of trials made, the average weight of steam generated per ton of ingots was 987.5 lb., and under more favourable conditions it is thought that at least 1200 lb. of steam would be obtained. Assuming a steam consumption at the mill engines of 3200 lb. per ton of ingots, the saving in boiler fuel is considerable. The average temperatures of the gases in one test were: entering boiler, 577° C.; entering economiser, 303° C.; entering fan, 171° C.—C. A. K.

*Basic steel; Influence of some elements on the tenacity of —.* A. McWilliam. Iron and Steel Inst., Sep., 1918. [Advance copy.] 13 pages.

AN attempt has been made to devise a formula on a rational basis which would give reasonably correct results for the tensile strength of steels within and above the range of the British Standard Specifications for Structural Steels (28 to 33 tons per sq. in.). Assuming values for the various elements that are fairly well established for the lower tempers, the strength of absolutely pure iron was estimated to be about 38,000 lb., or 17 tons per sq. in. The unit of any element is taken as 0.01%. The results were obtained by trial and error on a very large number of steel tests made during inspection work. The effects of the several elements may be summarised. Phosphorus adds 1000 lb. per unit, carbon 800 lb. (1000 lb. where the carbon

is over 0.4%); silicon 120 lb.; manganese 100 lb. (150 lb. where the carbon is over 0.4%). Sulphur is ignored. The formula now employed for obtaining the maximum load per sq. in. is:—

$$ML = 38,000 + [800 + 4(C - 20)]C + 120Si + [100 + 2(C - 20)]Mn + 1000P.$$

The formula has given good results for a large number of Indian basic steels, and the agreement with Harbord's basic Bessemer series is also good, but hitherto the results have not been as favourable for acid steels as for basic steels.—C. A. K.

*Steel; Cooling of — in ingot and other forms.* J. E. Fletcher. Iron and Steel Inst., Sep., 1918. [Advance copy.] 40 pages.

MOLTEN steel, however carefully melted, contains a considerable volume of gas in suspension, and the finishing additions to the bath lead to reactions which continue in the fluid metal even when in the ladle and ingot mould. From observations of the metal in ingot moulds and in sand moulds for steel castings, the author concludes that the shrinkage is closely connected with the escape and reduction in volume of the occluded gas on cooling. If the imprisoned gas is allowed to escape from the metal under a liquid head, the cavities are filled in and contraction of the casting is lessened. The primary crystallites forming on the surface of the mould expel the gas which they contained when liquid into the interior mother liquor, and the gas travels to the mass centre of the ingot, the segregatory constituents being driven in the same direction. By a too rapid freezing of the metal, piping is produced. The lines of most rapid freezing and cooling follow the paths of steepest temperature gradients, these being the bisecting planes of the mould sides and those bisecting the base angles, and time teeming and the efficacy of slow pouring in comparatively heavy moulds are only now being generally understood. It is evident that the temperature gradient is an index to the chilling effect; the steeper the gradient, the more carbide is retained in the metal. It might be expected that with steels of higher carbon percentage than 0.5%, the outer skin would be hard, due to the chilling action of the mould, but since the temperature gradient rapidly decreases as the axis is approached, this hardening is removed by an annealing effect unless the mean temperature of the ingot falls rapidly to below the Ac point.—C. A. K.

*Molybdenum [in ferromolybdenum]; Estimation of — as lead molybdate.* R. Strebing. Oesterr. Chem. Zeit., 1917, 20, 226—228. Z. angew. Chem., 1918, 31, Ref., 278.

FROM 0.5 to 1.0 grm. of ferromolybdenum is decomposed by fusion with 10 grms. of sodium peroxide in an iron crucible, the melt dissolved in warm water, boiled for a short time, and washed into a 500 c.c. flask. After settling, 100 c.c. of the liquid is neutralised with acetic acid, the solution boiled for a short time, and the following freshly prepared solutions quickly added in succession: (1) 2–5 grms. of lead acetate and 2 c.c. of acetic acid in 50 c.c. of water, and (2) 10 grms. of ammonium acetate in the same quantity of water. Boiling is continued for a few minutes and the solution is then allowed to stand for six hours, after which it is filtered through filter-pulp. The precipitate is washed with about 200 c.c. of ammonium acetate solution, and is then transferred, together with the moist filter-pulp, to a beaker in which it is heated to boiling with 20 c.c. of dilute nitric acid, after which a solution of 10 grms. of ammonium acetate and 5 c.c. of dilute acetic acid in 50 c.c. of water is added. The solution is boiled for 1 to 2 mins., filtered after 12 hours, and the residue, after having



been washed with hot ammonium acetate solution until the filtrate is free from lead, is dried, incinerated at the lowest possible temperature, ignited, and weighed as  $PbMoO_4$ .—L. A. C.

[Gold] ores; Roasting of sulphotelluride — for amalgamating and cyaniding. A. L. Blomfield and J. M. Trott. Amer. Inst. Min. Eng., Sep., 1918. Eng. and Min. J., 1918, 106, 400—404.

THE Golden Cycle Mining and Reduction Co., Colorado Springs, treats Cripple Creek ores practically free from base metals, containing 1.8% of sulphur, and varying amounts of lime (1.6—5%). The ore is bedded, the beds (class A, 66% of total ore, not more than 2%  $CaO$ ; class B more than 2%  $CaO$ ) being made as large as possible; it is crushed in ball mills so that not more than 30% remains coarser than 10-mesh, and roasted for 6 hours in lignite-fired Edwards Duplex furnaces; the hearth of the furnace slopes  $\frac{1}{2}$  in. per foot towards the discharge end, which causes the ore to move forward under the action of the rabbles. The roasting results in the formation of calcium sulphate which, unless an excess of air is maintained, becomes converted into sulphide. The sulphate is very troublesome in the subsequent leaching, as it crystallises out and causes the pulp to set, but if the roasting is conducted at a temperature above  $870^\circ C$ , calcium sulphate is rendered practically insoluble. The calcine traverses a cooling-hearth and is then ground in Chilean mills under cyanide solution; the pulp is passed through a Dorr bowl classifier, and the sand and slime are cyanided separately.—W. R. S.

[Gold.] The Crowe system of de-aerating cyanide solutions. T. B. Crowe. Amer. Inst. Min. Eng., Sep., 1918. Eng. and Min. J., 1918, 106, 413.

VACUUM precipitation has been adopted at the Portland Gold Mining Co.'s mill (Colorado Springs). A continuous vacuum is applied to the cyanide solution during its flow to the precipitation presses, whereby the dissolved air is removed from the liquid. The zinc consumption has thus been halved, and that of cyanide nearly so, because the addition of solid cyanide at the head of the precipitation plant is no longer necessary for satisfactory gold precipitation.—W. R. S.

Radium ores; Metallurgical treatment of —. R. B. Moore. Amer. Inst. Min. Eng., Sep., 1918. Eng. and Min. J., 1918, 106, 410—412.

THE average radium content of commercial ores is 5—10 mgrms. per ton. The treatment aims at first obtaining a barium radium sulphate concentrate, which may be effected by several methods. The ore may be boiled with alkaline carbonate solution or fused with the solid salt, whereby uranium and vanadium are rendered soluble. The residue is leached with hydrochloric acid and the solution precipitated with sulphuric acid. In the Radcliffe process the ore is fused with saltcake, the fusion product crushed and agitated with water, and the liquor separated from the heavy unattacked material. The turbid solution is left to settle, when it deposits sulphates of barium, lead, and radium. In the U.S. National Radium Institute, the material is boiled with 40% nitric acid, in which the alkaline earth sulphates are appreciably soluble. The hot acid solution is rapidly filtered under pressure through a plate of "filtros" or ahundum forming the bottom of an earthenware filter set in a steel shell. The filtrate deposits barium and radium sulphates. The sulphate concentrate, which should carry 1 mgrm. of radium per kilo., is reduced by ignition with charcoal, and

the resulting sulphide dissolved in hydrochloric acid. Further concentration is effected by fractional crystallisation of the mixed chlorides, and, later on, of the mixed bromides from acid solution, the radium accumulating in the less soluble fractions.—W. R. S.

Zinc content of furnace ashes. O. Mühlhaeuser. Metall u. Erz, 1918, 15, 259—265.

THE zinc content of the ashes from the muffles varied from 2.9 to 14.5%, the total loss being on the average about 10%. The ashes near the throat of the muffle contained about 35% Zn, compared with an average of just over 4% in the hinder part. The zinc present varied from 2.9 to 57% along the whole length of the muffle, due chiefly to the lower temperature at the throat, but also to the deposition of zinc oxide formed by the action of air entering between the muffle and the condenser. The zinc content of the ash increases but slowly at first from 3 to 6% over a distance of 105 cm. from the rear end of the muffle, finally rising rapidly in the last 20 cm. of the muffle. The zinc is present in the ashes chiefly as oxide and sulphide; if iron is present the latter is reduced to metal.—F. C. Th.

Aluminium pig; Remelting of — in the electric furnace. D. D. Miller. Chem. and Met. Eng., 1918, 19, 251—254.

AN electric remelting furnace in use at the works of the United States Aluminium Co., at Massena, N.Y., U.S.A., is of the stationary resistance type, rated at 500 kilowatts, and designed for single-phase, 25-cycle operation. It consists of a rectangular metal casing, having a door at each end and three rabbling doors on the side opposite the tap-hole. The hearth is bowl-shaped and capable of holding 3 or 4 tons of molten metal. Resistor troughs, rectangular in cross-section and open at the top, made of highly refractory carbide mixed with a binder, are disposed along each side of the interior; these are filled with resistor material (finely broken carbon or graphite) and supported on separate brick piers. Current enters the trough through large copper terminals attached at the ends; and the heat generated is radiated mainly to the furnace roof and deflected thence to the hearth. The temperature is regulated by voltage control, for which purpose a special regulating transformer, an indicating and integrating wattmeter, and a thermo-electric pyrometer are provided. The voltage actually employed ranges from 500 down to 240; and, with continuous operation, the furnace has a capacity of 1 ton per hour, and a daily output of 20 tons. As compared with oil- and coal-fired furnaces previously employed, the metal losses, particularly with alloys containing volatile constituents, are much smaller and the products more uniform; the cost of electric power also compares very favourably with the increased cost of fuel oil. In addition to saving space per ton of metal melted, the electric furnace requires less labour, is cleaner and healthier for the workmen, and offers obvious advantages in the control of atmosphere, temperature, and application of heat.—W. E. F. P.

Antimony in the Transvaal. N. M. Galbreath. J. S. Afr. Assoc. Anal. Chem., 1918, 1, [2], 9—10.

THE deposits of the Murchison Range (N.E. Transvaal) contain up to 10% Sb, and 4 dwt. Au per ton.\* Part of the latter is apparently enclosed in the stibnite, as cyaniding does not give a good extraction. Liquefaction on sloping triangular hearths has been tried; one-third of the antimony is thus ex-

tracted, while the bulk of the gold remains in the residue. Flotation was fairly successful on an experimental scale (cf. this J., 1915, 964); 8 lb. of lubricating oil and 9 lb. of sulphuric acid were used, the extraction reaching 60% for antimony and 80% for gold.—W. R. S.

**Chromium and carbon. High temperature investigations.** XI. O. Ruff and T. Foehr. Z. anorg. Chem., 1918, 104, 27—46.

A CHEMICAL and metallographic study of the alloys of carbon and chromium. Alloys containing up to 8.5% carbon are completely soluble in hot 24% hydrochloric acid. The two constituents present are free chromium and a carbide  $\text{Cr}_3\text{C}_2$ . By continued extraction of the powdered alloy with cold 2N hydrochloric acid until all the free chromium is dissolved, the carbide is obtained in the form of silvery crystals, sp. gr. at  $24.8^\circ/4^\circ\text{C}$ , 6.915, m.p.t.,  $1665^\circ\text{C}$ , which are not attacked by *aqua regia*. An alloy containing 8.5% carbon consists of practically pure carbide. Microscopic sections of this alloy show a uniform mass of crystals, but alloys less rich in carbon show crystals of the carbide embedded in a eutectic. Alloys with over 8.5% carbon contain no free chromium, but show the presence of two carbides,  $\text{Cr}_3\text{C}_2$  and  $\text{Cr}_7\text{C}_3$ , and a variable amount of graphite. The carbon content of alloys saturated with carbon increases as the temperature of preparation is raised. At  $1840^\circ\text{C}$ . the carbon content is 12.42%, at  $2567^\circ\text{C}$ . 23.12%, the excess of carbon at higher temperatures being present as graphite. When these alloys are treated with hot 24% hydrochloric acid the carbide  $\text{Cr}_3\text{C}_2$  dissolves, whilst a residue of graphite and  $\text{Cr}_7\text{C}_3$  is left behind. After extraction of the graphite the carbide  $\text{Cr}_7\text{C}_3$  remains in a pure condition. Its sp. gr. at  $21.3^\circ/4^\circ\text{C}$ . is 6.683 and its melting point  $1890^\circ \pm 10^\circ\text{C}$ . The existence of these acid-resisting chromium carbides explains the valuable properties of other alloys, particularly those of iron, containing chromium and carbon. The carbide  $\text{Cr}_3\text{C}_2$  has strong reducing properties at high temperatures (above  $1850^\circ\text{C}$ ), reducing magnesia, alumina, and zirconia. Chromium alloys saturated with carbon boil at about  $2570^\circ\text{C}$ . under 8—14 mm. pressure, the vapour being pure chromium. (See also J. Chem. Soc., Nov., 1918.)—E. H. R.

**White metal; Determination of antimony in —.** E. W. Kaiser. Metall., 1917, 320—321. Z. angew. Chem., 1918, 31, Ref., 230.

ONE GRM. of drillings is dissolved in 20 c.c. of concentrated sulphuric acid, cooled, and diluted with 50 c.c. of water. 15 c.c. of concentrated hydrochloric acid is added and the solution boiled till all smell of chlorine has disappeared. 100 c.c. of water is added when cold and 15 c.c. of a solution containing per litre 67 grms. of manganese sulphate, 135 c.c. of phosphoric acid (sp. gr. 1.7), and 130 c.c. of concentrated sulphuric acid. The mixture is titrated with  $\text{N}/10$  permanganate standardised against pure antimony powder. If tin and copper are present ( $\% \text{Sn} \times 0.4 + \% \text{Cu} \div 100$  c.c. is deducted from the result of the titration. Arsenic may be subtracted directly. If more than 0.3% iron is present the preliminary solution must be made in hydrochloric acid and potassium chlorate and the antimony precipitated from the diluted solution by an iron rod, filtered off, and treated as above. For the estimation of tin the antimony is precipitated and filtered off through a filter strewn with iron filings. Carbon dioxide is passed through the filtrate, which is titrated with iodine and starch.

—F. C. Th.

**Heat-content-temperature curves of metals.** F. Wüst, A. Meuthen, and R. Durrer. Stahl u. Eisen, 1918, 38, 177—781.

MEASUREMENTS were made of the total heat, specific heat, latent heat, and heat of transformation of many metals with the following results:—

$$\text{Total heat} = a + bt + ct^2; \text{ mean specific heat} = at^{-1} + b + ct; \text{ true specific heat} = b + 2ct.$$

Metal	Temperature interval $^\circ\text{C}$ .	a	b	$c \times 10^6$
Chromium ..	0—1500	—	0.10233	33.47
Molybdenum ..	0—1500	—	0.06162	10.99
Tungsten ..	0—1500	—	0.03325	1.07
Platinum ..	0—1500	—	0.03121	3.54
Tin ..	0—232	—	0.06829	—
.. (liquid) ..	232—1000	14.33	0.07020	-18.30
Bismuth ..	0—270	—	0.03141	5.22
.. (liquid) ..	270—1000	10.31	0.03107	5.41
Cadmium ..	0—321	—	0.05550	6.28
.. (liquid) ..	321—1000	6.30	0.06952	6.37
Lead ..	0—327	—	0.03591	-11.47
.. (liquid) ..	327—1000	6.07	0.02920	3.30
Zinc ..	0—419	—	0.08777	43.48
.. (liquid) ..	419—1000	14.34	0.13340	-16.10
Antimony ..	0—630	—	0.05179	3.30
.. (liquid) ..	630—1000	39.42	0.05090	2.96
Aluminium ..	0—657	—	0.22200	38.57
.. (liquid) ..	657—1000	102.39	0.21870	24.00
Silver ..	0—961	—	0.05725	5.48
.. (liquid) ..	961—1300	53.17	0.00710	28.30
Gold ..	0—1064	—	0.03171	1.30
.. (liquid) ..	1064—1300	26.35	0.01420	8.52
Copper ..	0—1084	—	0.10079	3.05
.. (liquid) ..	1084—1300	130.74	-0.04150	65.6
Manganese (a) ..	0—1070	—	0.12037	25.41
.. (b) ..	1130—1210	-7.41	0.17700	—
.. (liquid) ..	1230—1250	3.83	0.19800	—
Nickel (a) ..	0—320	—	0.10950	52.40
.. (b) ..	330—1450	0.41	0.12931	0.11
.. (liquid) ..	1451—1520	50.21	0.13380	—
Cobalt (a) ..	0—950	—	0.09119	40.77
.. (b) ..	1100—1478	22.00	0.11043	14.57
.. (liquid) ..	1478—1600	57.72	0.14720	—
Iron (a) ..	0—725	—	0.10545	56.84
.. (b) ..	785—919	-1.63	0.1592	—
.. (c) ..	919—1405	18.31	0.14472	0.05
.. (d) ..	1405—1528	-77.18	0.21416	—
.. (liquid) ..	1528—1600	70.03	0.15012	—

For iron the A2 change at  $725^\circ$ — $785^\circ\text{C}$ . is accompanied by a heat change of 6.56 cal. per gm., A3 by 6.67, and A4 at  $1405^\circ\text{C}$ . by 1.94. The transformation in manganese at  $1070^\circ$ — $1130^\circ\text{C}$ . results in an evolution of 24.14 cal. per gm., in nickel at  $320^\circ$ — $330^\circ\text{C}$ . of 1.33, and in cobalt at  $950^\circ$ — $1100^\circ\text{C}$ . of 14.70 cal. The hysteresis in iron at A3 is  $8^\circ\text{C}$ . A2 and A4 both being reversible. The following latent heats of fusion in cal. per gm. were determined:—Tin 13.79, bismuth 10.23, cadmium 10.81, lead 5.47, zinc 23.01, antimony 38.86, aluminium 93.96, silver 26.02, gold 15.87, copper 40.97, manganese 36.65, nickel 56.08, cobalt 58.23, and iron 49.35.

—F. C. Th.

#### PATENTS.

**Thomas [steel] process; Method of improving the —.** F. Wüst, Aix-la-Chapelle, Germany. Eng. Pat. 107,770, July 5, 1917. (Appl. No. 9736 of 1917.)

IN the application of the Thomas process a large amount of iron is burnt and lost in the slag during the oxidation of the phosphorus. It is proposed to add a proportion of completely refined metal from a previous charge to the pig iron. The process is then carried out in the usual way, the burning of the carbon being greatly accelerated by the high initial temperature of the charge. During the blowing the high heat of oxidation of phosphorus is distributed over the whole charge, and excessive rise in temperature at the end of the heat is thus avoided, with consequent saving of metal, and it becomes possible to produce economically steel of a



lower phosphorus content than hitherto. Part of the molten completely refined metal may be left in the furnace for the following charge, or may be added to the charge in a preliminary pig-iron mixer.

—C. A. K.

*Magnetic material [iron and iron alloys] for use in static transformers and other electrical apparatus; Treatment of —.* A. F. Berry, London. Eng. Pat. 118,425, June 30, 1917. (Appl. No. 9489 of 1917.)

IRON or an alloy of iron or other material in which high magnetic properties are desired is subjected to an alternating magnetising and demagnetising force whilst kept under a vacuum and in a heated condition. This may be done by surrounding the material with an insulated coil of wire through which an alternating current is passed. In the manufacture of such material it may be subjected in vacuum to such force during the time it is molten and afterwards when in a heated solid condition.

—C. A. K.

*Ingot moulds for the manufacture of steel.* B. Talbot, Middlesbrough. Eng. Pat. 118,488, Sep. 27, 1917. (Appl. No. 13,966 of 1917.)

THE enlarged head of an ingot mould is lined with a slag (*e.g.* blast-furnace slag) which will melt at the casting temperature of steel. The slag lining may be formed by casting molten slag round a core of suitable section, fitted inside the recessed head of the mould, or may be separately cast, with or without the addition of iron or steel turnings and fitted in the solid form. When the liquid steel is poured into the mould, the face of the slag lining fuses and, floating on the top of the steel, causes the steel to remain liquid in the centre, and the segregation of impurities to be brought nearer to the top of the ingot. A cover plate of slag may be used to retain further the heat in the head of the mould.

—C. A. K.

*Cast-iron articles; Art of permanently heat-expanding —.* W. A. Knapp, Assignor to C. Schwartz, Washington, D.C. U.S. Pat. 1,274,896, Aug. 6, 1918. Date of appl., Oct. 28, 1915.

IRON castings are uniformly heated throughout to a temperature between 1400° and 1600° F. (760°–870° C.), and then cooled immediately, the treatment being repeated until the desired degree of expansion is attained.—C. A. K.

*Silicon-iron alloys; Prevention of graphite precipitation in the manufacture of acid-resisting high —.* Maschinenfabrik Esslingen, Esslingen. Ger. Pat. 306,001, Nov. 20, 1917.

IN the manufacture of silicon-iron castings containing 12–18% Si for acid-resisting purposes, low-carbon iron and ferrosilicon are melted simultaneously in cupolas, tapped, and mixed in a ladle, and the castings poured.—F. C. Th.

*Heat treatment; Indicating the condition of steel, iron, or other magnetisable metal during —.* L. W. Wild and E. P. Barfield, London. Eng. Pat. 118,732. (Appl. Nos. 16,072, Nov. 3, 1917, and 1905, Feb. 1, 1918.)

IN the apparatus described in Eng. Pat. 112,894 of 1917 (this J., 1918, 153 A), a compensating device is provided to prevent the indications being affected by small accidental fluctuations in the current supplied to the primary coil.

*Cementation of iron and steel.* W. Beyer, Leipzig-Stötteritz. Ger. Pat. 306,037, Nov. 7, 1917.

THE article to be carburised is embedded in carburising material within a hollow body and the latter is heated in a salt or metal bath.—F. C. Th.

*Blast-furnace stoves.* T. G. Wrightson, J. M. Ringquist, and Head, Wrightson and Co., Ltd., Thornaby-on-Tees. Eng. Pat. 118,203. (Appl. Nos. 17,368, Nov. 24, 1917, and 8586, May 23, 1918.)

A BLAST-FURNACE stove is provided with an outer annular combustion chamber, the brickwork at the bottom being of undulatory form, whereby dust-collecting spaces are formed. The gaseous fuel and hot blast mains extend around the combustion chamber and have a number of connections into it.

—C. A. K.

*Open-hearth furnaces; Cooling of interior of —.* W. Reichpietsch, Bochum. Ger. Pat. 305,757, Mar. 17, 1914.

THE roof and arches are cooled by air blown in through nozzles above the gas port. The air channels are placed between the top and lower layers of the roof.—F. C. Th.

*Crucible melting furnaces.* L. C. Harvey, and The Morgan Crucible Co., Ltd., Battersea. Eng. Pat. 118,346, Sep. 8, 1917. (Appl. No. 12,912 of 1917.)

THE inner cover of a crucible melting furnace, of the type having both outer and inner covers, is made in sections which slide on the top of the inner lining to allow of ready removal from above the furnace chamber. The furnace base is hinged, and has a ribbed dish upon its upper surface to facilitate the removal of spilled metal.—C. A. K.

*Are soldering; Electrodes for —.* A. B. Pescatore, London. Eng. Pat. 118,296, May 21, 1917. (Appl. No. 7329 of 1917.)

AN electrode is made of varying cross-section so that on fusion of the electrode the arc travels around its circumference and causes an even fusion of metal. The slag-forming coating also adheres better during the handling of the electrode. Such an electrode may be formed by wrapping an iron or steel wire spirally around a steel rod, by twisting two wires round one another, by twisting a single rod of polygonal section, or by cutting a screw thread on a round electrode. The electrode so made may be coated by dipping it in an agglutinant and spraying with a suitable material in the form of powder.—C. A. K.

*Electrode; Spark-plug — and alloy therefor.* W. B. Driver, East Orange, N.J. U.S. Pat. 1,274,395, Aug. 6, 1918. Date of appl., May 15, 1918.

THE electrodes in a spark-plug are composed of an alloy of nickel, manganese, and chromium, substantially in the proportions; Mn 2%, Cr 3½%, Ni (or Ni and Co) 94½%.—C. A. K.

*Platinumising metal surfaces; Process of —.* B. B. Crombie, Port Chester, N.Y. U.S. Pat. 1,274,995, Aug. 6, 1918. Date of appl., Mar. 29, 1918.

THE surface of the metal to be plated is cleansed by mechanical and chemical means and a paste consisting of 1 part of ammonium platinochloride, 8 of potassium tartrate, and 3 parts of distilled water is applied by means of a friction pad until sufficient platinum has been deposited. The plated article is then treated electrolytically in a bath consisting of 12 parts of platinum chloride, 4 of ammonium platinochloride, and 84 parts of water, using the article as cathode and pure platinum foil as anode.—L. A. C.

*Zinc; Process for utilising the residues obtained in the distillation of* — R. Köhler and O. Ballin, Lippe, Germany. U.S. Pat. 1,275,045, Aug. 6, 1918. Date of appl., Jan. 3, 1914.

A NEW charge of zinc ore is placed above the glowing residue, containing a portion of the flux, of a previous charge. Metallic vapours are driven from the residue by the passage of a strong current of air, and are partly condensed by the new charge, which is afterwards heated and distilled, the blast having been shut off.—C. A. K.

*Powdered ores, flue dust, etc.; Furnace for drying, calcining, and sintering* — W. Schumacher, Berlin. Ger. Pat. 306,032, Feb. 9, 1916.

A VERTICAL furnace is fitted with double walls, the inner wall being so arranged, i.e., constructed of superposed steeply inclined air-cooled or water-cooled hollow iron plates, that gases may pass through, but the material, which is fed in at the top, is prevented from passing out of the central shaft. Hot gas, e.g. air, is led into the space between the walls at the bottom of the furnace and, by means of partitions, is caused to flow three times through the central shaft before it leaves the furnace at the top. Two or more furnaces may be arranged in series.—L. A. C.

*Briquettes [of metals or mineral "fines"]; Machines for manufacture of* — General Briquetting Co., New York, Assignees of T. Gilmore, jun., Brooklyn, N.Y., U.S.A. Eng. Pat., 113,279, Feb. 5, 1918. (Appl. No. 2082 of 1918.) Under Int. Conv., Feb. 6, 1917.

*Metal-sheet coating apparatus for use in plant for plating such sheets. Means for cooling metal sheets discharged from the furnaces of plating plant. Furnaces for heating metal sheets.* The Thermic Plating Process Co., Inc., Assignees of W. E. Watkins, New York. Eng. Pats. 116,292, 116,294, and 116,295, June 11, 1917. (Appl. Nos. 9069, 9071, and 9072 of 1918.) Under Int. Conv., June 10, 1916.

*Rabble-arms for use in mechanical roasting furnaces.* Huntington, Heberlein, and Co., Ltd., and H. C. Bingham, London. Eng. Pat. 118,564, Mar. 13, 1918. (Appl. No. 4427 of 1918.)

*Ore-flotation process.* H. C. Colburn, Colorado Springs, and E. A. Colburn, Denver, Colo. Re-issue 14,497, July 30, 1918, of U.S. Pat. 1,226,062, May 15, 1917. Date of appl., Jan. 19, 1918.

SEE this J., 1917, 721.

*Separation of mixed metallic sulphides.* L. Bradford, Broken Hill, N.S.W. U.S. Pat. 1,274,505, Aug. 6, 1918. Date of appl., Oct. 22, 1914.

SEE Eng. Pat. 21,880 of 1914; this J., 1915, 1256.

*Alloy.* P. R. Kuehnrich, Sheffield. U.S. Pat. 1,273,877, July 30, 1918. Date of appl., June 17, 1916.

SEE Eng. Pat. 14,939 of 1915; this J., 1916, 1117.

*Oxidising apparatus.* U.S. Pat. 1,272,009. See I.

*Oxidation of tin. Production of zinc oxide.* Eng. Pats. 118,664 and 118,665. See VII.

## XI.—ELECTRO-CHEMISTRY.

*New method for the determination of [electrical] conductivity.* Newbery. See XXIII.

### PATENTS.

*Electro-osmotic process of separating solid constituents from an emulsion.* Ger. Pat. 305,217. See I.

*Electrodes for arc soldering.* Eng. Pat. 118,296. See X.

*Treatment of magnetic material [iron and iron alloys] for use in static transformers and other electrical apparatus.* Eng. Pat. 118,425. See X.

*Spark-plug electrode and alloy therefor.* U.S. Pat. 1,274,395. See X.

## XII.—FATS; OILS; WAXES.

*Oils; Optical dispersion of — from an analytical point of view.* P. J. Fryer and F. E. Weston. Analyst, 1918, 43, 311–317.

The optical dispersion of oils was determined by means of a Zeiss-Pulfrich refractometer, using a hydrogen tube with a pressure of about 2 mm., the red and blue rays from which corresponded with the C and F lines of the spectrum. The readings were made at 40° C. (except in the case of Japan wax and spermaceti, 56° C.), and the refractive power calculated according to the formula:—

$$\omega = \frac{n_D - n_C}{n_D - 1}$$
 It was found that nearly all the oils and fats examined gave very similar dispersions (0.0186–0.0207), with the exception of coconut oil (0.0167), linseed oil (0.0218), and tung oil (0.0371). Palm-kernel oil and butter fat also gave comparatively low figures (0.0180 and 0.0182 respectively), and the results indicate that the dispersion is lowered by the presence of glycerides of low molecular weight, and increased by that of glycerides of highly unsaturated fatty acids. The difference between the dispersion of coconut oil and palm-kernel oil is comparatively greater than the difference between the refractive indices. The dispersive power is reduced by about 0.00002 for each increase of 1° C. Free acidity in the oil has but little influence on the optical dispersion. Linseed oil exposed in a thin film for 28 hours showed an increase in dispersive power from 0.0218 to 0.0223, whilst niger seed oil exposed for 50 hours showed an increase from 0.0198 to 0.0204. Heat-polymerisation of these two oils caused a large increase in the refractive index and a notable decrease in the dispersive power, especially in the case of linseed oil. In the case of tung oil, however, there was pronounced progressive reduction both of the refractive index and of the dispersion on heating. Hence it would appear that the thickening and solidification of tung oil on heating must be due to some factor other than polymerisation. In the case of hydrocarbon oils little variation was observed in the dispersions of petroleum distillates of lower b.pt. (petroleum naphtha, paraffin oil, solar oil, and American "mineral sperm"), but a higher fraction (American "engine oil") showed a pronounced increase. Members of the benzene series show relatively high dispersions, which are reduced by the addition of the side-chain in toluene and xylene, whilst turpentine closely resembles fatty oils both in refractive index and dispersive power.—C. A. M.



*Fish oils.* W. Fahrion. Chem. Umschau, 1917, 57. Chem.-Zeit., 1918, 42, Rép., 136.

THE statement by the author, made nearly twenty-five years ago (this J., 1893, 938), that fish oils contained jecoric acid,  $C_{18}H_{30}O_2$ , an isomer of linolenic acid, has been confirmed by the examination of sardine oil. Besides the more unsaturated acid, clupanodonic acid,  $C_{18}H_{28}O_2$ , discovered by Tsujimoto (this J., 1906, 818), this oil contains jecoric acid and probably also a less unsaturated fatty acid. The occurrence in natural fats of fatty acids containing an odd number of carbon atoms is still open to doubt, but later investigations make it appear possible that asellinic acid,  $C_{17}H_{32}O_2$ , isolated by the author from liver oils, is an individual acid and not a mixture of stearic and palmitic acids.—W. P. S.

*Opium wax.* J. N. Rakshit. Analyst, 1918, 43, 321—322.

Ox extracting 200 grms. of dry powdered opium with successive portions of cold petroleum spirit, shaking the united extracts with 5 successive portions of 4% hydrochloric acid until the aqueous extract ceased to give a precipitate with Mayer's reagent, and neutralising, drying, filtering, and evaporating the petroleum spirit extract, 14.35 grms. of a brown sticky mass was obtained. This opium wax had the following characters:—Iodine value, 152.5; Reichert value, 2.0; saponif. value, 114.5; and unsaponifiable matter, 28.7%, with iodine value, 138.5, and m.pt. about  $86^{\circ}C$ . Opium wax is insoluble in water, and nearly insoluble in methyl alcohol, ethyl alcohol, and glacial acetic acid, sparingly soluble in acetone and amyl alcohol, but soluble in carbon tetrachloride, chloroform, petroleum spirit, turpentine, and ether, and readily soluble in carbon bisulphide, benzene, and toluene. Nitric acid makes it slightly browner, sulphuric acid dark brown, and a mixture of hydrochloric and nitric acids, pale yellow.—C. A. M.

*Soap; Distillation method for the determination of water in —.* R. Hart. J. Ind. Eng. Chem., 1918, 10, 598—599.

A QUANTITY of the soap sufficient to yield about 3 c.c. of water is distilled with the addition of its own weight of "red oil" or oleic acid and 150 c.c. of water-saturated xylene; 85 c.c. of distillate is collected in a receiver already containing 5 c.c. of water-saturated xylene. The condenser is washed out with the xylene and the washing added to the receiver; the latter consists of a cylinder holding about 120 c.c. and constricted at the bottom to form a tube about 4 cm. long and graduated in tenths of a c.c. The volume of the water collected is read off after 30 mins.; a slight turbidity usually noticed in the xylene layer does not affect the results obtained.—W. P. S.

*Device for determining the dropping point (melting point) of fats, waxes, paraffin wax, etc., and especially bitumens.* Dupré. See IIa.

*Occurrence of carotin in oils and vegetables.* Gill. See XIXa.

#### PATENTS.

*Oils, etc.; Steaming —.* A. Lowenstein, Chicago, Ill. U.S. Pat. 1,274,175, July 30, 1918. Date of appl., Aug. 28, 1916.

OILS, fats, waxes, fatty acids, and like products are subjected to the action of steam superheated at a low pressure (not exceeding 6 lb. per sq. in.) to a temperature above  $500^{\circ}F$ . ( $260^{\circ}C$ ). The process may be used for distilling fatty acids and for deodorising glycerides.—A. B. S.

*Decoloriser [for fatty oils].* P. A. Boeck, New York, Assignor to Celite Products Co., Los Angeles, Cal. U.S. Pat. 1,272,197, July 9, 1918. Date of appl., June 14, 1917.

A DECOLORISING agent, especially suitable for use in refining fatty oils, is composed of a mixture of calcined kieselguhr and fullers' earth, preferably in equal proportions. The kieselguhr, in addition to its own decolorising action, increases that of the fullers' earth, in that the effective surface of the latter is increased. Moreover, in admixture with kieselguhr, finer grades of fullers' earth can be used than hitherto, without danger of clogging the filter.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Cobalt [in varnishes, paints, etc.]; Colorimetric determination of —.* E. G. Jones. Analyst, 1918, 43, 317—319.

SMALL quantities of cobalt (0.01 to 0.1%) in varnishes, zinc oxide paints, etc., may be rapidly determined colorimetrically by means of a solution of  $\alpha$ -nitroso- $\beta$ -naphthol as used by Atack (this J., 1915, 641), the effect of moderate quantities of many other metals being eliminated by the addition of ammonium citrate solution (500 grms. in 250 c.c. of water plus 500 c.c. of ammonia solution of sp. gr. 0.880). In the case of varnishes the ash from a weighed quantity of the sample is treated with strong hydrochloric acid, followed by *aqua regia*, and later by hydrochloric acid to remove the nitric acid. The solution is evaporated to dryness, the residue dissolved in hot water with the addition of a few drops of hydrochloric acid, and the solution made up to definite volume. An aliquot portion is treated with 5 c.c. of the ammonium citrate solution, the mixture diluted nearly to 100 c.c., 5 c.c. of the reagent added, and the colour compared with that given by different quantities of a standard cobalt solution mixed with the same amounts of ammonium citrate and  $\alpha$ -nitroso- $\beta$ -naphthol. Copper when present in more than a trace should be removed by means of hydrogen sulphide, and nickel separated by dimethylglyoxime, the excess of oxime being subsequently destroyed. Manganese when present in much greater quantity than the cobalt may be removed to a sufficient extent by boiling with sodium bismuthate, and the cobalt estimated in the filtrate.—C. A. M.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Vulcanisation researches.* B. J. Eaton. Agric. Bull. Fed. Malay States, 1918, 6, 315—316.

AS has already been observed for crêpe rubber, the use of even five times the minimum quantity of acetic acid for coagulation has very little effect in retarding the rate of vulcanisation of matured "slab" rubber; the use of sodium bisulphite in the preparation of rubber also causes a comparable slight retardation in the rate of vulcanisation of slab and crêpe rubbers; the addition of sodium bisulphite is ineffectual in the production of pale-coloured slab rubbers by the present method, as darkening occurs on allowing the coagulum to mature in the open. During the washing and crêping of slab rubber little or none of the natural accelerators of vulcanisation is lost, rubber which had been "wormed" without washing vulcanising even a little more slowly, possibly on account of its invariable tendency to "spot disease." The

use of alum as a coagulant on certain small holdings is regarded with disfavour, the period of optimum vulcanisation being found to range from 1½ to 4½ hours in a number of samples of sheet rubber thus produced.—D. F. T.

#### PATENTS.

*India-rubber and the like; Treatment of —.* W. J. Mellers-Jackson, London. From Morgan and Wright, Detroit, Mich., U.S.A. Eng. Pat. 118,305, Aug. 9, 1917. (Appl. No. 11,473 of 1917.)

THE extent to which rubber expands during vulcanisation is indicative of the degree of vulcanisation attained and the most efficient vulcanisation is effected at the point when the expansion has reached a maximum. An apparatus is described with which it is possible to observe the expansion corresponding with various periods of vulcanisation of a cylindrical test-piece of a rubber mixing. A chart is constructed for each mixing giving the expansion for various periods of vulcanisation and steam pressures, and with the aid of this it is possible to pre-determine the extent of expansion which the mixing will show at the desired degree of vulcanisation. The method is particularly applicable to articles vulcanised in moulds which, for this purpose, must be provided with a gauge to record the expansion.—D. F. T.

*Rubber or other materials or articles; Apparatus for the drying or heat treatment of —.* F. Moore, Birmingham. Eng. Pat. 118,394, Mar. 19, 1918. (Appl. No. 4801 of 1918.)

ARTICLES of rubber or other material are attached to a circular rotating framework, a segment of which is covered by the oven, the remainder acting as a cooling area. The tray is rotated intermittently, the intervals allowing for the removal of articles after treatment and for the attachment of other articles; the period of complete rotation should suffice for a single heat treatment. The number of rotations is recorded by a counting device.—D. F. T.

*Latex; Process and apparatus for treating —.* E. M. Slocum, Medan, Sumatra, Assignor to General Rubber Co. U.S. Pat. 1,268,638, June 4, 1918. Date of appl., Feb. 23, 1917.

LATEX is coagulated in a chamber provided with a spiral partition, so that the coagulum is obtained in a spiral strip instead of a compact slab.—D. F. T.

*Latex; Process for treating — and products obtained thereby.* E. M. Slocum, Medan, Sumatra, Assignor to General Rubber Co. U.S. Pat. 1,268,639, June 4, 1918. Date of appl., Feb. 23, 1917.

IN the preparation of rubber from latex, the coagulation process is assisted by the addition of the enzyme coagulase.—D. F. T.

*Latex; Process of coagulating — and product thereof.* E. M. Slocum, Medan, Sumatra, Assignor to General Rubber Co. U.S. Pat. 1,270,887, July 2, 1918. Date of appl., Feb. 23, 1917.

RUBBER is obtained from latex by the addition of a metallic compound adapted to form an insoluble material in the latex whilst substantially free from any tendency to accelerate coagulation.—D. F. T.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Glues; Determining the comparative melting points of — as a measure of the jelly strength.* C. F. Sammet. J. Ind. Eng. Chem., 1918, 10, 595.

THE dried glues are ground and 1-grm. portions of the samples which pass a 20-mesh sieve but are retained by a 40-mesh sieve are placed in small beakers with 10 c.c. of water at 10° C. and soaked for 1 min. A small quantity of each is then transferred to a smooth brass plate, 6 in. by 1½ in., and  $\frac{1}{16}$  in. thick; the glue particles are placed about 3 in. from the end of the plate and a portion of each, about the area of five pin heads, is separated by means of a spatula and pushed to within 2 in. of the end. The plate is then dipped to a depth of 0.5 in. in water at 40° C. The poorer grades of glue melt and slide rapidly down the plate whilst the better grades melt some time before a sliding effect occurs. If desired, a small disc of copper foil may be pressed on each heap; the copper adds weight and slides quickly at the first indication of the jelly melting. Comparison with standard glues shows that the melting point may be taken as a measure of the jelly strength.—W. P. S.

#### XVI.—SOILS; FERTILISERS.

*Soil extracts and physiological solutions; Determination of ammonia and nitric nitrogen in —.* B. S. Davisson. J. Ind. Eng. Chem., 1918, 10, 600—605.

AMMONIA is determined by the aeration method, 250 c.c. of the extract being treated with 2 grms. of sodium oxalate, 10 grms. of sodium carbonate, and a few drops of oil, and the aeration continued for 3 hrs. at the rate of 1080 litres of air per hr. The residual solution is then treated with 5 c.c. of concentrated sulphuric acid, boiled, 5 c.c. of Stutzer's cupric hydroxide reagent is added, the mixture filtered, the filtrate boiled for 30 mins. with the addition of sodium hydroxide, and the nitrate then reduced with Devarda's alloy. The resulting ammonia is determined by distillation. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Water hyacinth as a source of potash.* F. W. F. Day. Agric. Bull. Fed. Malay States, 1918, 6, 309—314.

EXAMINATION of the ash of the water hyacinth (*Eichornia crassipes*), a local weed of rapid growth, shows that this constitutes approximately 10—15% of the sun-dried plant and consists essentially of potash (25% expressed as K<sub>2</sub>O or 40% as KCl) together with sodium and magnesium chloride, sulphate, and carbonate and a little phosphate; the ash of the stalks is richer than that of the other parts in soluble potassium salts. The variable composition of the plant with respect to potash content as reported earlier (Finlow and Maclean, this J., 1918, 253 A), is largely due to fine particles of silt, etc., incorporated in the roots or adhering to the plant; the greater the ash the smaller its percentage of potash. The plant should be of value not only as a nitrogenous, potassic, and phosphatic fertiliser, but also as a source of potash for other purposes.—D. F. T.

*Phosphates [in fertilisers]; Gravimetric determination of —.* W. R. Mummery. Analyst, 1918, 43, 324.

THE following modification of Ullmann's method of determining phosphates is recommended, especially for the analysis of guano and fertilisers.



After the digestion, the nitric acid solution should be filtered prior to being made up to standard volume. Phosphomolybdates are precipitated at 60° C. and the mixture allowed to stand 30 mins. at 60° C. before filtering. Magnesium ammonium phosphate is precipitated at 80° C. and allowed to stand for 3 hours at the ordinary temperature. It is essential to use precipitating and washing reagents of the prescribed strength. (See also J. Chem. Soc., Nov., 1918.)—C. A. M.

*Sugar cane; Fertiliser experiments with —.*  
J. Mirasol y Jison. Philippine J. Sci., 1918, A, 13, 135–143.

On planting Los Baños white cane in a clay loam soil, it was found on treating 15 plots, each of 450 sq. metres area, with different fertilisers and combinations of them that the highest purity of the juice was obtained with potassium sulphate alone and with a complete fertiliser containing potassium sulphate, sodium nitrate, and double superphosphate; whereas the purity of the juice was lowered to a considerable extent with double superphosphate in combination with nitrogen either in the form of ammonium sulphate or sodium nitrate, the effect on the percentage of sucrose in the juice running parallel with that on the purity of the juice. Ammonium sulphate in combination with potassium sulphate or with double superphosphate produced the highest yield of cane, dried blood and potassium sulphate apparently having the contrary effect, though an increased yield of cane was shown not to result in a greater nett amount of 96° sugar. Tests carried out with complete fertilisers indicated sodium nitrate to be superior to ammonium sulphate as a source of nitrogen for the cane, and it is concluded that under the conditions of the tests the highest financial returns would be obtained with a complete fertiliser containing potassium sulphate, sodium nitrate, and double superphosphate (the nitrogen, potash, and phosphoric acid being in the ratio 8 : 6 : 8).—J. P. O.

## XVII.—SUGARS; STARCHES; GUMS.

*Oxidases; Presence and functions of — in the sugar cane.* R. Narain. Agric. J. India (Special Science Congress Number), 1918, 47–64.

EXTRACTS of the oxidases present in the various parts of the sugar cane were prepared by crushing the sliced parts in a mortar with sterilised sand and filtering the expressed liquids through a Buchner funnel or a porcelain filter candle. The extracts retained their activity for several months in the presence of chloroform (5% by vol.), toluene and ether both having an inferior preservative effect. Oxidases were found to be more active in the upper than in the lower part of the stem of the cane; in the rind than in the pith; in the pith from the upper than in that from the lower parts; in the nodes than in the internodes; and in the open flat portion of the leaf than in the sheath or curled-up part or in the stem of the leaf. They were extremely sensitive to the reaction of the medium, the activity (as indicated by the guaiacum test) diminishing as the acidity of the extract increased; the presence of acid in quantity such as is normally present in the plant is not, however, harmful to sugar-cane oxidase. It is possible that the activity of the oxidases in the growing plant may be controlled by variations in the acidity of the sap, since it was found both in unripe and in fully developed canes that the acidity is distinctly lower in the afternoon when the sun is strongest and other photosynthetic processes are in progress than

in the earlier part of the day. Whereas Moore and Whitley (Biochem. J., 1909, 4, 136) found the activity of oxidases to be completely destroyed by the action of reducing agents, the author found that the activity of cane oxidases is inhibited only for a time by hydrogen sulphide, there being a subsequent recovery, which is more rapid in the presence of chloroform. Similarly, on boiling cane oxidase extracts their activity, though at first apparently destroyed, is actually only suspended temporarily, and gradually returns almost to its original value. Even after boiling twice, there is a recovery. Further, it was possible to obtain the guaiacum reaction from extracts of cane leaves which had been dried at 100° C. during 6 hours, while even the ash of these leaves gave a faint but distinct indication with the same reagent. It is considered that it is impossible longer to regard oxidases (or at least those present in the cane) as enzymes, as has hitherto been done, since they are not destroyed by heat, and experiments indicate that they are not capable of acting upon a practically indefinite amount of substrate.—J. P. O.

*Lavulose; Determination of — in the presence of aldoses.* Herzfeld and Lenart. Zentr. Zuckerind., 1918, 68, 227. Chem.-Zeit., 1918, 42, Rep., 135.

A QUANTITY of the substance containing about 1 grm. of levulose is dissolved in water, the solution clarified with lead acetate, diluted to 100 c.c., and filtered; 50 c.c. of the filtrate is then inverted (Clerget-Herzfeld method), cooled, and treated with bromine, 1 c.c. of the latter being added for each grm. of aldose present. The mixture is shaken occasionally for 24 hrs., whereby the aldoses, and these only, are oxidised. The excess of bromine is evaporated on the water-bath, the mixture neutralised, then treated with a few drops of acetic acid, diluted to 100 c.c., and the lavulose determined from the reducing power of the solution, using the Hönig-Jesser table.—W. P. S.

*Fertiliser experiments with sugar cane.* Mirasol y Jison. See XVI.

### PATENT.

*Vegetable glue; Process of manufacturing — [from starchy materials].* V. G. Bloede, Catonsville, Md. U.S. Pat. 1,273,571, July 23, 1918. Date of appl., Dec. 17, 1917.

AMYLACEOUS matter is heated with water containing a neutral salt to or near the boiling point until a smooth and homogeneous paste is formed. The salt must be one having the property of increasing the solubility of the starch granules, but must not prevent or materially retard the action of "starch-reducing" enzymes or bacilli. The paste is cooled and is then acted upon by "starch-reducing" enzymes or bacilli until of the desired consistency. The vegetable glue so obtained is evaporated at a low temperature to solid form.—T. St.

## XVIII.—FERMENTATION INDUSTRIES.

*Enzymes; Chemical composition and formation of —.* XV. Further experiments with *B. acidilactis* (*Streptococcus lactis*). II. Euler and O. Svanberg. Z. physiol. Chem., 1918, 102, 176–184.

THE evolution of carbon dioxide during lactic acid fermentation by *Streptococcus lactis* (see Euler and Griese, this J., 1917, 1283) is not confirmed. The fermentation of lactose by *Streptococcus lactis* is

inhibited by sodium phosphate, whereas the action of *B. casei*, E., was found to be accelerated. Sodium lactate also exerts an inhibiting action on lactic acid fermentation.—H. W. B.

*Refractometer; Use of the dipping* — [in the examination of alcoholic liquids]. W. W. Randall. J. Ind. Eng. Chem., 1918, 10, 629—630.

In determining the alcoholic strength of a solution by means of the immersion refractometer, low readings are obtained when the usual metal cup is used for holding the solution. This appears to be due to the conduction of heat from the atmosphere to the contents of the cup through the metal parts of the instrument after the cup and its contents have been brought to the proper temperature for the observation. Alteration in temperature of the liquid does not take place when small glass beakers are used in place of the metal cups. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Action of ozone on artificial culture media and on various bacteria, yeasts, and moulds.* Heise. See XIXb.

#### PATENTS.

*Beer; Manufacture of non-alcoholic malt* — L. Block, Manaroneck, N.Y. U.S. Pat. 1,271,269, July 2, 1918. Date of appl., July 10, 1915.

In the production of a non-alcoholic malt beverage, the alcohol is removed, under the influence of a vacuum, at the temperature of, and immediately after the main fermentation. The liquid is then cooled, subjected to a secondary or aging fermentation (kriusening), carbonated, and filtered.

*Cooling plates for continuous rectification apparatus.* Eng. Pat. 110,910. See I.

### XIXa.—FOODS.

*Cream; Pasteurisation of sour, farm-skimmed* — for butter making. O. F. Hunziker, Spitzer, H. C. Mills, and H. B. Switzer. Purdue Univ. Agric. Exper. Stat., Bull. No. 208, 1917, 20, 1—76. Bull. Agric. Intell., 1918, 9, 695—897.

PASTEURISATION at 145° F. (63° C.) for 20 mins. was more effective than rapid (flash) heating at 165° F. or 185° F. (74° C. or 85° C.); it improved the quality of the butter, even of that made from inferior cream, and did not materially alter the composition of the fat. During storage the lactose decreased and the acidity increased, but not proportionately, whilst there was always a certain amount of decomposition of proteins, the changes being least in butter made from pasteurised cream.

—W. P. S.

*Butter fat; Method for the detection of foreign fats in* — A. Seidenberg. J. Ind. Eng. Chem., 1918, 10, 617—621.

A METHOD described previously by the author (this J., 1917, 1128) for fractionating fats and oils may be adapted to the detection of foreign fats in butter fat. Ten grms. of the sample is dissolved in a 150 c.c. graduated cylinder (27 cm. high and 3.1 cm. diam.) in a mixture of ether, 90, and absolute alcohol, 10 parts by vol., sufficient of the solvent being added to make the total volume 96 c.c. The cylinder is closed by a rubber stopper through which pass a thermometer and two tubes, one short and the other reaching nearly to the bottom of the cylinder. By raising or lowering the thermometer, the surface of the liquid is brought between the 100 and 102 c.c. marks, and air, previously passed through absolute alcohol, is drawn through the

solution at such a rate that it is reduced to about the 60 c.c. mark in not more than 12 or less than 8 mins. The temperature is maintained between 10° and 15° C. by means of a bath of warm water. The volume of the solution is noted at the point where a distinct turbidity appears, the aspiration is stopped, the solution filtered rapidly, and the precipitated glycerides weighed. With pure butter fat the turbidity is produced when the volume is between 44 and 68 c.c. (of 100 samples examined 94 gave a turbidity at a volume between 50 and 60 c.c.). The maximum insoluble residue found with pure butter fat was 0.449 gm. If the volume of the solution at the turbidity point is more than 68 c.c., the presence of tallow, lard, or hydrogenated fat is indicated; when the volume is lower than 44 c.c., coconut stearine is probably present.

—W. P. S.

*Carotin in oils and vegetables; Occurrence of* — A. H. Gill. J. Ind. Eng. Chem., 1918, 10, 612—614.

It was shown previously that the peculiar bluish-green reaction of palm oil with acetic anhydride containing a trace of sulphuric acid was due to carotin (this J., 1917, 336). Carotin has now been found to be present in maize, pumpkin, orange peel, flax seed, mustard seed, black sesame seed, butter fat, tallow, grass, and carrots, but not in rape seed, white sunflower seed, turnip, safflower, or turmeric.

—W. P. S.

*Eggs; Determination of loosely bound nitrogen in* — as ammonia. N. Hendrickson and G. C. Swan. J. Ind. Eng. Chem., 1918, 10, 614—617.

TWENTY-FIVE grms. of the sample is mixed in a cylinder with 100 c.c. of water, 75 c.c. of alcohol, 1 gm. of sodium fluoride, 2 c.c. of 50% sodium carbonate or potassium carbonate solution, and 1 c.c. of kerosene, and air is blown through the mixture for 4 hrs.; the air containing the ammonia is passed through absorption vessels containing standard acid, and the ammonia is determined volumetrically or colorimetrically. Frozen eggs yield about 0.003% of nitrogen as ammonia, but the quantity increases with incipient decomposition. Magnesium oxide or sodium hydroxide cannot be used in place of potassium or sodium carbonate in the method.—W. P. S.

*Action of ozone on artificial culture media and on various bacteria, yeasts, and moulds.* Heise. See XIXb.

*Method for the separation and quantitative estimation of the lower alkylamines in presence of ammonia.* Weber and Wilson. See XXIII.

*Adamkiewicz reaction [of proteins] and transformation of glyoxylic acid into formaldehyde.* Voisenet. See XXIII.

#### PATENTS.

*Temperature-changing apparatus [for pasteurising milk].* W. J. Davis, Chicago, Ill. U.S. Pat. 1,272,232, July 9, 1918. Date of appl., July 23, 1917.

SEVERAL tubes of relatively large diameter are connected together at alternate ends to form a tortuous passage for the heating fluid, and within each is a number of smaller tubes for the liquid, e.g. milk, to be heated. Curved guide plates are provided at the ends of the groups of smaller tubes to ensure uniform distribution of the liquid among the tubes.



*Evaporated vegetable products and method of preparing the same.* H. G. C. Fairweather, London. From Pacific Evaporator Co., Portland, Oreg., U.S.A. Eng. Pat. 118,466, Sep. 5, 1917. (Appl. No. 12,732 of 1917.)

SEE U.S. Pats. 1,259,631, 1,259,632, and 1,259,635 of 1918; this J., 1918, 320 A.

*Evaporated fruit and method of preparing the same.* H. G. C. Fairweather, London. From Pacific Evaporator Co., Portland, Oreg., U.S.A. Eng. Pat. 118,673, Sep. 5, 1917. (Appl. No. 12,733 of 1917.)

SEE U.S. Pats. 1,259,633 and 1,259,637 of 1918; this J., 1918, 320 A.

*Evaporated apples and method of preparing the same.* H. G. C. Fairweather, London. From Pacific Evaporator Co., Portland, Oreg., U.S.A. Eng. Pat. 118,674, Sep. 5, 1917. (Appl. No. 12,734 of 1917.)

SEE U.S. Pats. 1,259,634 and 1,259,636 of 1918; this J., 1918, 320 A.

*Antiseptic or sterilising agent for use in the preservative treatment of food.* A. E. Sherman, London. U.S. Pat. 1,274,793, Aug. 6, 1918. Date of appl., Mar. 7, 1918.

SEE Eng. Pat. 112,473 of 1916; this J., 1918, 135 A.

## XIXB.—WATER PURIFICATION; SANITATION.

*Ozone; Action of — on artificial culture media and on various bacteria, yeasts, and moulds.* R. Heise. Arb. Kaiserl. Gesundheits.-Amt, 1917, 50, 418. Chem.-Zeit., 1918, 42, Rep., 137.

AN ozone concentration of 3 mgrms. per cub. metre of air was sufficient to kill in 3–4 hrs. more than 95% of the bacteria present as individuals on the surface of a culture medium. The ozone penetrates with difficulty into the medium and into masses of bacteria, consequently colonies were hardly affected. Low temperature favoured the action of the ozone. Similar results were obtained with yeasts, which proved to be slightly more sensitive. Both the spores and the mycelia of moulds growing on the surface of a medium were killed, but any mycelium below the surface was not killed. From these experiments it is concluded that only a partial destruction of organisms present on the flesh of animals preserved in cold storage can be expected by the use of ozonised air, but sufficient to improve the keeping qualities of the meat.—J. H. J.

### PATENTS.

*Distilling and sterilising liquids, especially water; Apparatus for —.* M. C. Rimmer, London. Eng. Pat. 118,482, Sept. 18, 1917. (Appl. No. 13,413 of 1917.)

THE apparatus consists of a spherical boiler with a neck and a side tube at the water level. The neck projects through the base of a dome-shaped condenser. Part of the cooling water leaving the condenser passes through a tube with a regulating cock and drips into a cup at the end of the side tube of the boiler. The water condensed inside the dome collects on the base of the dome and runs away by a pipe in the base. The boiler is supported on a sand bath and is heated by a Bunsen burner or other means. The boiler and condenser are made of earthenware, glass, or other non-metallic material.—J. H. J.

*Filters; Method of operating and cleansing —.* K. Morawe, Berlin-Friedenau. Ger. Pat. 304,658, Sep. 9, 1916.

A WIND-BOX is provided on the clean water side of the filter. During filtration uniform pressure is maintained over the whole surface of the filter. To cleanse the filter, water under pressure is forced by means of the compressed air in the wind-box upwards through the filtering material so as to agitate it and free it from sludge. Compressed air from an outside source may be introduced to aid in the cleansing of the filter. The wash water from the upper part of the filter flows downwards through a central pipe, provided with a jet device, back to the wind-box, and is thus circulated repeatedly through the filtering material, so that only a comparatively small quantity of wash water is required.

*Cresols and mixtures of cresols; Solid preparations [disinfectants] containing — and the manufacture thereof by means of salts of fatty acids.* C. A. Cofman-Nicoresti, Snaresbrook. Eng. Pat. 118,667, Sep. 3, 1917. (Appl. No. 12,621 of 1917.)

A DISINFECTANT in the form of solid tablets is prepared by heating a mixture of crude or refined cresol with salts of fatty acids (e.g. sodium stearate or animal soap) with or without pressure. Formalin and suitable colours and perfumes may be added if desired. The tablets are dissolved in water or alcohol as required.—L. A. C.

*Water; Process of softening —.* H. Heller, Assignor to Oelwerke Stern Sonneborn A.-G., Cologne, Germany. U.S. Pat. 1,273,857, July 30, 1918. Date of appl., Nov. 13, 1914.

SEE Eng. Pat. 22,362 of 1914; this J., 1915, 977.

*Water; Process of purifying —.* V. Kobelt, Berlin. U.S. Pat. 1,274,560, Aug. 6, 1918. Date of appl., Aug. 14, 1912.

SEE Fr. Pat. 440,351 of 1912; this J., 1912, 948.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Morphine; Polarimetric determination of — in opium.* J. N. Rakshit. Analyst, 1918, 43, 320–321.

SIXTEEN grms. of dried opium powder is triturated with 4 grms. of slaked lime until thoroughly mixed, 50 c.c. of water freshly saturated with ether is added, and, after complete admixture, 110 c.c. of the same water is added, and the whole mixed for 15 mins. It is then filtered through a covered filter, and 100 c.c. of the filtrate shaken in a separating funnel for at least 3 mins. with 100 c.c. of ether saturated with water. The separated aqueous layer is shaken in another funnel with an equal volume of the saturated ether, and this process of extraction again repeated. Finally 51 c.c. of the separated aqueous liquid is placed in a 250 c.c. flask with 1.5 c.c. of strong hydrochloric acid, shaken with 10 grms. of well-washed dried animal charcoal for 10 mins., and filtered. The filtrate is examined in the polarimeter, and the amount of morphine calculated, taking the specific rotation of anhydrous morphine in dilute hydrochloric acid to be  $-127^{\circ}$  at  $25^{\circ}$  C. with white light. The result is multiplied by 1.05 as a correction for the increase in volume due to the addition of acid, and 51 c.c. instead of 50 c.c. taken as representing

5 grms. of opium. The use of water saturated with ether and ether saturated with water prevents the formation of emulsions in the separation of the calcium morphinate from the other alkaloids.

—C. A. M.

*α-Oxycinchonine.* E. Léger. Bull. Soc. Chim., 1918, 23, 328—335.

*α-OXYCINCHONINE* behaves towards hydrobromic acid as do cinchonine and its isomers, the product being hydrobromocinchonine and a certain amount of the isomers of cinchonine. Similarly with sulphuric acid *α*-oxycinchonine yields the same isomers of cinchonine as does cinchonine itself. It is concluded that the so-called *α*-oxycinchonine is really *α*-oxydihydrocinchonine, the vinyl side chain of cinchonine being replaced by the group  $\text{CH}_2\text{CH}(\text{OH})$ .

—W. G.

*Alkaloids of the Calabar bean. VII. Degradation by successive iodomethylations of the nuclei of eserine and geneserine.* M. and M. Polonovski. Bull. Soc. Chim., 1918, 23, 355—356.

With a view to elucidating further the constitution of eserine and geneserine certain derivatives of this series have been subjected successively to the action of methyl iodide and alkali and a number of new derivatives are described. (For details see J. Chem. Soc., Nov., 1918.)—W. G.

*Alkaloids of the Calabar bean. VIII. Hydrogenation in the eserine, geneserine, and  $\psi$ -geneserine series.* M. Polonovski. Bull. Soc. Chim., 1918, 23, 356—361.

ESERINE and its derivatives when reduced by zinc and hydrochloric acid yield dihydro-derivatives. Geneserine and its derivatives on reduction yield first the corresponding eserine derivatives and then their dihydro-derivatives. (See further J. Chem. Soc., Nov., 1918.)—W. G.

*Theobromine; Determination of* —. W. O. Emery and G. C. Spencer. J. Ind. Eng. Chem., 1918, 10, 604—606.

ABOUT 0.1 grm. of the sample together with an equivalent amount of sodium acetate is dissolved by heating with 2 c.c. of glacial acetic acid, the solution is diluted with 5 c.c. of hot water and transferred to a 100 c.c. flask containing 50 c.c. of N/10 iodine solution. 20 c.c. of saturated sodium chloride solution and 3 c.c. of concentrated hydrochloric acid are added, the mixture filtered after about 15 hrs., and the excess of iodine titrated in an aliquot portion of the filtrate. The quantity of theobromine is calculated from the amount of iodine used to form insoluble theobromine periodide,  $\text{C}_7\text{H}_8\text{N}_4\text{O}_2 \cdot \text{HI} \cdot \text{I}_4$ . The method may be used when sodium salicylate is present. (See also J. Chem. Soc., Nov., 1918.)

—W. P. S.

*Digitalis; Relative activity of separated portions of* —. E. L. Newcomb and C. H. Rogers. Amer. J. Pharm., 1918, 90, 580—588.

SELECTED clean digitalis (compare this J., 1918, 322 A) when tested physiologically on guinea-pigs was found to be twice as active as the arbitrary standard of Reed and Vanderkleed, and about 12% more active than average digitalis when tested by the intravenous method on cats. Relatively the Reed and Vanderkleed standard was much lower than the accepted standard for digitalis by the cat method. The petioles of digitalis were from one fourth to one fifth as active as the entire leaf, and the greatest physiological activity was

shown chiefly in those parts of the leaf where photosynthetic activity was most pronounced. Dirt siftings separated in cleaning the drug had in one instance one fifth to one sixth of the activity of the clean drug, but in the other cases infusions of the dirt siftings produced effects on cats and guinea-pigs not characteristic of digitalis. The hairs of the digitalis leaf contain, at most, a relatively small amount of the glucosidal principles of the drug.

—C. A. M.

*Clitral; Modification of the Hiltner method for determining* —. C. E. Parker and R. S. Hiltner. J. Ind. Eng. Chem., 1918, 10, 608—610.

THE addition of 1% of oxalic acid to the alcoholic *m*-phenylenediamine hydrochloride solution used as the reagent in Hiltner's colorimetric method (this J., 1910, 172) prevents the formation of a green or blue colour given by certain lemon and orange oils and extracts, and the normal yellow coloration due to clital is obtained. (See also J. Chem. Soc., 1918, ii., 377.)—W. P. S.

*Cholesterol; Energetic oxidation of* — by nitric acid. A. Windaus. Z. physiol. Chem., 1918, 102, 160—165.

ACETIC, succinic, methylsuccinic, and *α*-methylglutaric acids and dinitroisopropane are among the products of oxidation of cholesterol by nitric acid. Acetone, hydroxyisobutyric acid, methyl isohexyl ketone, and octane have previously been obtained, and the author points out that all these substances may be regarded as derived from an iso-octyl side-chain in cholesterol.—H. W. B.

*Potassium guaiacolsulphonate; Identification and determination of* —. S. Palkin. J. Ind. Eng. Chem., 1918, 10, 610—612.

To identify potassium guaiacolsulphonate in admixture with gums, resins, alkaloids, etc., a portion of the sample is mixed with dilute hydrochloric acid, filtered, and a portion of the filtrate is tested for sulphate; another portion of the filtrate is heated with the addition of sodium peroxide, and the still acid solution again tested for sulphate. In the absence of sulphate in the first test, subsequent formation of sulphate by oxidation indicates the presence of sulphonate. A red coloration is obtained when a portion of the filtrate is rendered alkaline and treated with a drop of diazotised *p*-nitroaniline solution, should the sample contain guaiacolsulphonate. A further test consists in distilling a solution of the sample with syrupy phosphoric acid containing a small quantity of sodium chloride; in the presence of guaiacolsulphonate, the distillate gives a green coloration with ferric chloride and reduces ammoniacal silver nitrate solution. To determine potassium guaiacolsulphonate, the substance is oxidised by repeated evaporations with hydrochloric acid, bromine, and nitric acid, and the resulting sulphuric acid precipitated as barium sulphate.—W. P. S.

*Hexamethylenetetramine tablets; Evaluation of* —. W. O. Emery and C. D. Wright. J. Ind. Eng. Chem., 1918, 10, 606—608.

THE tablets are powdered and 0.5 grm. is boiled for 15 mins. under a reflux condenser with 100 c.c. of water and 25 c.c. of 10% hydrochloric acid; the solution is then cooled and diluted to 250 c.c. 10 c.c. of this solution is added to a mixture of 20 c.c. of potassium mercury iodide solution (mercuric chloride, 10, potassium iodide, 30, acacia, 5 grms.,



water, 200 c.c.) and 10 c.c. of 15% sodium hydroxide solution cooled previously to 0° C. The reaction proceeds according to the equation:

$\text{CH}_2\text{O} + \text{K}_2\text{HgI}_4 + 3\text{KOH} = \text{Hg} + \text{HCOOK} + 4\text{KI} + 2\text{H}_2\text{O}$ .  
The separated mercury is determined iodometrically by adding to the mixture 10 c.c. of 40% acetic acid and 20 c.c. of N/10 iodine solution and titrating the excess of iodine with thiosulphate solution. Each c.c. of N/10 iodine solution is equivalent to 0.001167 gram. of hexamethylenetetramine.

—W. P. S.

*Organic chemical reagents. I. Dimethylglyoxime.*  
R. Adams and O. Kamni. J. Amer. Chem. Soc., 1918, 40, 1281—1289.

METHODS of manufacturing the less common organic reagents are being thoroughly worked out at the University of Illinois and full details of the processes will be published with the object of inducing manufacturers to take up the production of the more useful of such reagents. The present paper deals with the production of dimethylglyoxime (1 kilo. in 8 hours) by Gandarin's method (this J., 1908, 1083). The removal of coloured impurities in dimethylglyoxime is effected by treating its saturated solution in 8% sodium hydroxide solution with an excess of concentrated ammonium chloride solution; the colourless precipitate of dimethylglyoxime is filtered off immediately. The purification is repeated if necessary.—C. S.

*Naphthalene [; Some arsenic] derivatives [of —].* A. A. Boon and J. Ogilvie. Pharm. J., 1918, 101, 129—130.

$\alpha$ -NAPHTHYLAMINE arsenate,  $\text{C}_{10}\text{H}_7\text{NH}_2\cdot\text{H}_3\text{AsO}_4$ , colourless needles, melting at 170° C., which become blue on exposure to the air or when heated above 165° C., is obtained when a mixture of  $\alpha$ -naphthylamine and arsenic acid is recrystallised from hot water. When one part of  $\alpha$ -naphthylamine is heated with  $\frac{2}{3}$  parts of the above arsenate for a short time at 200° C., a purple tarry mass is obtained containing 1-aminonaphthyl-4-arsinic acid together with a purple amorphous substance,  $\text{C}_{13}\text{H}_{11}\text{NO}$ , which can be extracted from the tar with concentrated sulphuric acid. (See also J. Chem. Soc., 1918, i., 461.)—D. F. T.

*Opium wax.* Rakshitt. See XII.

*Method for the separation and quantitative estimation of the lower alkylamines in presence of ammonia.* Weber and Wilson. See XXIII.

#### PATENTS.

*Tooth stopping preparations; Manufacture of —.*  
V. V. J. Andresen, Copenhagen, Denmark. Eng. Pat. 110,154, Aug. 15, 1917. (Appl. No. 11,744 of 1917.) Under Int. Conv., Sep. 30, 1916.

A MIXTURE of eugenol, formaldehyde solution, and alum is heated to 110°—125° C. in an open vessel and the gelatinous product is worked with a suitable amount of zinc oxide into a stiff paste. The resulting mass possesses the advantage for dental practice that it does not merely serve as a mechanical stopping, but also causes a local dentine anæsthesia and sterilises the parts under treatment.  
—D. F. T.

*Camphor; Purifying —.* E. C. R. Marks, London.  
From E. I. Du Pont de Nemours and Co., Wilmington, Del., U.S.A. Eng. Pat. 118,489, Sep. 28, 1917. (Appl. No. 14,034 of 1917.)

CRUDE camphor is heated in a closed vessel at 300°—500° F. for 30—90 mins., whereby the organic

impurities, such as camphor oil, are converted into substances differing in volatility from camphor or are rendered non-volatile or insoluble. Lime may be introduced to absorb water. The product is dissolved in alcohol, light petroleum, or other organic solvent with the aid of heat, filtered, and cooled in pans, when pure camphor crystallises out, or pure camphor is recovered from the product by sublimation.—J. H. J.

*Hydrogenisation and dehydrogenisation of carbon compounds.* C. Bosch, A. Mittasch, and C. Krauch, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,271,013, July 2, 1918. Date of appl., June 4, 1914.

IN the hydrogenation and dehydrogenation of carbon compounds a catalyst is used consisting of co-precipitated nickel and a compound, difficult to reduce, of oxygen with a metal which is capable of forming only a weak basic oxide (e.g. aluminium oxide). The product has the catalytic properties of nickel reduced at about 300° C.—L. A. C.

*Urea; Preparation of compounds of — with a calcium salt.* Knoll und Co., Ludwigshafen. Ger. Pat. 306,804, Sep. 8, 1916.

UREA is combined with calcium chloride (1 mol.  $\text{CaCl}_2$  to 4 mols. urea) by evaporating a solution of these substances to dryness or by concentrating an alcoholic solution until crystallisation occurs; the product can be injected subcutaneously for the treatment of such ailments as hay fever and bronchial asthma, and unlike calcium chloride alone does not give rise to pain and inflammation.  
—D. F. T.

*Glycolyl-p-aminophenol ethers; Preparation of —.*  
Farbw. vorm. Meister, Lucius, u. Brünig. Ger. Pat. 306,938, Aug. 24, 1916.

GLYCOLYL-*p*-AMINOPHENOL ethers, in particular the methyl and ethyl ethers which are of use as antipyretics, can be obtained by heating together the corresponding ethers of *p*-aminophenol with an anhydride or ester of glycollic acid. Under the term "anhydride" are included not only glycolide and polyglycolide but also the insoluble crystalline product formed on heating glycollic acid at 200°—250° C., which regenerates glycollic acid on prolonged contact with water.—D. F. T.

*Calcium halogenides and albumin; Manufacture of preparations of —.* E. Ritsert, Pharm.-chem. Institut, Frankfurt. Ger. Pat. 306,978, Sep. 16, 1915.

THE preparations are obtained by bringing together approximately equal weights of calcium halogenide and albumin in presence of a small quantity of water. The calcium chloride compound if dried at a gentle heat retains its power of swelling on treatment with water, but by stronger heating becomes insoluble, and this property renders it suitable for use in the finishing, etc., of textiles.

*Urea; Manufacture of —.* C. Bosch, A. Mittasch, and C. Neresheimer, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,274,503, Aug. 6, 1918. Date of appl., Apr. 16, 1915.

SEE Eng. Pat. 24,042 of 1914; this J., 1916, 71.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Photo-chemical reactions in photography.* F. Schanz. *Z. wiss. Phot.*, 1918, 17, 261—263.

The author has previously shown that all organic substances are decomposed by light of the wavelength which they absorb; colourless transparent substances are decomposed by ultra-violet light, or, if they are stable to daylight, by radiation of still shorter wavelength ("outer" ultra-violet). It is now suggested that the light-sensitiveness of photographic materials is due chiefly to the organic carrying medium of the silver salt (albumin, gelatin, collodion) rather than to the silver salt itself. The absorption spectra of dialysed albumin, 10% gelatin solution, and collodion solution to ultra-violet and outer ultra-violet are given; with the two latter absorption is from about 350  $\mu$  upwards and with albumin from 300  $\mu$ . The spectra for potassium bromide and silver nitrate solutions are also given, the absorption being negligible. The author suggests also, in analogy with the increase in light-sensitiveness of albumins by dyeing, that in the colour-sensitising of photographic plates it is the relationship between the dye and the medium which is important.—B. V. S.

*Spectral intensities; A method for the determination of — by photographic means.* G. Holst and L. Hamburger. *Z. wiss. Phot.*, 1918, 17, 264—267.

In front of the slit of a Hilger quartz-spectrograph is placed a revolving sector with an aperture increasing radially according to a logarithmic law. The relationship between the intensity of the spectral line and its length in the resulting photograph is given approximately by  $\log I_2/I_1 = 1.5(I_2 - I_1)$ ,  $l$  being measured in cm. The accuracy of the method is about 10% with a range of intensity ratio of 1:20. The method has been used in quantitative determinations of the influence of various factors on the light emission of gases.—B. V. S.

*Photometer liquids in tablet form.* W. Hausmann. *Z. wiss. Phot.*, 1918, 17, 268.

For greater convenience in handling, although with some loss of sensitiveness, photometer liquids are mixed with a sterilised agar-agar suspension, the mixture being allowed to set in Petri dishes. Eder's solution (mercuric chloride-ammonium oxalate) gives a colourless jelly in which a white opaque precipitate is formed on exposure to light; Roussin's liquid (sodium nitroprusside-ferrie chloride) gives a brownish coloured jelly turning to a strong blue colour.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Mercury fulminate; Colour reaction of — with phenylhydrazine.* A. Langhans. *Z. angew. Chem.*, 1918, 31, 161—163.

When dry mercury fulminate (0.2 to 0.3 gm.) is treated with 0.5 to 1 c.c. of phenylhydrazine and allowed to stand, the powder becomes olive-green changing to grey, whilst the liquid becomes brown and then reddish-brown. When diluted, after a few hours, with alcohol, and treated with dilute sulphuric acid, the liquid gives a fine reddish-violet coloration. The reaction, which is very sensitive, takes place more rapidly on heating. It can be used for the detection of mercury fulminate in the presence of potassium chlorate, antimony sulphide,

glass, and adhesive substance as contained in detonator and percussion cap compositions. The reaction is also produced by hydrochloric or nitric acids though not so sharply. Alkalis change the colour to yellow and give a precipitate, whilst acids restore the original colour. A solution of mercury fulminate in sodium thiosulphate or in pyridine also gives the reaction. If, prior to the reaction, the phenylhydrazine is treated with methyl iodide an intense violet to bluish-violet coloration of the character of methyl violet is obtained. The dye-stuff can be extracted with chloroform, and its reactions indicate that it is pararosanniline. (See also *J. Chem. Soc.*, Nov., 1918.)—C. A. M.

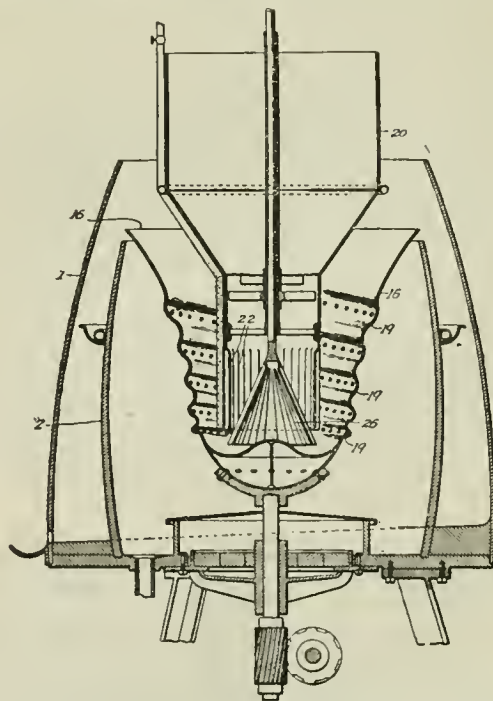
### PATENTS.

*Explosives; Ammonium perchlorate* —. Aktiebolaget Carlit, Stockholm, Sweden. Eng. Pat. 112,417, Aug. 21, 1917. (Appl. No. 12,009 of 1917.) Under Int. Conv., Dec. 30, 1916.

From 0.005 to 1% of a chlorate other than ammonium chlorate is added to or left in ammonium perchlorate, to render ammonium perchlorate explosives more sensitive to shocks and to increase the velocity of explosion.—T. St.

*Separators; Centrifugal* — [for nitrocellulose]. F. Matousek, Gary, Ind., U.S.A. Eng. Pat. 118,262, June 8, 1918. (Appl. No. 9441 of 1918.)

The material to be separated, e.g., a mixture of nitrocellulose and nitrating acid, is fed from the rotating hopper, 20, through the corrugated outlet, 22, which can be closed more or less by the corru-



gated cone, 26, into the bowl, 16, which is provided with a perforated spiral corrugation, 19. The liquid is discharged through the perforations into the inner casing, 2, and the solid portion is discharged over the lip of the bowl, 16, into the annular space between the casings, 1 and 2. Means are provided for flooding or flushing out both the feed hopper and the bowl with water.—W. H. C.



*Nitrated product and process of making same.*  
E. A. Barnes, Giant, Cal. U.S. Pat. 1,273,568,  
July 23, 1918. Date of appl., Oct. 11, 1915.

REFINED petroleum distillates containing a preponderance of saturated hydrocarbons and a smaller proportion of benzenoid hydrocarbons, are treated at a low temperature with a mild nitrating agent consisting of nitric acid, sulphuric acid, and water, whereby the benzenoid hydrocarbons are converted mainly into their mono-nitro-derivatives. These are separated, and by treatment with a stronger nitrating agent at a higher temperature are converted into polynitro-derivatives.—T. St.

*Edge runners, pan and like grinding mills.* Eng.  
Pat. 118,465. See I.

### XXIII.—ANALYSIS.

*Ultra-filter; New form of —. Some of its uses in biological and synthetic organic chemistry.* P. A. Kober. J. Amer. Chem. Soc., 1918, 40, 1226—1230.

QUANTITATIVE dialysis has been made possible by the discovery that all semipermeable membranes pervaporate (see this J., 1917, 1038). An apparatus for ultra-filtration by dialysis is figured and described. It consists essentially of a dialyser connected by a siphon to a pervaporator, and its action depends on pervaporating both the dialysate and the diffusate solutions during dialysis. Solutions containing humic material or highly coloured by-products which cannot be removed by charcoal are rapidly and completely clarified by ultra-filtration.—C. S.

*[Electrical] conductivity; New method for the determination of —.* E. Newbery. Chem. Soc. Trans., 1918, 113, 701—707.

ALTHOUGH the method introduced by Kohlrausch is almost universally used for the determination of the electrical conductivity of solutions, a critical examination shows that it is based on a number of assumptions which have not so far been subjected to the test of experiment. A new method is described in which continuous current is used and in which polarisation effects are completely eliminated. It is analogous to a method commonly used for measuring the resistance of a metallic conductor by determining the fall of potential along it during the passage of a known current. The non-polarisable electrodes employed in the method vary somewhat according to the nature of the solution under examination. The following values were obtained for the specific conductivity at 25° C.: N/1 KCl 0.1124, N/1 AgNO<sub>3</sub> 0.07796, N/1 H<sub>2</sub>SO<sub>4</sub> 0.2137. For the KCl solution the value is 0.3% greater, and for the H<sub>2</sub>SO<sub>4</sub> solution 3.6% less, than the value recorded by Kohlrausch.—H. M. D.

*Phosphoric acid; Determination of — as magnesium pyrophosphate. IV.* D. Balareff. Z. anorg. Chem., 1918, 104, 53—56.

EXPERIMENTS have been made on the precipitation of magnesium ammonium phosphate by Schmitz' method, which consists in adding ammonia to a hot acid solution of the phosphate-magnesia mixture. Whether precipitation takes place immediately or only on cooling depends upon the dilution and the quantity of ammonia added, but such changes in conditions do not affect the final results, nor do wide variations in the quantity of ammonium chloride present. The precipitate, however, is not pure, but loses some phosphoric acid on ignition, the residue being relatively rich in magnesium and

poor in phosphoric acid, but the errors involved approximately compensate one another. It is not advisable to redissolve and reprecipitate after ignition of the first precipitate, as is sometimes recommended. In presence of sulphate ions the results tend to be high and in presence of acetate ions, low, whilst nitrate ions are without influence. (See also J. Chem. Soc., Nov., 1918.)—E. H. R.

*Boric acid; Use of manna in the determination of —.* L. E. Hies. Analyst, 1918, 43, 323.

A FRESHLY prepared neutralised solution of manna may be used in place of glycerol in the determination of boric acid. As a rule a solution containing about 5 grms. of manna, which should be free from waxy portions, is equivalent to about 25 c.c. of 80% glycerol.—C. A. M.

*Alkylamines; Method for the separation and quantitative estimation of the lower — in the presence of ammonia.* F. C. Weber and J. B. Wilson. J. Biol. Chem., 1918, 35, 385—410.

THE new method depends upon the reaction of ammonia with mercuric oxide in alkaline solution and upon the reactions of the three classes of amines with nitrous acid and the subsequent reduction of the diaminonitrosoamine formed to diamine with nascent hydrogen. The ammonia is precipitated with mercuric oxide (François, this J., 1917, 432, 721) and then the total amines are determined by distillation into standard acid. After the titration, the distillate is evaporated with concentrated sulphuric acid, diluted, and an aliquot part treated with nitrous acid for 30 mins. in Van Slyke's apparatus, the amount of nitrogen evolved being a measure of the monoamines. The remainder of the solution is treated with nitrous acid for  $\frac{3}{4}$ —1 hour, then with a quantity of sodium hydroxide insufficient to neutralise it, allowed to stand for several hours, made alkaline, and distilled, the trialkylamine being absorbed in standard acid. The dialkylamine is determined by difference, or the distillate is treated with hydrochloric acid and zinc to reduce the dialkylaminonitrosoamine, and the diamine then determined by distillation. The method appears to give accurate results and may be applied to the analysis of fish products, putrid flesh, bad sausages, biological fluids, etc. (See further J. Chem. Soc., 1918, ii., 377.)—H. W. B.

*Adamkiewicz reaction [of proteins] and the transformation of glyoxylic acid into formaldehyde.* E. Voisenet. Bull. Soc. Chim., 1918, 23, 361—369.

AS opposed to the view of Hopkins and Cole (Roy. Soc. Proc., 1901, 68, 21) the author considers that this colour reaction (violet coloration on adding concentrated sulphuric acid to a solution of a protein in glacial acetic acid) is due to the presence of formaldehyde and not of glyoxylic acid. Acetic acid always contains traces of formaldehyde formed by oxidation. Glyoxylic acid also is readily decomposed with formation of formaldehyde. The method recommended for carrying out the test is to add to the protein sulphuric acid, followed successively by 1 drop of 0.4% formaldehyde and 1 drop of 1% sodium nitrite solution.—W. G.

*Device for determining the dropping point (melting point) of fats, waxes, paraffin wax, etc., and especially bitumens.* Dupré. See IIa.

*Determination of phthalic anhydride from crude phthalic acid.* Downs and Stupp. See III.

*Determination of nitrites.* Dienert. See VII.

*Determination of molybdenum [in ferromolybdenum] as lead molybdate.* Streblinger. See X.

*Determination of antimony in white metal.* Kaiser. See X.

*Optical dispersion of oils from an analytical point of view.* Fryer and Weston. See XII.

*Distillation method for the determination of water in soap.* Hart. See XII.

*Colorimetric determination of cobalt [in varnishes, paints, etc.].* Jones. See XIII.

*Determining the comparative melting points of glues as a measure of the jelly strength.* Sammet. See XV.

*Determination of ammonia and nitric nitrogen in soil extracts and physiological solutions.* Davisson. See XVI.

*Gravimetric determination of phosphoric acid [in fertilisers].* Mummery. See XVI.

*Determination of levulose in the presence of aldoses.* Herzfeld and Lenart. See XVII.

*Use of the dipping refractometer [in the examination of alcoholic liquids].* Randall. See XVIII.

*Method for the detection of foreign fats in butter fat.* Seidenberg. See XIXa.

*Determination of loosely bound nitrogen as ammonia in eggs.* Hendrickson and Swan. See XIXa.

*Polarimetric determination of morphine in opium.* Rakshit. See XX.

*Determination of theobromine.* Emery and Spencer. See XX.

*Modification of the Hiltner method for determining citral.* Parker and Hiltner. See XX.

*Evaluation of hexamethylenetetramine tablets.* Emery and Wright. See XX.

*Identification and determination of potassium guaiacolsulphonate.* Palkin. See XX.

*Organic chemical reagents. I. Dimethylglyoxime.* Adams and Kamm. See XX.

*Method for the determination of spectral intensities by photographic means.* Holst and Hamburger. See XXI.

*Photometer liquids in tablet form.* Hausmann. See XXI.

*Colour reaction of mercury fulminate with phenylhydrazine.* Langhans. See XXII.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Aubert. Regenerative tipping furnaces. 16,518. Oct. 10. (Fr., Oct. 22, 1917.)  
 Bedford. 15,990. See II.  
 Bolton. Liquid-sealed furnaces. 16,471. Oct. 10.  
 Candy. 16,023. See XIX.  
 Cruyt, and Heenan and Froude. Apparatus for filtering air or gases. 16,348. Oct. 8.  
 Davis and Twigg. Gas-fired regenerative furnaces. 16,324. Oct. 7.  
 Evans (Lourtie). Filters. 16,021. Oct. 2.  
 Fadden. Apparatus for producing mineral distillates of definite composition. 16,229. Oct. 7.  
 Gardiner and Sharp. Mixing machines. 16,440. Oct. 9.  
 Hall. Grinding, crushing, pulverising, and air-separating machinery. 16,130. Oct. 4.  
 Hamer and Mordecai. Intensive fractionation of volatile liquids. 15,999. Oct. 2.  
 Mauss. Centrifugal separation. 16,528. Oct. 10.  
 Morterud. Processes for evaporating liquids. 15,959, 15,961, and 15,962. Oct. 1. (Norway, Oct. 26 and Nov. 23, 1917.)  
 Morterud. Flow condenser. 15,960. Oct. 1.  
 Newhouse. Comminuting-mill. 15,856. Sep. 30.  
 Rafn. Cooling viscous liquids. 15,893. Sep. 30. (Norway, Nov. 30, 1917.)  
 Shaw, and Shaw and Co. Grinding, mixing, etc. machines. 15,845. Sep. 30.  
 Soc. l'Air Liquide. Effecting exothermic chemical syntheses. 16,160. Oct. 4. (Fr., Feb. 15.)  
 Zwermann. Kilns. 15,971. Oct. 1. (U.S., June 8.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 13,817 (1917). August and Hooson: Regenerative furnaces. (119,495.) Oct. 16.  
 13,965 (1917). Sharples. Separation of solids from liquids. (119,288.) Oct. 9.  
 14,963 (1917). Yates and others. See II.  
 17,265 (1917). Smallwood. Apparatus for cooling liquids or exposing them to air. (119,571.) Oct. 16.  
 146 (1918). Brand. Tunnel furnaces. (119,366.) Oct. 9.  
 227 (1918). Breakell. Means for covering in and discharging mills for crushing, grinding and mixing colours, chemicals, etc. (119,367.) Oct. 9.  
 462 (1918). Bynoe. Drying air. (119,590.) Oct. 16.  
 7813 (1918). De Bije. Furnaces. (115,644.) Oct. 9.



10,536 (1918). Roy, and Morson and Son. Means for carrying out catalytic or contact reactions between gases and/or vapours. (119,639.) Oct. 16.  
11,392 (1918). Ward and Ward. Centrifugal separators. (119,642.) Oct. 16.

## II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### APPLICATIONS.

Barbet et fils et Cie. 16,457 and 16,599. *See* III.  
Bedford. Drying vegetable and animal matter for fuel, etc. 15,990. Oct. 2.  
Broxburn Oil Co., and Findlater. Utilising residues from bituminous shales, cannel coals, etc. 16,542. Oct. 11.  
Bruyn. Coal fuel. 16,547. Oct. 11.  
Bury, Bury, and Olander. Recovery of gaseous products from plants for destructive distillation of organic materials. 16,121. Oct. 4.  
Clark. Coal fuel. 16,357. Oct. 8.  
Davis and Twigg. 16,324. *See* I.  
Engel. Clarifying and refining crude oils. 16,439. Oct. 9.  
Evans. Coal substitute. 15,991. Oct. 2.  
Fadden. 16,229. *See* I.  
Farman. Smokeless coal substitute. 16,255. Oct. 7.  
Helmès. Apparatus for treating oils. 16,170. Oct. 4.  
Lopez. Liquid fuel for internal-combustion engines. 16,398. Oct. 8.  
Palmer. Making natural-gas substitute. 16,304. Oct. 7. (U.S., Oct. 5, 1917.)  
Poore. Destructive distillation of wood, etc. 15,933. Oct. 1.  
Poore. Apparatus for destructive distillation of carbonaceous substances. 15,938. Oct. 1.  
Poore. Destructive distillation of wood, woody fibre, etc. 15,939. Oct. 1.  
Schaubermann. Lubricant. 16,314. Oct. 7.  
Taylor. Artificial fuel. 16,503. Oct. 10.

### COMPLETE SPECIFICATIONS ACCEPTED.

9713 (1917). Alexander (Adams). Method of decomposing hydrocarbon oils. (119,485.) Oct. 16.  
13,955 (1917). Mandle. Process of producing hydrocarbons. (119,284.) Oct. 9.  
14,963 (1917). Yates, Brayshaw, and Brayshaw. Gas-heated furnaces. (119,553.) Oct. 16.  
549 (1918). Thorsell and Lunden. *See* VII.  
4588 (1918). Pickard and Dobson. Apparatus for producing oil gas. (119,399.) Oct. 16.  
6230 (1918). Simpson. Heating-flues of chamber ovens or retorts for producing gas and coke or fuel. (119,413.) Oct. 9.

## III.—TAR AND TAR PRODUCTS.

### APPLICATIONS.

Barbet et fils et Cie. Recovery of tar from pyroligneous liquids. 16,457. Oct. 9. (Fr., Oct. 11, 1917.)  
Barbet et fils et Cie. Treatment of pyroligneous liquids derived from carbonisation of wood. 16,599. Oct. 11. (Fr., Oct. 11, 1917.)  
Soc. d'Eclairage, Chauffage, et Force Motrice. Production of high-percentage carbazole. 16,291. Oct. 7. (Fr., Dec. 4, 1917.)

### COMPLETE SPECIFICATIONS ACCEPTED.

9713 (1917). Alexander. *See* II.  
13,955 (1917). Mandle. *See* II.

## IV.—COLOURING MATTERS AND DYES.

### APPLICATION.

Halvorsen and Horbye. Manufacture of sulphur dyes. 16,381. Oct. 8.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

### APPLICATIONS.

Johnston and Sutherland. Method of degumming and cleansing stalk fibres. 16,181. Oct. 5.  
Langwell. Fermentation of cellulose. 15,942. Oct. 1.  
MacDonald. Solutions of cellulose esters. 16,017. Oct. 2.  
MacDonald. Manufacture of vulcanised fibre, and apparatus for washing fibrous and like substances to recover chemicals. 16,315. Oct. 7.  
Privett, and Thames Paper Co. Apparatus for drying paper, paper board, etc. 16,219. Oct. 5.  
Spicer, and Spicer and Sons. Filter paper, etc. 16,216. Oct. 5.

### COMPLETE SPECIFICATIONS ACCEPTED.

14,290 (1917). Wegmann & Co. Treatment of fabric pieces. (111,466.) Oct. 16.  
832 (1918). Aitken. Paper-making machines. (119,594.) Oct. 16.  
3336 (1918). Dreyfus. Manufacture of unflammable celluloid. (114,304.) Oct. 16.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### APPLICATIONS.

Crosland, Dixon, and Hawley. Machines for mercerising, dyeing, etc., hose, etc. 16,472 and 16,473. Oct. 10.  
Gott. Dyeing and bleaching textile materials. 16,430. Oct. 9.  
Macintyre. Machine for dyeing, bleaching, washing, etc., yarn in hank. 16,149. Oct. 4.  
Tütsch. Bleaching boiler plant. 15,965. Oct. 1. (Ger., Oct. 2, 1917.)

### COMPLETE SPECIFICATIONS ACCEPTED.

14,391 (1917). Calico Printers' Assoc., and Ashton. Printing of textile fabrics. (119,298.) Oct. 9.  
681 (1918). Dudley. Dyeing machine. (112,630.) Oct. 9.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

### APPLICATIONS.

Barbet et fils et Cie. 16,599. *See* III.  
Bradbury. Manufacture of salt. 16,573. Oct. 11.  
Busvold. Manufacture of bismuth compounds and metallic bismuth. 15,862. Sep. 30. (Norway, Oct. 3, 1917.)  
Morris. Production of antimonious oxide. 16,333. Oct. 8.  
Morris. 16,334 and 16,335. *See* XIII.  
Tyrer. Manufacture of manganates and permanganates. 16,446. Oct. 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

12,058 (1917). Dutt and Dutt. Manufacture of sodium and aluminium hypochlorites. (119,261.) Oct. 9.

12,363 (1917). Ashcroft. Extraction of potash or potassium salts from potash-bearing materials. (119,492.) Oct. 16.

13,975 (1917). Grossmann. Utilisation of nitre cake. (119,290.) Oct. 9.

549 (1918). Thorsell and Lunden. Manufacture of hydrogen. (119,591.) Oct. 16.

## VIII.—GLASS; CERAMICS.

## APPLICATION.

McCoy. Apparatus for production of sheet glass. 16,591. Oct. 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

14,293 (1917). Mond. Glass chemical ware. (119,297.) Oct. 9.

2667 (1918). Dupont. *See X.*

12,788 (1918). Soc. Anon. des Prod. Réfractaires de l'Ouest. Manufacture of silica bricks. (118,116.) Oct. 9.

## IX.—BUILDING MATERIALS.

## APPLICATION.

Alexander. Manufacture of bricks, building and paving blocks, etc. 16,619. Oct. 11.

## COMPLETE SPECIFICATION ACCEPTED.

2958 (1918). Borch. Manufacture of Portland cement. (113,785.) Oct. 9.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

Annable, and Nickel Concentration, Ltd. Extraction of nickel from silicate ores. 16,539. Oct. 10.

Basset. Manufacture of iron and steel from ore. 16,034. Oct. 2. (Fr., Oct. 3, 1917.)

British Thomson-Houston Co. (General Electric Co.). Coating metals. 15,944. Oct. 1.

Busvold. 15,862. *See VII.*

Gordon, and Spear and Jackson. Manufacture of steel for valves of internal-combustion engines. 16,237. Oct. 7.

Minerals Separation, Ltd., and Wood. Concentration of ores. 16,208. Oct. 5.

Ness. Electroplating and electro-cleaning apparatus. 16,142. Oct. 4.

Parkes and Thomas. Concentration and refining of metallic residues. 15,985. Oct. 2.

Reece. Treatment of ore concentrates, fines, slimes, dust, etc. 16,325. Oct. 7. (Australia, Sep. 18, 1917.)

Schneider et Cie. Ascertaining hardness of metal articles. 16,390. Oct. 8. (Fr., Nov. 7, 1917.)

Stein et Cie. Annealing furnaces. 16,517. Oct. 10. (Fr., Aug. 24.)

## COMPLETE SPECIFICATIONS ACCEPTED.

10,273 (1917). Hodson. Metallic alloys. (119,486.) Oct. 16.

1710 (1918). Stabilimentl Biak Ing. A. Pouchain. Manufacture of alloys containing nickel and zinc. (113,272.) Oct. 16.

2667 (1918). Dupont. Enamelling metal articles, and furnace therefor. (114,618.) Oct. 16.

5113 (1918). Gravel. Cleaning metals. (119,618.) Oct. 16.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

Morgan Crucible Co., and Speirs. Current-regulating devices for electrically heated crucible retorts, muffles, etc. 16,673. Oct. 12.

Ness. 16,142. *See X.*

## COMPLETE SPECIFICATIONS ACCEPTED.

14,309 (1917). Fuller Accumulator Co., and Wood. Electric storage batteries or accumulators. (119,530.) Oct. 16.

14,833 (1917). Kievits, and Kynoch, Ltd. Electric furnaces of the heated-coil type. (119,302.) Oct. 9.

5866 (1918). Aktieselskab Arendal Smelteverk. Electrode-holders for electric furnaces. (114,841.) Oct. 16.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

Engel. 16,439. *See II.*

Helmes. 16,170. *See II.*

## COMPLETE SPECIFICATION ACCEPTED.

17,577 (1917). Greenhalgh. Extraction of oil from vegetable fruits. (111,676.) Oct. 16.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

## APPLICATIONS.

Kelly. Flexible waterproof antiseptic and anti-fouling composition. 15,940. Oct. 1.

MacLean. Washable distempers or water paints. 16,317 and 16,318. Oct. 8.

Morris. Manufacture of antimony colours. 16,334 and 16,335. Oct. 8.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,705 and 10,706 (1917). Wade (Redmanol Chemical Products Co.). Manufacture of phenolic condensation products. (119,252 and 119,253.) Oct. 9.

227 (1918). Breakell. *See I.*

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

## APPLICATIONS.

Marshall and Walworth. Grinding vulcanised rubber. 16,565. Oct. 11.

Marshall. 16,566. *See XV.*



## XV.—LEATHER; BONE; HORN; GLUE.

## APPLICATIONS.

Carmichael. Unhairing of hides and skins. 16,188. Oct. 5.

Doran. Manufacture of leather. 16,175. Oct. 4. (Norway, May 11.)

Goodwin and Pratt. Treatment of hides, skins, etc. 15,870 and 15,871. Sep. 30.

Hewitt and Kay. Hide-tanning by compressed air. 16,194. Oct. 5.

Marshall. Manufacture of substitutes for leather and rubber goods. 16,566. Oct. 11.

Trenaman. Composition for waterproofing leather articles. 15,969. Oct. 1. (U.S., Dec. 20, 1917.)

## COMPLETE SPECIFICATION ACCEPTED.

14,982 (1917). Ward. Substitute for leather. (119,304.) Oct. 9.

## XVI.—SOILS; FERTILISERS.

## APPLICATIONS.

August. Treatment of soil. 16,416. Oct. 9.

Fitzgerald. Manufacture of nitrogenous manures, etc. 16,220. Oct. 5.

## XVII.—SUGARS; STARCHES; GUMS.

## APPLICATIONS.

Alexander. Manufacture of edible syrup from sugar beetroot. 16,607. Oct. 11.

Marks (Ideal Coated Paper Co.). Adhesive. 16,954. Oct. 11.

## XVIII.—FERMENTATION INDUSTRIES.

## APPLICATION.

Langwell. 15,942. *See* V.

## COMPLETE SPECIFICATION ACCEPTED.

16,829 (1917). Kazman. Production of alcohol. (119,333.) Oct. 9.

## XIX.—FOODS; WATER PURIFICATION; SANITATION.

## APPLICATIONS.

Beer. Process for recovering and making cheese of food matters in whey. 16,131. Oct. 4.

Brodrick. Manufacture of drinks. 16,642. Oct. 12.

Candy. Filters for purifying liquids. 16,023. Oct. 2.

James, and Heenan and Froude. Refuse destructors. 16,400. Oct. 9.

Nieuwhof, Surie, & Co., and Surie. Manufacture of milk powder. 16,658. Oct. 12.

Vuyksteke. Water, etc., filters. 16,020. Oct. 2.

Weeks. Germicide solution. 16,606. Oct. 11.

Westaway. Means for preserving milk, cream, fish, etc. 16,310. Oct. 7.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,455 (1917). Lecomte. Sterilising milk and similar liquids. (116,490.) Oct. 9.

10,414 (1918). Turney. Process of making a new food product of milk. (119,430.) Oct. 9.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

## APPLICATIONS.

Imray (Soc. Chem. Ind. in Basle). Manufacture of esters of N-monoarylsulphomonoalkylamido-oxyaryl compounds and arylsulpho esters of amido-oxyaryl compounds. 16,379. Oct. 8.

Morris and Reeve. Medicinal preparation. 16,293. Oct. 7.

Rockefeller Institute for Medical Research. Arsenical compounds. 16,092—16,096. Oct. 3. (U.S., Oct. 3, 1917.)

## COMPLETE SPECIFICATIONS ACCEPTED.

8259 (1917). Marks (Du Pont de Nemours and Co.). Production of alcohols. (119,249.) Oct. 9.

8862 (1917). Boake and Durrans. Production of dimethyl sulphate. (119,250.) Oct. 9.

13,964 (1917). Imray (Soc. Chem. Ind. in Basle). manufacture of a preparation from *Secale cornutum*. (119,287.) Oct. 9.

14,150 and 14,151 (1917). Selden Co., and Gibbs. Process for oxidising aromatic hydrocarbons and the lower oxidation products thereof. (119,517 and 119,518.) Oct. 16.

8026 (1918). Utheim. Manufacture of chloroform. (116,094.) Oct. 16.

## XXIII.—ANALYSIS.

## COMPLETE SPECIFICATIONS ACCEPTED.

14,352 (1917). Parsons and Law. Means for detecting or measuring moisture. (119,533.) Oct. 16.

18,331 (1917). Towers and Harger. Burettes and the like. (119,580.) Oct. 16.

## I.—GENERAL ; PLANT ; MACHINERY.

## PATENTS.

*Ball-mill.* C. Candlot, Paris. Eng. Pat. 115,223, Dec. 13, 1917. (Appl. No. 18,459 of 1917.) Under Int. Conv., Apr. 25, 1917.

A BALL-MILL is provided with two series of oppositely directed helicoidal ribs which serve to heap up the material being ground towards the middle of the cylinder. A conical screen is provided at the outlet end.—W. H. C.

*Separation of solids from liquids; Apparatus for the continuous* —. A. Bontemps and G. N. Vis, Paris. Eng. Pat. 118,855, Mar. 16, 1917. (Appl. No. 3866 of 1917.)

A HOLLOW cone having a raised screw thread on its outer surface is rotated point downwards within another perforated cone surrounded by an outer casing. The material to be separated is forced into the space between the cones from below. The liquid passes through the perforated cone into the outer casing and the solid matter is carried upwards by the screw thread and is discharged at the upper end into a suitable receptacle. Steam or other treating agent may be passed into the interior of the hollow cone, the distance of which from the outer cone may be adjusted by a screw.

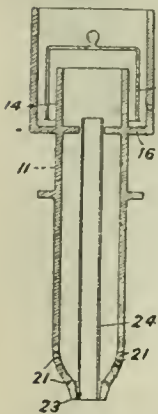
—W. H. C.

*Lagging and process for preparing the same.* E. Foster, St. Heliers Bay, New Zealand. Eng. Pat. 118,871, Aug. 9, 1917. (Appl. No. 11,454 of 1917.)

SODIUM silicate, 100 parts, is heated, and mixed with diatomaceous earth 40—60 parts, and lime 2—10 parts. The mass is placed in heated moulds and heated in a furnace to 400° F. (205° C.) or upwards. The product is porous and easily detached from the mould.—W. F. F.

*Spraying liquids; Apparatus for* —. I. Hechenbleikner, Assignor to Chemical Construction Co., Charlotte, N.C. U.S. Pat. 1,273,856, July 30, 1918. Date of appl., Feb. 14, 1918.

A DEVICE intended more especially for spraying acids into concentrating apparatus comprises a tube, 11, provided with spraying apertures, 21, and also with a central overflow tube, 24. The acid contained in the receptacle, 16, overflows over the upper edge of 14, and through openings into the tube, 11, and thence out through the spraying apertures, 21. Should these become clogged, the acid rises in the tube, and overflows into the inner tube, 24. This can be observed through the transparent cover, 25, and in such case the cover is removed and the tube, 24, raised, whereupon a rapid flow of acid down 11 and out through the opening, 23, clears the apertures, 21.



*Absorbing heat; Apparatus for* —. [Vacuum evaporator.] H. H. Dow and T. Griswold, jun., Assignors to The Dow Chemical Co., Midland, Mich. U.S. Pat. 1,275,231, Aug. 13, 1918. Date of appl., June 7, 1916.

A VACUUM pan has a number of approximately vertical upflow tubes for the liquid to be evaporated

and a downflow tube of considerably larger cross-section situated nearer the outlet for the heating medium than to the inlet. The upflow tubes are distributed uniformly throughout the chamber, except adjacent to the inlet and outlet for the heating medium, where two wedge-shaped spaces are left, the larger end of each of these spaces facing respectively towards the inlet and outlet for the heating medium.—W. H. C.

*Dust-collector.* W. J. P. Cloakey, Brantford, Ont., Canada. U.S. Pat. 1,275,663, Aug. 13, 1918. Date of appl., Jan. 5, 1918.

A SCREEN drum having radial longitudinal screen partitions extending part of the way from the periphery towards the axis is rotated about a stationary cylinder and dips into water in a tank. The cylinder has a number of external arc-shaped wings extending lengthwise, the arcs being of greater length than the distance between any two of the partitions. A port is provided in the side of the cylinder between the wings, and inside the cylinder but out of alignment with the port is a rotary beater into which water is conducted near its axis. The dust-laden air is led into the beater and passes thence into the cylinder.—W. H. C.

*Air-cleaner.* A. C. Bennett, Minneapolis, Minn. U.S. Pat. 1,276,016, Aug. 20, 1918. Date of appl., Feb. 28, 1917.

A VERTICAL cylindrical casing is provided on its inner wall with a helical conduit terminating near the lower end. The air to be treated is discharged through this conduit so that it passes into the lower part of the casing with a whirling motion. A conical baffle-plate is arranged with its apex at the level of the lower end of the conduit, and its base spaced from the lower wall of the casing. Separated solid material falls between the cone and the wall of the casing and is collected in a well in the bottom of the casing.—W. F. F.

*Vacuum dehydrator; Continuous* —. C. Biesel, El Paso, Tex. U.S. Pat. 1,276,163, Aug. 20, 1918. Date of appl., Jan. 11, 1917.

A ROTARY hollow drum is fitted with a peripheral suction chamber and with an outer filtering medium, and the filter-cake is removed from the drum face by a number of wires supported on a frame and arranged tangentially to the filtering surface. The filtered liquid is removed from the suction chamber without interrupting the vacuum.

—C. A. K.

*Heating boilers by waste gases; Means for* —. L. S. Schmidt, Pittsburgh, Pa. U.S. Pat. 1,276,421, Aug. 20, 1918. Date of appl., Feb. 4, 1916.

THE outlet of a waste gas flue is arranged between the inlet of a boiler flue and the inlet of a flue leading to the stack. The ports are adjacent to one another and in the same horizontal plane. A valve member with a curved passage is arranged over the ports so that the central waste gas port may be put in communication with either of the side ports by moving the valve member, while at the same time the port not in use is automatically covered by a flap. All the ports are provided with water seals on their horizontal surfaces out of the path of the waste gases.

—W. F. F.



*Dry-kiln.* H. D. Tiemann, Madison, Wis., and N. de W. Betts, Linwood, Utah, Assignors to the Government and people of the United States. U.S. Pat. 1,276,451, Aug. 20, 1918. Date of appl., Apr. 6, 1917. (Dedicated to the public.)

A DRYING chamber is provided with a flue adjacent to it for heating the circulating medium, and steam is discharged at a point adjacent to the heating device. The steam passes rapidly through the heating flue and induces a large circulation through the drying chamber, and at the same time becomes superheated.—W. F. F.

*Suspended particles; Process of freeing liquids from —.* V. M. Goldschmidt, Assignor to Titan Co. A/S. of Norway, Christiania, Norway. U.S. Pat. 1,276,644, Aug. 20, 1918. Date of appl., Jan. 31, 1918.

A LIQUID containing suspended solid particles of electropositive colloidal silicon hydroxide is mixed with a ferric salt and treated with hydrogen sulphide. Electronegative colloidal sulphur is precipitated, the particles of which envelop and coagulate the electropositive particles, which may then be removed.—W. F. F.

*Dephlegmator and rectifying column.* D. Pyzel, San Francisco, Cal. U.S. Pat. 1,276,690, Aug. 20, 1918. Date of appl., Oct. 10, 1916.

A VERTICAL cylindrical shell is provided with a vapour inlet at the bottom and outlet at the top. A cooling coil, fixed within the shell near the top, serves to condense the heavier products, which fall back into a rectifying column below and are there revaporised by heated vapours which circulate around the column. A heating coil in the bottom of the shell serves to revaporise any liquid condensing in the rectifying column.—W. H. C.

*Separating liquids from solids; Process of and apparatus for —.* A. M. Nicholas, Torrington, N.S.W. Eng. Pats. 110,349 and 110,350, Sep. 11, 1917. (Appl. Nos. 13,021 and 13,022 of 1917.) Under Int. Conv., Oct. 13, 1916.

SEE U.S. Pats. 1,268,273 and 1,268,274 of 1918; this J., 1918, 497 A.

*Cooling and otherwise treating gases with liquids; Apparatus for —.* A. W. Mathys, London. U.S. Pat. 1,275,583, Aug. 13, 1918. Date of appl., Nov. 8, 1916.

SEE Eng. Pat. 111,113 of 1917; this J., 1918, 141 A.

*Solid fuel for power.* Eng. Pat. 118,777. See IIA.

*Liquefying gases.* U.S. Pat. 1,273,929. See VII.

*Kilns.* U.S. Pats. 1,275,352 and 1,275,712. See VIII.

*Furnace.* U.S. Pat. 1,274,795. See XI.

*Drying food, etc.* Eng. Pat. 118,911. See XI(A).

## IIA.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Effects of moisture on spontaneous heating of stored —.* S. H. Katz and H. C. Porter. U.S. Bureau of Mines, Tech. Paper 172, 1917. 25 pages.

The relative rates of absorption of oxygen by dry and moist samples of two bituminous coals were determined. The dry samples were prepared by desiccation over phosphoric anhydride *in vacuo*

or in nitrogen; the moist samples were simply air-dried. 100 grms. of the coal (through 80-mesh, or 100-mesh sieve) was exposed to about  $4\frac{1}{2}$  litres of air dried by phosphoric anhydride or to moist air, in a closed vessel connected to a manometer and maintained at 25° C. in a water-bath for 100 days. Computations were made from the changes in pressure and analyses of the gaseous contents of the vessels. The results are tabulated and shown graphically. The volume of oxygen absorbed ranged between 279 c.c. and 521 c.c. in the different experiments. Carbon dioxide (10 c.c. to 43 c.c.) and a trace of carbon monoxide (4 c.c. to 10 c.c.) were produced, but the bulk of the absorbed oxygen remained in the coal substance. Dryness favoured the rate of oxidation for one coal but retarded it for the other. The authors conclude that as both coal and air contain moisture under natural conditions of coal storage, water, other than the excess that actually wets the coal, does not play any important part in increasing the danger of spontaneous heating. On the other hand, excess water, held by capillary action in the small coal of a storage pile, probably stifles ventilation of the pile, and consequently the heat of slow oxidation gradually accumulates. To stop this action, the segregation of small coal, when building a storage pile, must be prevented.—T. F. E. R.

*Blue water-gas practice in conjunction with benzol recovery.* E. F. Keable. Eastern Counties Gas Assoc., Oct. 3, 1918. Gas J., 1918, 144, 77—79.

The author describes results obtained from a "K and A (Smith's modification) plant." The producer consists of a steel shell containing two generators, each of which is surrounded in the upper portion by a regenerator. During the "blow" ( $\frac{3}{4}$  to 1 min.) the blast enters the bottom of both generators, passes through the fuel and meets secondary air on entering the regenerators. The "run" occupies 5 mins. and the steam passes down one regenerator (becoming superheated) into the respective generator, whence it proceeds under a midfeather into the second generator and then passes up through the column of cool coke feeding the latter generator, to the gas outlet. When carburetting, the oil is simultaneously cracked in the second regenerator and the oil-gas becomes mixed with the blue water-gas at the point of entry into the cool coke column. After the next "blow" the above operations are repeated in the reverse direction. With this plant, clinkering troubles are reduced, heat is given up by the blue water-gas to the cool coke, the gas leaves the plant freed from dust, and the content of carbon dioxide is low. The addition of 20—32% of blue water-gas (sp. gr. 0.6, cal. value 331 B.Th.U. gross per cub. ft.) to coal-gas of 660 B.Th.U. gross per cub. ft. produced a mixture which averaged 520 B.Th.U. gross, and which gave no trouble in use. 29.8 lb. of coke was used per 1000 cub. ft. of blue water-gas. The simultaneous working of the blue water-gas plant and the benzol-recovery plant necessitated very carefully balanced conditions to maintain a uniform quality of gas. 1.79 galls. of crude benzol was recovered per ton of coal carbonised.

—T. F. E. R.

*Gases; Differential pressure method for the determination of [benzene] vapours in —.* H. S. and M. D. Davis. J. Ind. Eng. Chem., 1918, 10, 709—712.

Two flasks are connected by a manometer; one is filled with air and the other with air containing benzene in quantity less than will saturate the air. A small bulb of benzene is then broken in each flask. In the flask containing air, the benzene will produce the total saturation pressure whilst in the

other flask it will only add the amount of pressure necessary to bring the pressure up to saturation. The manometer will, therefore, indicate a difference in pressure between the two flasks equal to the pressure of the benzene vapour in the original air-benzene mixture. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Gases; Application of the differential pressure method to the determination of benzene and total light oils in —.* H. S. and M. D. Davis and D. G. MacGregor. J. Ind. Eng. Chem., 1918, 10, 712–718.

The method described in the preceding abstract may be used for the determination of total benzene, toluene, and xylene in gases. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Gases; Absorption of light oils from —.* H. S. and M. D. Davis. J. Ind. Eng. Chem., 1918, 10, 718–725.

The flow of oil necessary to remove completely benzene from a gas may be calculated, since the vapour pressure of benzene in oil is governed by Henry's law for the solubility of gases in liquids and by Von Babo's law for the constancy of the fractional lowering of the vapour pressure from a solution over variations in temperature. A method, based on the determination of the molecular weight of an oil dissolved in benzene and calculation of the fractional lowering of the vapour pressure of benzene by the addition of 99% of the oil, is suggested for standardising oils used for absorbing vapours from gases. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Benzol; Production of — from Italian crude petroleum.* A. G. Rodano. Annali Chim. Appl., 1918, 10, 23–26.

A SAMPLE of crude petroleum from the province of Pavia had the following characters:—Sp. gr. at 15° C., 0.9122; viscosity (Engler) at 20° C., 1.77; flash-point, 80° C.; calorific power, 10,600 cals.; and total sulphur, 0.22%. It began to distil at 180° C., and on distillation 160 c.c. yielded the following fractions:—From 180° to 250° C., 26 c.c.; 250° to 280° C., 26 c.c.; 280° to 310° C., 18 c.c.; 310° to 360° C., 18 c.c.; 360° to 370° C., 6 c.c.; and residue, 6 c.c. The oil was only fit for heating purposes. When "cracked" (see this J., 1917, 1091), 100 c.c. of the crude oil yielded 25 litres of combustible gas, 9 c.c. of oils distilling up to 122° C.; 5 c.c. between 122° and 170° C., and 16 c.c. of residual tar completely soluble in aniline. The oil distilling up to 122° C. consisted of a mixture of benzol and toluol in which the latter predominated. On "cracking" the fractions (52 c.c.) of the original oil distilling up to 280° C. they yielded 94% of benzol and toluol, equivalent to 4.8% on the original petroleum, or only about half of that produced by "cracking" the original petroleum. On the other hand, in the second case the residue from the original oil after distilling off the lighter portion could be used as a lubricant.—C. A. M.

*Bitumens; Valuation of natural and artificial — [for insulating cables] by means of a new "bitumen tester."* Dupré. Chem.-Zeit., 1918, 42, 445–446. (See this J., 1918, 615 A.)

Of the few varieties of bitumen having the requisite elasticity and hardness for use as insulators for electric cables elastolite is the best known. Since, owing to the war, this is no longer procurable in Germany, attempts have been made to prepare substitutes ("insulation tar," "insulation masses," etc.) by incorporating up to 40% or more of kaolin,

chalk, etc., with natural or artificial bitumens having a dropping-point (melting-point) of about 40°–60° C. Such addition, however, only retards the flowing of the mass at higher temperatures, and cables insulated with such materials lose their shape as certainly, though not so soon, as if the unfilled bitumen were used. The improvement of ordinary bitumens, asphalts, pitches, etc., so as to raise the dropping-point, while retaining their elasticity and ductility, can only be effected by a deep-seated chemical alteration of the bitumens. For example, if the mineral matter is made to combine with the bitumen as in Schön and Co.'s patent process (not yet published), the dropping-point of a bitumen may be raised from, e.g., 41° C. to 115° C. The so-called "original Trinidad asphalt épuré" has a dropping-point of about 105° to 115° C., and contains about 30 to 40% of mineral substances. But, owing to their reduced elasticity, these products cannot be used as the permanent basic material for cable masses, and are, at best, only suitable for admixture with elastic bitumens of lower dropping-point. The second method of raising the dropping-point is by chemical treatment of the bitumen without the addition of mineral matter. Only first-grade bitumens are suitable for cable masses, viz., those with the requisite elasticity, ductility, and adhesiveness, and a dropping-point of at least 75° to 95° C., whilst if the "semi-bitumens," including natural or artificial tars, asphalts, petroleum pitch, etc., with a dropping-point of 40° to 60° C. be used, the cable mass will lose its form when exposed to an external temperature of 20° to 30° C.—C. A. M.

*Analysing spent oxide.* Wentzel. See VII.

#### PATENTS.

*Peat; Treatment of — for fuel and apparatus therefor.* K. E. Edgeworth, British Expeditionary Force, France. Eng. Pat. 118,903, Sep. 24, 1917. (Appl. No. 13,714 of 1917.)

WET peat, which has previously been heated to 150° C. in a closed vessel, is forced into a filtering vessel at a pressure of 600 lb. per sq. in. and a temperature of 200° C. The filtering elements are conical, with thick walls, and are formed of unglazed porcelain or earthenware. These elements are mounted on a vertical perforated outlet pipe in the centre of the vessel, and their outer edges are spaced from the walls of the vessel, so that the elements are entirely surrounded by the wet peat. The water percolates through the walls of the cones into the discharge pipe. To discharge the peat, the inlet pipe is closed, and the filtration allowed to continue till the temperature falls to 105°–130° C. An outlet passage is then opened and the sudden decrease in pressure and consequent generation of steam disintegrates and forces out the peat. Modified forms of filtering elements are also described.—W. F. F.

*Coal; Process of treating —.* C. H. Smith, Short Hills, N.J., Assignor to International Coal Products Corporation, Richmond, Va. U.S. Pats. (A) 1,276,427 and (B) 1,276,428, Aug. 20, 1918. Dates of appl., Jan. 20 and 31, 1916.

(A) COAL is heated until it reaches the plastic state and is then mixed with combustible material such as coke for the purpose of absorbing gases and condensed products entrained in the plastic residue. The mass is then moulded into briquettes by pressure applied at various stages of cooling. (B) The coal is heated until plastic, removed from the retort, subjected to a partial vacuum to remove gases entrained in the mass, and then moulded into briquettes.—T. F. E. R.



*Briquette and method of making the same.* C. H. Smith, Short Hills, N.J., Assignor to International Coal Products Corporation, Richmond, Va. U.S. Pat. 1,276,429, Aug. 20, 1918. Date of appl., Nov. 15, 1916.

BRICQUETTES are made (1) by carbonising a mixture of a binder and partially carbonised coal, (2) by mixing coal, coke, or partially carbonised coals, too low in volatile matter for direct briquetting, with a hydrocarbon binder so as to obtain a mixture containing 11–17% of volatile matter, then briquetting, and carbonising at 1400°–2000° F. (760°–1090° C.), (3) by partially distilling coal at a low temperature, compressing the residue into moulds and then distilling at a higher temperature to produce a strong coherent briquette.—T. F. E. R.

*Pitch; Burners for —.* South Metropolitan Gas Co., and A. Kirby, London. Eng. Pat. 118,890, Sep. 14, 1917. (Appl. No. 13,205 of 1917.)

MELTED pitch after passing through a steam jacketed chamber issues from an orifice into an injector jet and is then atomised, and passes into the furnace where it is burned. The outlet of pitch through the burner orifice is regulated by a rotating disc provided with holes of different sizes. Efficient atomisation is assured by employing a steam jet in close proximity to the injector jet.

—T. F. E. R.

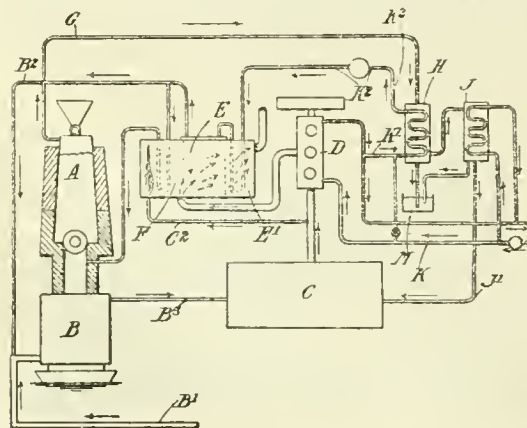
*[Coke ovens;] Plant for utilising waste heat [from —].* R. D. Martin, El Reno, Okla. U.S. Pat. 1,275,962, Aug. 13, 1918. Date of appl. June 20, 1917.

A NUMBER of coke ovens are arranged in two parallel rows, with a main flue extending between the rows midway of their height, and in contact with the sides of the ovens. Each oven has a tubular neck closed by a cover, and a flue leads from each neck over the curved top of the oven into the main flue. A steam generator is mounted on the setting of the ovens and a furnace and flue are arranged immediately below it and between the outer portions of two of the ovens. The furnace flue is connected to the main flue by a horizontal flue passing between the two ovens.

—W. F. F.

*Power production; Use of solid fuel for large scale —.* Merz and McLellan, and A. C. Michie, Newcastle-on-Tyne, and E. G. Weeks, Monk-seaton, Northumberland. Eng. Pat. 118,777, July 6, 1917. (Appl. No. 717 of 1918.)

COAL is fed continuously into a low-temperature vertical retort, A, and the coke is discharged at the bottom into a producer, B. Air and steam are



supplied by pipes, B<sup>1</sup>, B<sup>2</sup>, to the producer, and the resulting gas is passed through a stripping plant, C, to an internal combustion engine, D. The burnt gas passes through a steam generator, E, provided with an economiser, E<sup>1</sup>, and thence to the exhaust. The steam passes partly through a superheater, F, heated by a portion of the stripped gas supplied by the pipe, C<sup>2</sup>, to the retort, A, and partly through the pipe, B<sup>2</sup>, to the producer. Distillation gases from the retort pass by the pipe, G, to the cooler, H, supplied with water from the engine jackets, D, by the pipe, K<sup>1</sup>. The heated water then passes by the pipe, K<sup>2</sup>, to the steam generator, E. The gas leaving the cooler passes to a second cooler, J, cooled by cold water, and the condensed products from the two coolers collect in the vessel, M. The residual gas passes by the pipe, J<sup>1</sup>, to the stripping apparatus, C, and thence to the engine, D.

—W. F. F.

*[Gas] retort settings; Horizontal and inclined —.* R. Dempster and Sons, Ltd., and R. M. Brooke, Elland, Yorks. Eng. Pat. 118,804, Mar. 14, 1918. (Appl. No. 4504 of 1918.)

IN a setting for horizontal or inclined retorts having a step grate, producer gas is withdrawn from the furnace at the back, and passes through a flue extending through the setting from front to back to a number of horse-shoe burners along both sides of the combustion chambers. Secondary air is supplied through annular concentric passages in the burners.—W. F. F.

*Gas producers and the like; Open hearth —.* The Dowson and Mason Gas Plant Co., Ltd., and F. A. Webb, Manchester. Eng. Pat. 118,689, Sep. 12, 1917. (Appl. No. 13,049 of 1917.)

A VERTICAL producer is provided with a grate formed of a metal plate in the shape of an inverted frustum of a cone. The outer edge of the plate is attached to the bottom of the producer wall by a fixed metal ring provided with air holes. The ring is surrounded by a metal sleeve having corresponding holes, which can be rotated so as to vary the passages for secondary air to the producer. Below the conical grate and spaced from it, is a horizontal fire-bar grate of larger diameter. The bars are hollow and of triangular section with their bases at the top. They are threaded on to a steam pipe at one end, and their sloping sides are perforated so that the steam admitted to the fire is dried and superheated. On withdrawing the steam pipe, any one of the fire-bars may be removed without drawing the fire.—W. F. F.

*Gas-producers; Feeding mechanism for —.* C. W. Lummis, Worcester, Mass., Assignor to Morgan Construction Co. U.S. Pat. 1,274,176, July 30, 1918. Date of appl., June 28, 1915.

FUEL is fed intermittently from a hopper into a horizontal cylinder rotating in a casing, and is discharged when an opening in the side of the cylinder is over a discharge passage in the casing leading to the producer. The casing encloses not more than half of the lower surface of the cylinder, so that the latter is movable laterally. Means are provided for limiting the charge to a smaller volume than the full capacity of the cylinder, so that fuel is prevented from lodging between the cylinder and its seating during rotation.—W. F. F.

*Gas producer.* A. M. Taylor, Hegewisch, Ill., Assignor to General Chemical Co., New York. U.S. Pat. 1,275,789, Aug. 13, 1918. Date of appl., Feb. 10, 1916.

THE top of the producer has a stationary annular cover provided with poke-holes arranged at intervals around the central opening. A movable cover,

which rotates on a circular track on the stationary cover, carries a coal distributor and is provided with a poke-hole which registers in turn with the different poke-holes in the stationary cover. By this arrangement it is possible to distribute the coal uniformly over the entire horizontal area of the producer and to bring various portions of the producer within range of the poke-hole.—T. F. E. R.

*Hydrogen and carbon-black; Process for the manufacture of* —. R. H. Brownlee and R. H. Uhlinger, Assignors to American Nitro-Products Co., Pittsburgh, Pa. U.S. Pat. 1,276,487, Aug. 20, 1918. Date of appl., Jan. 21, 1914.

A SUITABLE hydrocarbon is decomposed by contact with heated refractory material in a closed chamber, in the absence of air or catalyst. After cooling, the separated carbon is collected and removed, and the hydrogen washed to remove impurities.—C. A. M.

[*Mineral*] *oil refining apparatus*. F. E. Wellman, Assignor to The Kansas City Gasoline Co., Kansas. U.S. Pat. 1,273,966, July 30, 1918. Date of appl., July 22, 1916.

LIQUID hydrocarbons are heated in a vessel and the vapours conducted to a condenser at a higher level, having a discharge pipe provided with means for drawing off the different fractions. This pipe is also connected with a pump for drawing off the heavier hydrocarbons, which are then cracked by means of a connected superheating coil. The resulting fractions are separated by means of a dephlegmator and condenser, and the heavier uncracked hydrocarbons from the dephlegmator are conducted back to the original source of supply.—C. A. M.

[*Mineral*] *oil-refining apparatus*. F. E. Wellman, Assignor to The Kansas City Gasoline Co., Kansas City, Kans. U.S. Pat. 1,275,337, Aug. 13, 1918. Date of appl., July 22, 1916.

A RETORT for high temperatures and pressures is contained in a housing adjacent to which are bomb-proof operators' and cleaners' vestibules, provided respectively with observation connections and bomb-proof closures communicating with the interior of the still-housing. Independent underground passages, the entrances to which are bomb-proof houses situated at a distance from the still, communicate respectively with all the operators' vestibules and with all the cleaners' vestibules.—T. St.

*Gasoline; Process of producing* —. J. C. Black, Los Angeles, Assignor to Standard Oil Co., Richmond, Cal. U.S. Pat. 1,275,648, Aug. 13, 1918. Date of appl., Feb. 21, 1916.

IN the fractional distillation of petroleum a fraction having an initial boiling point lower than the final boiling point of the gasoline fraction, and Beaumé gravity lower than that of the gasoline fraction, is caused to absorb natural hydrocarbon gas at approximately normal temperature. It is then possible by redistilling the gas-treated material to obtain a fraction which is identical in its gravity, boiling point range, and vapour tension, with the gasoline fraction.—T. St.

*Aromatic hydrocarbons [from petroleum products]; Process of obtaining* —. F. B. Holmes, Woodbury, N.J., Assignor to E. I. du Pont de Nemours and Co. U.S. Pat. 1,276,219, Aug. 20, 1918. Date of appl., Apr. 27, 1915.

GASOLINE containing aromatic hydrocarbons is treated with a liquid aromatic nitro-compound of

a higher degree of nitration than the mono-nitro-compound, and capable of causing the gasoline to separate into two parts, one of which contains a higher and the other a lower percentage of aromatic hydrocarbons than did the original gasoline.—T. St.

*Fuels; Liquid* —. F. C. Axtell, New York. Eng. Pat. 109,805, Sep. 6, 1917. (Appl. No. 12,809 of 1917.) Under Int. Conv., Sep. 5, 1916.

SEE U.S. Pat. 1,201,638 of 1916; this J., 1916, 1254. (Reference is directed, in pursuance of Sect. 7, Subsect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 17,841 of 1907, 28,072 of 1913, and 2515 and 18,225 of 1914; this J., 1908, 994; 1915, 860, 948, 1001.)

*Paraffin; Process of purifying* —. *Process of saturating oils. Purifying oils*. A. M. McAfee, Port Arthur, Tex., Assignor to Gulf Refining Co., Pittsburgh, Pa. U.S. Pat. 1,277,092, 1,277,328, and 1,277,329, Aug. 27, 1918. Dates of appl., June 10, 1918, Dec. 6, 1915, and Mar. 22, 1918.

SEE Eng. Pat. 22,922 of 1914; this J., 1916, 299.

## II.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Acetic acid and acetone*. Hibbert. See XX.

### PATENTS.

*Retorts for the destructive distillation of sawdust, shavings, small wood, peat, beans, nuts, and like carbonaceous substances; Mechanical* —. Blair, Campbell, and McLean, Ltd., J. K. Ross, and E. P. Corner, Glasgow. Eng. Pat. 118,960, Dec. 13, 1917. (Appl. No. 18,478 of 1917.)

A VERTICAL retort is provided with a series of superposed trays and with a central rotating vertical shaft carrying horizontal arms, which revolve over the trays. The material to be carbonised is fed in at the top and travels round each tray towards a slot through which it passes to the next lower tray, the residue being discharged at the bottom. An annular space is formed around the shaft at each tray for the passage of the distillation products. Successive arms in each set are arranged at different heights. In an alternative form of apparatus the trays are rotated and the arms remain stationary.—T. F. E. R.

*Coal; Apparatus for distillation of* —. J. L. Grallin, Astoria, N.Y. U.S. Pat. 1,274,033, July 30, 1918. Date of appl., Nov. 20, 1917.

A VERTICAL retort is heated by hot gases from a furnace, which pass through zig-zag flues surrounding it. The retort extends below the lowest part of the flue, so that direct heating of this part is avoided. The base of the retort carries a false bottom within the cooler zone, and means are provided for drawing off liquid from the space between the false bottom and the bottom.—W. F. F.

*Calcium acetate and by-products; Process and apparatus for making* —. J. Gendreau, Shanghai, China. U.S. Pat. 1,276,643, Aug. 20, 1918. Date of appl., May 3, 1918.

WOOD distillate containing methyl alcohol, acetic acid, and tarry material less volatile than acetic acid, is caused to flow down an inclined conduit and is heated by a heating fluid out of contact



with it. The tarry matter passes out at the bottom, and the volatilised alcohol and acid pass into the lower end of an inclined absorbing conduit mounted on the vaporising conduit. Milk of lime flows down through the absorbing conduit, and the vapour passes upwards and repeatedly bubbles through it by the aid of suitable baffles. The milk of lime is kept hot by the vapour and by contact with the vaporising conduit, and the alcohol, which is not absorbed, passes out by a pipe at the top to a condenser. The acetate solution is collected at the bottom.—W. F. F.

*Electrode for arc lamps.* G. M. Little, Pittsburgh, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,274,725, Aug. 6, 1918. Date of appl., Aug. 6, 1915.

AN electrode for arc lamps is formed of transverse layers of dissimilar materials arranged alternately in the form of rings disposed upon a central support, so as to provide a constant supply of slag-forming material at the arc, and retard the transference of heat therefrom.—B. N.

### III.—TAR AND TAR PRODUCTS.

*Coal tar industry; Methods of analysis used in the —. I. Crude tars.* J. M. Weiss. J. Ind. Eng. Chem., 1918, 10, 732—738.

A LIST is given of methods suitable for the analysis of crude tars; many of these methods are already widely used. Particular attention is given to sampling, and four methods, applicable to specific cases, are described. Water is determined by distillation with naphtha. The tar is dried for use in subsequent determinations by distilling it up to 170° C., separating any oils in the distillate from the water, and returning these oils to the tar. The dry tar is used for the determination of sp. gr., free carbon (insoluble in benzene), fixed carbon, ash, and viscosity (Engler viscometer). Sulphur is determined by ignition with a mixture of magnesium and sodium carbonate and oxidation to sulphate.—W. P. S.

*Phenol; Synthetic —.* A. G. Peterkin. Amer. Inst. Chem. Eng., June, 1918. Chem. and Met. Eng., 1918, 19, 255—260.

THE method of Dennis and Bull (U.S. Pats. 1,211,923, 1,212,612, 1,227,894, 1,228,414, 1,229,593, and 1,247,499; this J., 1917, 208, 382, 866; 1918, 52A) for the sulphonation of benzene is founded on the discovery of the former that, although the solubility of pure sulphonic acid in benzene is negligible, benzene can dissolve from a mixture of sulphuric and sulphonic acids between 2 and 3% of its volume of the sulphonic acid. A process has been devised in which sulphonation and extraction of the sulphonic acid proceed simultaneously. Benzene at 60° C. is passed continuously through a series of four "extractors" containing solutions of sulphuric and sulphonic acids in varying proportions. When all the sulphonic acid has been removed by the benzene from the first extractor, the residual acid, consisting of 77% sulphuric acid, is withdrawn and the acid charges of the remaining extractors are dropped to the next of the series, the last extractor being charged with 98% sulphuric acid. On leaving the last of the extractors, the benzene, which contains about 2% of sulphonic acid, passes on to a series of three washers in which the sulphonic acid is dissolved out by means of water. The washers work on the same principle as the extractors, i.e., as soon as the first of the series contains sufficient sulphonic acid in solution,

the liquid is removed to an evaporator and the remaining charges are moved on to the next washer of the series, the last being filled with water. In the evaporator, the small portion of benzene remaining in solution is removed and the solution neutralised, either with the sodium carbonate solution from the phenol blowers or with solid sodium sulphite. The sodium benzenesulphonate obtained contains from 5 to 6% of sodium sulphate. The benzene from the washers passes through a separator in which any sulphonic acid solution mechanically carried over settles out; it then passes through heaters and back to the extractors. The process has many advantages over the old discontinuous method of sulphonation: initial cost of plant is reduced to one-half; labour is considerably reduced; a better yield is obtained; the formation of disulphonic acid and of sulphone is negligible; the quantity of material required is considerably less and thus a great reduction of tonnage to be transported is effected. Another improvement in the manufacture of synthetic phenol has been effected by the adoption of the so-called "liquid fusion" in which the sodium benzenesulphonate is introduced as a saturated solution into liquid caustic soda at a point above the reaction temperature. For neutralisation of the sodium phenolate, the use of carbon dioxide generated from limestone is recommended as being the most economical method.—L. A. C.

*Determining benzene, etc., in gases.* Davis. See IIA.

*Absorbing oils from gases.* Davis. See IIA.

*Benzol from petroleum.* Rodano. See IIA.

*Bitumens.* Dupré. See IIA.

#### PATENTS.

*Hydrocarbons [benzol]; Process of purifying crude —.* J. E. Thompson, Mayville, Wis. U.S. Pat. 1,276,284, Aug. 20, 1918. Date of appl., July 25, 1917.

CRUDE benzol containing carbon bisulphide is mixed with aqueous ammonia solution of about the same, but not greater, sp. gr. than the benzol. The impurities which combine with the ammonia are allowed to subside, and the supernatant purified benzol is drawn off.—C. A. M.

*Pitch burners.* Eng. Pat. 118,890. See IIA.

*Aromatic hydrocarbons.* U.S. Pat. 1,276,219. See IIA.

*Paint.* U.S. Pat. 1,275,778. See XIII.

*1-Hydroxy and 1,8-dihydroxyanthranols.* Ger. Pats. 296,091, 301,452, and 305,886. See XX.

### IV.—COLOURING MATTERS AND DYES.

*Indigo; Practical observations on the determination of —.* W. Heinisch. Färber-Zeit., 1918, 29, 183—184, 194—197.

THE best technical method for the determination of indigotin is by titration with permanganate. The results vary according to the dilution at which the titration is made and the oxidation cannot be expressed in terms of a simple chemical equation;

nevertheless the titration can be sharply standardised, and hitherto it has been usual to employ a highly purified sample of indigotin for this purpose. Experiments show that constant titration values are obtained at any dilution exceeding 1 part of indigotin to 18,000 of water. The author recommends a dilution of 1:25,000 as a standard for all titrations. As regards the course of the oxidation reaction, it has always been assumed that 1 mol. of indigotin takes up 2 atoms of oxygen to form isatin, but careful experiments by the author show that the decolorisation is complete at a point equivalent to exactly 90% of this amount of oxygen, i.e., that 10 mols. of indigotin are oxidised by 18 atoms of oxygen instead of by 20. It is therefore possible to carry out the analysis of indigo without having recourse to a pure standard sample, by using permanganate standardised in any known manner, for instance against ferrous ammonium sulphate, potassium tetroxalate, or crystallised oxalic acid. The following procedure is prescribed: 0.3–0.5 gm. of the finely powdered sample is warmed at 70°–80° C. with 10 c.c. of concentrated sulphuric acid in the presence of 50 grms. of garnets for half an hour and the solution is made up to 500 c.c. Twenty-five c.c. of the solution is measured out into each of two beakers and diluted with 300 c.c. of water. Both are then incompletely titrated with permanganate (100 c.c.=0.539 gm. of ferrous ammonium sulphate=0.1 gm. of indigotin) until only a faint green colour is perceptible; 0.1 c.c. of permanganate is then added to beaker A which, when compared against a white ground with beaker B, will show a slightly paler green; 0.2 c.c. is next added to beaker B, so that it now shows the paler tint. Then after bringing A to the same tint by the addition of 2 drops of permanganate, 1 drop more in either will completely discharge the colour of the indigotin. This method of comparing tints is the only one by which a definite end point can be accurately judged in the presence of the residual reddish colour of most samples of natural indigo.—J. F. B.

*Logwood extracts; Analysis of* — G. Savini. *Annali Chim. Appl.*, 1918, 10, 26–32.

*Water*: 5 grms. of the extract is heated for 6 hours at 105° C. in a platinum basin 10 cm. in diameter. *Ash*: The residue from the determination of the water is heated for about 1 hour in an oven above 105° C., the temperature being gradually raised to 170°–180° C. The incineration is then completed in a muffle, care being taken to avoid fusion of the ash. *Sugars*: 40 grms. of fluid extract or 0.2 gm. of dry extract is dissolved in about 100 c.c. of boiling water, 50 c.c. of basic lead acetate solution added, with vigorous shaking, and the liquid rapidly cooled, and made up to 200 c.c., with an additional 15 to 20 c.c. of water to compensate for the volume of insoluble substances (20 c.c. for extracts containing less than 10% of sugar, and 15 c.c. for those containing more than 10%). The flask is shaken, the liquid filtered rapidly, and 100 c.c. of the filtrate treated with a concentrated solution of sodium sulphate and phosphate to precipitate the lead, made up to 150 c.c., and filtered. Sucrose is determined in the filtrate by Clerget's method, and reducing sugars by the usual method. *Detection of molasses*: Pure logwood extracts yield a relatively small amount of ash (about 2%), about a quarter of which consists of potassium carbonate, whilst molasses contains about 6 to 7% of potassium carbonate. The amount of potassium carbonate may be rapidly determined by titrating the alkalinity of the soluble ash with N/10 sulphuric acid, the results being in close agreement with those obtained by gravimetric determination of the potassium. In the case of adulterated samples the amount of potassium carbonate should

correspond with the amount of sucrose in the extract, taking 50% as the average quantity of sucrose in molasses. In doubtful cases, such as extracts adulterated with tannin extract and containing sulphurous acid, the potassium should also be determined gravimetrically.—C. A. M.

*Colouring matter as indicator.* Marini. See XXIII.

#### PATENTS.

*Colouring matter [Vandyke brown]; Manufacture of organic — and of pigments therefrom.* W. A. Allsebrook, Derby. Eng. Pat. 118,735, Nov. 7, 1917. (Appl. No. 16,246 of 1917.)

ABOUT equal parts of finely powdered leather waste and 33% caustic soda lye are ground together for about half-an-hour until the product becomes solid and is soluble in water; the reaction takes place more readily if the mixture is heated not above 70° C. A pigment is prepared from the soluble colouring matter by mixing the solution with a substratum such as silica and precipitating with aluminium sulphate. If calcium sulphate be used as substratum no aluminium salt is required. If the leather waste be contaminated with cellulose waste, an alkaline oxidising agent such as a hypochlorite or peroxide is added to the caustic soda solution.—J. F. B.

*Dyes [with soap]; Universal — and method of producing same.* C. C. Huffman, Assignor to Sunbeam Chemical Co., Chicago, Ill. U.S. Pats. (A) 1,274,046, (B) 1,274,047, (C) 1,274,048, (D) 1,274,049, July 30, 1918. Date of appl., Sep. 24, 1917.

(A) A UNIVERSAL alkali-proof blue dye is prepared by saponifying a vegetable oil with a quantity of sodium hydroxide solution (sp. gr. 1.40) in excess of that required for saponification, and adding a solution of Methylene Blue to the mixture just before saponification is complete. (B) A yellow dye is prepared in a similar manner by the addition of a solution of Chrysoidine to the soap stock. (C) A brown dye is prepared by adding separately to the soap stock solutions of dyestuffs in the following proportions: 7.5 parts of Aniline Acid Red, 7.5 parts of Chrysoidine Yellow, 5 of Direct Black, and 0.5 of Methylene Blue. The reaction following the addition of each colour must be substantially complete before the next is added. (D) A red dye is prepared by adding a solution of Aniline Acid Red to the soap stock just before complete saponification and then raising the temperature of the mixture to 122° F. (49° C.).—J. F. B.

*Naphthol [sulphur] dye and process of making same.* C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,274,351, July 30, 1918. Date of appl., Jan. 2, 1917.

$\beta$ -NAPHTHOL or a mixture of naphthol and dinitrophenol is fused with sulphur and alkali or an alkali sulphide between 160° and 180° C. for about 5 hours to produce a black or dark-coloured dye-stuff.—J. F. B.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Silk; Degumming of — without soap.* E. Ristenpart. *Färber-Zeit.*, 1918, 29, 181–182.

According to a process of Voigt and Buschblütter (Ger. Pat. 291,159; this J., 1916, 833) silk may be degummed by saturating it with a solution of an



alkali salt of a weak acid, *e.g.*, sodium carbonate, and steaming. The addition of 0.5 gm. of Monopol soap per litre is an advantage, and after steaming, especially in the case of yellow silks, the goods are rinsed out in cold water and preferably washed in a hot soap bath to remove the residues of gum and colouring matter. The soap bath may be used repeatedly and afterwards employed for dyeing purposes. Experiments made by this process to determine the most favourable concentration of alkali showed that the degumming action of the soda depends on the proportion of soda to silk and also on the concentration of the soda. For any given concentration of soda solution a certain minimum quantity of alkali must be present to effect complete degumming and the presence of an excess has no useful result. With decreasing concentrations of soda the minimum quantity required for degumming also decreases. Thus, for a concentration of 1% of sodium carbonate, the minimum quantity of soda required lies between 1.64 and 2.35% of the weight of the silk; for 0.5% concentration the quantity required is between 0.85 and 1.63%; and for 0.2% concentration it is between 0.54 and 1.0%. On the large scale centrifuges are used, and the quantity of soda required depends on the quantity of soda solution left in the goods after centrifuging. If the silk is centrifuged so as to retain twice its weight of solution, the concentration of the latter should be 1%; if centrifuged to retain 2.5 times its weight of liquid, the concentration should be 0.5%; and if it retains 3 times, the concentration should be 0.2%. The above variations are explained by the increased hydrolytic dissociation of the carbonate ions into bicarbonate and hydroxyl ions, which takes place with decreasing concentration. The extension of the principle is limited by the danger of uneven saturation when larger quantities of liquid are left in the goods. Since only the necessary quantity of soda is used to combine with the sericin, there is no danger of the fibroin being attacked and all traces of alkali are removed in the washing.—J. F. B.

*Artificial silks; The cross-sections of —.* De Chardonnet. *Comptes rend.*, 1918, 167, 489–491.

The cross-sectional contours of artificial silks are a characteristic indication of the physical conditions of spinning and therefore a useful guide in the identification of origin. The first basis of classification is the manner of coagulation, *viz.*, coagulation by evaporation in air and coagulation by precipitating liquids. Other things being equal, threads formed by precipitation tend to be neither so strong nor so transparent as those formed by evaporation, since precipitation implies discontinuity. As a rule the silks formed by precipitation show a more or less circular contour in the cross-section; those produced by drying show peculiarities which influence their industrial application. Collodion silks are produced by the Chardonnet process in both ways. When spinning by coagulation in water, a very fluid collodion is employed containing, for instance, 50 grms. of pyroxylin per litre of alcohol-ether. When the fluid thread emerges in a cylindrical form from the spinning orifice its surface is immediately coagulated on contact with the water, and the film so formed, although very thin, preserves its integrity and, during the drying of the thread, it is folded up on itself as the cross-sectional area is reduced to correspond with the final solid volume. The cross-sections of threads spun under such conditions show re-entrant angles and have generally a stellate contour. If a thicker collodion be spun in the same way, the ratio between the original volume and the final volume of the thread is not so large and the irregularities of the cross-section

are less pronounced. With a little practice, the concentration of the collodion may be estimated by an examination of the sections. When spinning into air, very much thicker collodions are employed, *e.g.*, 200–250 grms. of hydrated pyroxylin, containing about 26% of water, per litre of solvent, and, on the one hand, coagulation takes place far less rapidly, while on the other, the final solid has a larger sectional area. Hence it follows that the contour is much less wrinkled and the sections have a flattened curved shape. The “covering power” of the silk increases in inverse ratio to its apparent density and, other things being equal, silks with round and regular contours cover less space. In this respect the collodion silks, particularly those spun in water, possess an advantage; but other considerations have favoured the process of spinning in air, and the superior covering power of these threads is sufficient to compensate for the slight difference in real density as compared with natural silk. Laboratory samples of extreme fineness having perfectly circular cross-sections may be prepared from a solution of 2–3 grms. of pyroxylin in 60 grms. of alcohol and 40 of ether by spinning into dilute alcohol. The alcohol retards the drying of the surface of the thread, so that the centre dries as fast as the periphery and the cylindrical form is preserved.—J. F. B.

*Cellulose of cereal straw; Nature of the —.* II. E. Heuser and A. Haug. *Z. angew. Chem.*, 1918, 31, 166–168, 172–176. (See also this J., 1918, 365 A.)

The crude cellulose prepared by the chlorination method with the use of caustic soda (*loc. cit.*) contained only 0.35% of ash, whereas that prepared with the use of sodium sulphite contained 1.1%. The yield was 54.60% of crude cellulose with furfural value 13.30%, equivalent to 22.34% of xylan. Hence the calculated yield of true cellulose was 42.97%. The original straw had furfural value 15.4, equivalent to 25.62% of xylan on the dry and ash-free basis; thus 47.32% of the original xylan remained in the cellulose. The proportion of xylan remaining in the cellulose varies inversely as the yield of cellulose and is a function of the concentration of the caustic soda solution used for extracting the chlorinated products. For instance the furfural value of 13.3 was found when a 1% solution of caustic soda was employed; with a 2% solution the furfural value of the crude cellulose fell to 10.4, and with a 3% solution to 9.3. The furfural value of commercial straw cellulose also varies with the yield and with the severity of the chemical treatment; it may reach 18%. Most of the xylan may be removed from the crude straw cellulose preparations by repeated extraction with 6% caustic soda solution, but it has not been found possible to reduce the furfural value of the cellulose below 1.95% in this manner. Attempts to remove the whole of the xylan by extraction before chlorination as well as afterwards led to a similar result, and a fully extracted preparation from commercial bleached straw pulp still gave 2.02% of furfural. Commercial bleached straw cellulose shows a “copper value” of 3.0; unbleached straw cellulose on the other hand has a “copper value” of 0.94–0.99, and bleached straw cellulose which has been fully extracted until the furfural value is reduced to the limit of 2.0% shows a “copper value” of only 0.61–0.78. Moreover, by further bleaching and the production of oxycellulose, the “copper value” of this product may be increased to 15.5 without any effect on its furfural value. Hence it is concluded that straw cellulose does not correspond to a special type of “natural oxycellulose” but is an ordinary cellulose similar to that of cotton or wood, strongly contaminated with

a pentosan and modified by bleaching under industrial conditions in such a way that the commercial pulp contains a substantial amount of oxycellulose. The only outstanding question is the nature of the residual 2% of furfural which cannot be eliminated by extraction of the purified cellulose. On hydrolysis with 1% sulphuric acid at 135° C. for half-an-hour, this furfural-yielding residue is divided half in the hydrolysed liquid and half in the hydro-cellulose. An examination of the liquid and the preparation of the benzoate and osazone, m.p. 160°—180° C., suggested the presence of xylose, and it is probable that the residue in question consists merely of a trace of xylan, equivalent to less than 1% of furfural, which is obstinately retained by the cellulose, whilst 1.0—1.5% of furfural may be attributed to the cellulose itself, just as in the case of cotton cellulose. Hydrolysis with 72% sulphuric acid followed by digestion of the diluted liquid at 120° C. for 2 hours, according to the method of Ost and Wilkening (this J., 1910, 688), was carried out on the purified straw cellulose. The results were compared with those obtained with pure dextrose, observations being made of cupric-reducing power, polarisation, yield of alcohol by fermentation, and the m.p. of the osazone. These were all in close agreement and afforded satisfactory evidence that the resolution of straw cellulose to dextrose is practically complete and that its constitution corresponds with that of cotton cellulose.—J. F. B.

*Alcohol from sisal waste.* See XVIII.

#### PATENTS.

*Waste products [textiles from oil cloth]; Process of recovering* —. J. M. Moness, Long Island City, N.Y. U.S. Pat. 1,274,184, July 30, 1918. Date of appl., Mar. 31, 1917.

THE textile base of materials such as oil-cloth is recovered by digesting the cloth with a cold 1 to 5% solution of caustic soda. The textile fabric so obtained is washed and bleached.—C. A. K.

*Peat; Process and apparatus for treating* —. C. Bouillon, Paris. Eng. Pat. 118,993, Mar. 15, 1918. (Appl. No. 4622 of 1918.)

PEAT is fed from a hopper into a machine with rollers in which it is milled and crumbled, whence it passes on to a draining transporter which conducts it to a tank containing a solution capable of coagulating blood, e.g., a dilute solution of calcium or ferric chloride. The mixture is then led into a rotary sieve capable of retaining the vegetable fibres which are collected by the aid of water and a "launder." Several of such rotary sieves with wires of different meshes may be used in succession. The impurities are then separated out in a settling tank in which a movement may be given to the liquid by the aid of a piston moving in a cylinder near to the tank, which is provided with small partitions forming compartments. Afterwards the peat is washed free from the calcium chloride and dried.—J. F. B.

*Cellulose, particularly wood cellulose; Process of producing soda* —. Aktiebolaget Cellulosa, Stockholm. Eng. Pat. 116,288, May 27, 1918. (Appl. No. 8750 of 1918.) Under Int. Conv., June 1, 1917.

WOOD is digested with caustic soda without the use of sodium sulphide by carrying out the process in the presence of a contact substance favouring reduction. Mercury is especially suitable for this purpose. The digestion liquor employed is weaker

than that commonly used and should contain about 60 grms. of Na<sub>2</sub>O as caustic soda per litre. To maintain this dilution and provide sufficient alkali for the digestion, the wood may either be boiled with about double the usual quantity of lye or a portion of strong caustic lye may be added later in the process when the initial portion is exhausted. The principal extraction of the incrusting substances should take place at a temperature not exceeding 170° C. and the process may be accelerated by the application of air pressure. The action of the lye may be moderated by the addition of a certain proportion of waste lye, preferably freed from lignin by precipitation with carbon dioxide.—J. F. B.

*Celluloid; Manufacture of non-inflammable* —. H. Dreyfus, London. Eng. Pat. 118,891, Sep. 14, 1917. (Appl. No. 13,208 of 1917.)

NON-INFLAMMABLE celluloid masses, particularly those having a basis of cellulose acetate, are subjected in the course of manufacture to pressures of 300 kilos. per sq. cm. and upwards, which is at least double the pressure usually employed. The resistance of the material to weather, chemicals, etc., is considerably increased.—J. F. B.

*Dentifrice [from cellulose hydrate] and method of making the same.* W. F. Spies, New York, and H. D. Pease, Richmond Hill, N.Y., Assignors to L. V. Skight, New York. U.S. Pat. 1,275,779, Aug. 13, 1918. Date of appl., Dec. 20, 1917.

A NON-ABRASIVE, hygroscopic dentifrice powder with a basis of finely divided non-structural cellulose hydrate is prepared by changing viscose into a gelatinous mass, e.g., by treating viscose solution with an acid salt, such as sodium bisulphate, drying and pulverising the precipitate, washing the powder until neutral or slightly alkaline, and then drying and impregnating it with xyleneol or other antiseptic.—C. A. M.

*Cellulose acetate; Preparation of* —. G. J. Esselen, jun., Swampscott, and H. S. Mork, Brookline, Assignors to Chemical Products Co., Cambridge, Mass. U.S. Pat. 1,275,884, Aug. 13, 1918. Date of appl., Mar. 6, 1918.

CELLULOSE is acetylated without destroying its fibrous form, the excess of acetic anhydride is separated from the acetylated product, the fibrous cellulose acetate is dissolved and further treated in an acetic acid solution containing a catalyst until the desired degree of solubility is obtained, and the acetone-soluble variety of cellulose acetate is precipitated in granular form.—J. F. B.

*Paper; Device for testing the tearing strength of* —. R. O. Wood, Brookline, Assignor to A. D. Little, Inc., Boston, Mass. U.S. Pat. 1,273,972, July 30, 1918. Date of appl., May 5, 1917.

THE sheet of fibrous material is held firmly along a straight-edge, and means are provided for moving a lever connected with the gripping device up an inclined plane at an angle to the base-plane of the straight-edge, the force required to move the lever so as to tear the sheet being indicated upon a pressure gauge.—C. A. M.

*Paper article; Reinforced* —. Phonograph record and method of making same. P. Poetschke, Milford, Del. U.S. Pats. (A) 1,274,323 and (a) 1,274,324, July 30, 1918. Dates of appl., (A) Nov. 5 and (B) Nov. 12, 1917.

(A) SHEETS of paper, perforated and non-perforated, are combined to form a laminated mass with an associated body of mineral cementitious material



consisting of magnesium oxide and silic reduced to a plastic condition by magnesium chloride; the outer surface may be coated with a waxy, resinous, water-repellent substance. (B) A sound record is made by building a body of layers of fibre sheets with a cement of magnesium oxychloride and a filler, subjecting the body to pressure, allowing it to harden, and applying a record-receiving facing.

—J. F. B.

[Paper] machine; Fourdrinier —. J. Alpine, Stevens Point, Wis. U.S. Pat. 1,275,826, Aug. 13, 1918. Date of appl., June 2, 1917.

THE frame of the machine is carried on a platform pivoted on a fixed bed at the receiving end and adjustably connected with the bed at other points so that the further end may be raised or lowered. The frame carries a breast roll at the pivoted end, a couch roll at the end distant from the pivoted end, and a set of suction-boxes between the two rolls. Another suction-box is supported by the platform beyond the couch roll and a take-off roll is similarly supported between the couch roll and the last suction-box, a take-off conveyor being arranged to travel over the take-off roll and the suction-box associated with it. Means are provided for adjusting the inclination of the platform and frame by lowering or raising the couch roll end and for maintaining the tension of the take-off conveyor in various positions of platform adjustment. The take-off roll swings with the platform and the conveyor passes over rolls which are in fixed relation to the bed.—J. F. B.

Paper-pulp strainers. The Watford Engineering Works, Ltd., and J. Paramor, Watford. Eng. Pat. 118,981, Feb. 7, 1918. (Appl. No. 2234 of 1918.)

Sizing composition [for paper]; Absorbent material —. E. Fues, Hanau, Germany. U.S. Pat. 1,276,206, Aug. 20, 1918. Date of appl., June 15, 1914.

SEE Fr. Pat. 473,405 and Eng. Pat. 19,816 of 1914; this J., 1915, 546, 1049.

Glucose. Eng. Pat. 118,651. See XVII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

Soluble starch for laundry purposes. O. Reinke. Chem.-Zeit., 1918, 42, 422.

SINCE rice starch is no longer obtainable in Germany, potato starch which has been rendered soluble is suggested as a substitute for starching collars, etc.—W. P. S.

### PATENTS.

Dyeing machine. H. M. Dudley, Philadelphia, Pa. U.S. Pat. 1,275,878, Aug. 13, 1918. Date of appl., Aug. 3, 1917.

WITHIN a receptacle having non-perforated sides are two foraminous plates which can be maintained at any position of separation from each other so as to form a fibre chamber of any desired size; two outer foraminous plates are spaced respectively from the inner ones. The top of the receptacle is revolvable so as to allow access to the interior. Two series of curved tubular members open into the upper and the lower part of the receptacle respectively. There are also provided a

liquid supply tank, a pipe leading from above the upper inner plate to the liquid reserve tank and one leading from below the lower inner plate to the same tank and a pump, whereby a liquid may be pumped in opposite directions through the curved members, foraminous plates, the fibre chamber, and the reserve tank. A small testing device of similar construction is connected externally with the circulating system.—J. F. B.

Silk and other textile goods in hanks; Apparatus for treating — by means of liquids. A. W. Schmid, Basle, Switzerland. U.S. Pat. 1,277,136, Aug. 27, 1918. Date of appl., Oct. 12, 1917.

THE patent relates to mechanical means for raising, lowering, and rotating the hank support and for holding it in its raised position.

Copper compounds of substantive ortho[hydr]-oryazo dyestuffs; Process for producing on the fibre —. E. Anderwert, H. Fritzsche, and H. Schobel, Assignors to Soc. of Chem. Ind. in Basle, Switzerland. U.S. Pat. 1,276,307, Aug. 20, 1918. Date of appl., Nov. 4, 1915.

SEE Eng. Pat. 12,932 of 1915; this J., 1917, 382.

[Aniline] Black; Production of — upon textile fibres. E. A. Fourneaux, Manchester. U.S. Pat. 1,277,056, Aug. 27, 1918. Date of appl., Aug. 9, 1917.

SEE Eng. Pat. 116,562 of 1917; this J., 1918, 463 a.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

Sulphuric acid manufacture; Large-scale —. A. M. Fairlie. Chem. and Met. Eng., 1918, 19, 404—408.

SULPHURIC acid is manufactured by the Tennessee Copper Co. by the chamber process, from the waste fumes of copper blast furnaces. There are two plants, one of two units and one of one unit, each unit having a daily capacity of 300 tons of acid of 60° B. (sp. gr. 1.71). The flue leading from the blast furnaces to the Glover towers has a sectional area of 200 sq. ft., and a length of 200 ft. to No. 1 plant, and 500 ft. to No. 2 plant. The waste gases contain 3—6% SO<sub>2</sub> and vary in temperature between 600° and 900° F. (315°—480° C.) at the entrance to the Glover towers. To avoid the stoppage of print due to the partial blocking up of the flue by deposits of nitre caused by leaking or overflowing of the nitre-pots, a system of external fire-heated retorts is used. These retorts, 10 ft. in height and 8 ft. in diam., take 1000 lb. of nitre and are fed automatically with sodium nitrate and sulphuric acid in the correct proportions. The Glover towers are octagonal in shape and are 55 ft. high and 30 ft. in diam. The lead curtains are lined with "chemical brick," laid in acid-proof cement. The chamber acid is concentrated to 60° B. (sp. gr. 1.71). No. 1 plant is provided with 34 lead chambers, 67—77 ft. high, of a total capacity of 3,944,800 cub. ft. No. 2 plant has four large chambers, 40 ft. high, with a total capacity of 2,227,840 cub. ft. The chambers are fitted with the usual thermometers and hydrometers, and the process is also regulated by the special analytical method of chamber control devised by the author (this J., 1917, 196). The Gay-Lussac towers of No. 1 plant have lead curtains lined with acid-proof brick walls. They are eleven in number, and are of various dimensions. No. 2 plant has only one Gay-Lussac tower, and

this is without lead curtains. It is built of acid-proof brick laid in acid-proof cement, and is divided into 56 compartments provided with a special system of acid feed pipes. No advantage has been obtained by the use of this type of tower. Large lead-antimony blowers are used to supplement the furnace blast pressure in forcing the gases through the chambers. Blow-cases constructed of riveted wrought iron are used for elevating the acid. Coolers of special design have been devised to deal with the large quantities of acid from the Glover towers. Atomised water is used in the chambers instead of steam, an electrically driven atomiser, capable of atomising 50 galls. of water per hour, being employed.

—S. S. A.

*Nitric acid manufacture in the United States; Development in — since 1914.* E. J. Pranke. Chem. and Met. Eng., 1918, 19, 395—396.

It is computed that the production of nitric acid (calculated as 100% acid) from sodium nitrate has increased from 89,000 tons to 650,000 tons since 1914, and that in the spring of 1919 the ammonia-oxidation plants now under construction will be producing at the rate of 225,000 tons of 100% acid per annum. This gives a total of 875,000 tons of 100% acid, or about nine times the pre-war production. The erection of many new nitric acid plants using sodium nitrate has provided opportunity for improvements to be made. The installation of modern fire-boxes in place of Dutch ovens under the retorts has effected a reduction in coal consumption of 25%. Acid-proof iron has been largely used to replace chemical stoneware, and a larger absorption tower capacity has been provided. The average charge of nitrate has been increased 50%. These improvements, together with modifications in the method of operation, have effected an increase in the amount of nitrogen recovered as nitric acid from about 78—80% to 92—94%. Progress is recorded in the recovery of nitrous gases from nitration units. In the ammonia-oxidation process, the catalyst used in several of the new plants is a single fine platinum gauze, with an area of about two square feet, electrically heated. Another type of catalyst, adopted by the Government experimental plants at Sheffield, Alabama, consists of several layers of platinum gauze, welded together at points, and rolled into the form of a cylinder. This is not electrically heated, the heat of reaction maintaining the necessary temperature after the oxidation has been started by external heating. No figures are available for comparison of the efficiencies of these catalysts. The cost of production of concentrated nitric acid from sodium nitrate and from autoclave ammonia gas is about the same, but ammonia gas is a cheaper form of nitrogen than sodium nitrate, so that the total cost is less with ammonia. This difference in the cost of the production of the acid rather than considerations relating to plant will probably be the decisive factor in determining whether in the future the ammonia-oxidation process will replace the older process.

—S. S. A.

*Oxalic acid; Titration of — by the conductance method.* H. S. Harned and C. N. Laird. J. Amer. Chem. Soc., 1918, 40, 1213—1218.

If the electrical conductivity of a solution of oxalic acid is measured at successive stages in the addition of a CO<sub>2</sub>-free sodium hydroxide solution, there is found to be a sharp change in the direction of the curve, which is obtained by plotting conductivity against the quantity of added alkali, when the formation of the acid oxalate is just complete. The change in the direction of the curve

at this point varies with the dilution and becomes less distinct with increasing concentration. When the oxalic acid is dissolved in 50 c.c. of water, and titrated with N/10 sodium hydroxide, the conductance method gives a sharp end point for quantiles of oxalic acid up to 0.3 gram. By the use of N/2 sodium hydroxide, quantiles not exceeding 0.8 gram. may be satisfactorily titrated. If the sodium hydroxide is not free from carbonate, the method yields high results. It is probable that the method is applicable to other dibasic acids comparable in strength with oxalic acid and may be used in the titration of mixtures of the acids and acid salts with sodium hydroxide or in the titration of mixtures of the acid salts and neutral salts with hydrochloric acid.—H. M. D.

*Potash from kelp; Extraction of —.* C. A. Higgins. Chem. and Met. Eng., 1918, 19, 432—433.

The extraction of potash from seaweed by incineration is very costly, and will probably continue only as long as the present high price of potash is maintained. Even under modern methods, the cost of harvesting alone, in Californian coastal waters, amounts to about \$1.10 (4s. 7d.) per ton of raw kelp containing 1.3% K<sub>2</sub>O; which is equivalent to about \$85 (say £18) per short ton of K<sub>2</sub>O, without the cost of drying and incineration. In a process operated successfully by the Hercules Powder Co., San Diego, Cal., since 1915, raw kelp is fermented with the production of acetic acid. Fermentation is allowed to proceed until the greater part of the kelp leaf dissolves, when the insoluble residue is removed by screening, and the solution is then neutralised with limestone. The liquid is concentrated and three products are obtained at various stages, the first consisting mainly of calcium acetate and potassium chloride, the second of the calcium salts of acetic, propionic, butyric, and valeric acids, and the third containing the bulk of the potassium iodide. The first product is distilled to recover acetone and acetone oils, and the residue lixiviated for potassium chloride; the second is treated with alcohol and sulphuric acid for the production of ethyl esters which are afterwards separated by fractionation; and the third is treated with chlorine for the production of iodine which is subsequently recovered by sublimation. The residual unfermented leaves are treated with sodium carbonate to extract, in the form of a soluble salt, algin which is afterwards precipitated and purified. Algin may find extensive uses in the future as a substitute for gelatin and vegetable gums. Probably the total production of all the harvesting concerns in present operation (in the U.S.A.) does not exceed 25 tons of 80% potassium chloride per day; and of this more than half is recovered in the form of high-grade salt (95% KCl) by the fermentation process.—W. E. F. P.

*Potash recovery; The Cottrell process for —.* L. Bradley. Chem. and Met. Eng., 1918, 19, 457—460.

The recovery of potash as a by-product in the iron and cement industries is briefly reviewed, special reference being made to the iron ores of Alabama as potential sources of potash, and to the use of potash-bearing silicates, such as feldspar and the Cambrian slates of Georgia, in the manufacture of Portland cement.—W. E. F. P.

*Gas works; Analysis of spent purifying materials from —.* Wentzel. Z. angew. Chem., 1918, 31, 176.

The author criticises the agreement recently made between the Deutsche Gold und Silberscheidanstalt and the German gas works (this J., 1918,



465 A). He contends that a quantity of 25 grms. is far too little to afford a good average sample for the determination of moisture. The absolute moisture content of the material cannot be accurately determined on account of the decomposition of the ferrocyanide, and many large gas works base their results on the air-dried material. The method proposed for the determination of crude sulphur is criticised on account of the large inaccuracies of the volumetric methods of measurement adopted and the high volatility of the solvent (carbon bisulphide) employed. On the other hand, the Drehschmidt method is said to be satisfactory and accurate, being based on gravimetric principles; differences up to 6% of sulphur have been found by the two methods and always settled in favour of the Drehschmidt method. The oxidation by fuming nitric acid presents no difficulties and with 0.5 gm. of substance may be completed within 4 hours with a consumption of not more than 30 c.c. of acid. In the new method about 240 c.c. of carbon bisulphide is consumed per analysis, whereas in the Drehschmidt method about 50 c.c. is used and immediately recovered by distillation.

—J. F. B.

*Metastannic acid and its compounds.* A. Kleinschmidt. *Monatsh. Chem.*, 1918, **39**, 149—178.

Nitric acid diluted with  $1\frac{1}{2}$ —2 volumes of water dissolves tin, yielding stannous nitrate, and at  $0^{\circ}$ — $15^{\circ}$  C. the formation of this substance can be detected even using undiluted acid, but it is then accompanied by the formation of normal or basic stannic nitrate; the yellowish white precipitate obtained between  $0^{\circ}$  and  $15^{\circ}$  C. is a mixture of a white stannic nitrate soluble in water with an insoluble yellowish-white basic stannic nitrate. Stannic nitrate at the ordinary temperature slowly changes into metastannic nitrate, the transformation being more rapid at higher temperatures. The powdery oxidation product of tin with hot nitric acid is not metastannic acid but the nitrate of this substance,  $5\text{SnO}_2 \cdot 2\text{HNO}_3 \cdot 3\text{H}_2\text{O}$ , which undergoes hydrolysis with water yielding metastannic acid; the nitrate obtained with cold acid has the composition  $3\text{SnO}_2 \cdot 2\text{HNO}_3 \cdot \text{H}_2\text{O}$  and is probably a mixture or compound of stannic nitrate  $\text{SnO}(\text{NO}_3)_2$  with metastannic nitrate,  $5\text{SnO}_2 \cdot 2\text{HNO}_3 \cdot 3\text{H}_2\text{O}$ , the latter of which is also obtainable by the action of nitric acid on metastannic acid. Metastannic acid and hydrochloric acid react yielding a stannyl chloride  $5\text{SnO}_2 \cdot 2\text{HCl} \cdot 3\text{H}_2\text{O}$ , which with sodium hydroxide gives a sodium metastannate  $5\text{SnO}_2 \cdot \text{Na}_2\text{O} \cdot 4\text{H}_2\text{O}$ . It is suggested that metastannic acid has the formula  $\text{Sn}_5\text{O}_5(\text{OH})_{10}$ , the above nitrate and chloride being represented respectively as  $\text{Sn}_5\text{O}_5(\text{OH})_4(\text{NO}_3)_6$  and  $\text{Sn}_5\text{O}_5(\text{OH})_4\text{Cl}_2$ . (See also *J. Chem. Soc.*, Nov., 1918.)—D. F. T.

*Rare earths. VII. Separation of holmium.* L. F. Yntema and B. S. Hopkins. *J. Amer. Chem. Soc.*, 1918, **40**, 1163—1167.

ATTEMPTS to separate holmium from yttrium have been made by fractional hydrolysis of the phthalates. The mixed hydroxides were added gradually to phthalic acid suspended in water, the acid being kept in excess to prevent the formation of an insoluble basic compound. The proportion of phthalic acid in the resulting solution was adjusted so that hydrolysis began between  $50^{\circ}$  and  $60^{\circ}$  C., and by raising the temperature stepwise, a series of fractions of basic phthalate was obtained in which the ratio of holmium to yttrium gradually diminished. By means of four series of such hydrolyses, the holmium content of a mixture was increased from 12% to 25.4%. Fractional precipitation by the addition of sodium nitrite to a neutral nitrate solution was also tried and in a

series of such precipitations in which ten fractions were obtained, it was found that the first fraction contained 12.5% of holmium as compared with about 4.8% in the original rare earth mixture. The nitrite method is considered the better of the two methods of separation, not only in respect of efficiency, but also of the cost of the materials used and the relative simplicity of the operations involved.—H. M. D.

*Potash from cement dust.* Dean. See IX.

*Sodamide and potassamide.* Wöhler and Stang-Lund. See XI.

*Fertiliser.* Hüttner. See XVI.

*Acetic acid and acetone.* Hibbert. See XX.

*Double rubidium- and cesium-metal chlorides.* Vermande. See XXIII.

#### PATENTS.

*Sulphuric acid; Process of and apparatus for concentrating* —. H. G. Klink, Moundsville, W. Va. U.S. Pat. 1,276,377, Aug. 20, 1918. Date of appl., Mar. 23, 1917.

THE weak acid is caused to pass from the top to the bottom of a tower packed with inert material, through the interstices of which a counter-current of hot air is maintained. Means for collecting the concentrated acid are provided at the lower end of the tower, and the upper end communicates with a condenser through which the gaseous current is passed. The liquid from the condenser is concentrated in a series of evaporating pans and then discharged into the top of the tower.

—W. E. F. P.

*Alumina; Purified crystalline — and method of making the same.* F. J. Tone, Assignor to The Carborundum Co., Niagara Falls. U.S. Pat. 1,276,134, Aug. 20, 1918. Date of appl., Jan. 5, 1918.

A MIXTURE of aluminous material, containing between 0.1 and 1% of titanium oxide, and carbon is fused, separated from the reduced impurities, and a solid oxidising agent such as iron oxide is added and the mixture re-fused so as to oxidise the carbon and carbides present.—A. B. S.

*Magnesium chloride; Process and apparatus for preparing [anhydrous]* —. C. Dantszen, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,276,499, Aug. 20, 1918. Date of appl., Apr. 22, 1916.

ANHYDROUS magnesium-ammonium chloride is charged into a bath of magnesium chloride heated electrically to above  $300^{\circ}$  C. in a closed chamber. This communicates with a second chamber in which the vaporised ammonium chloride is condensed; portions of the fused magnesium chloride are removed from the bath at intervals.—W. E. F. P.

*Gases [air]; Method of treating [liquefying]* —. A. C. Morrison, Essex Fells, N.J. U.S. Pat. 1,273,929, July 30, 1918. Date of appl., Apr. 4, 1914.

IN a method of liquefying a paramagnetic gas at a temperature slightly above the normal boiling point, the gas is subjected to the action of a powerful magnetic field. In the treatment of air and

other oxygen mixtures, separation is effected by causing the gas to flow at high velocity through a spiral tube located within the magnetic field, the constituents being removed separately from the outer and inner portions of the tube.—W. E. F. P.

*Spraying liquids.* U.S. Pat. 1,273,856. See I.

*Removing suspended particles.* U.S. Pat. 1,276,644. See I.

*Hydrogen.* U.S. Pat. 1,276,487. See IIa.

*Calcium acetate* U.S. Pat. 1,276,643. See IIb.

*Aluminium nitride.* U.S. Pat. 1,274,797. See XI.

*Manganese depolariser.* U.S. Pat. 1,275,666. See XI.

## VIII.—GLASS; CERAMICS.

*Alumina; Influence of — on the fusibility of glasses.* L. Springer. *Keram. Rundsch.*, 1917, 48, 243. *J. Soc. Glass Tech.*, 1918, 2, Abs., 88—89.

THE author's experiments were similar to those of Singer (this J., 1918, 582 A), except that the mixtures were heated in small crucibles in a glass furnace and the progress of fusion followed by withdrawing samples at frequent intervals. It was found that in a high-alkali glass or in a normal glass of the standard type  $0.5\text{Na}_2\text{O}$ ,  $0.5\text{CaO}$ ,  $3\text{SiO}_2$ , addition of alumina, even in small quantities, decreased the fusibility and retarded the firing. In glasses high in lime and low in alkali, the fusibility was increased and the firing accelerated by addition of alumina, the effect, within limits, being in proportion to the amount of alumina added. It was immaterial whether the alumina was added as kaolin or as hydrated alumina. Inconclusive results in regard to fusibility were obtained when pegmatite was used as source of alumina, but the glasses made with pegmatite fired more rapidly in every case than those without pegmatite. The effect of alumina on fusibility appears to depend on many factors, and particularly on the composition of the glass, and no definite rule can be formulated. The total heat consumed in heating the batch materials to the maximum temperature of the melting furnace, in decomposing the sulphates and carbonates present in the batch, in melting the silicates produced, and in dissolving the excess silica is about twice as great in the case of a soda-ash batch and three times as great with a salt-cake batch as the heat liberated by chemical combination between the acidic and basic oxides of the batch. If, therefore, part of the lime and alkalis is introduced already combined as silicates, as is the case when pegmatite is used, less heat is required in the furnace to melt the glass. The heat balances for four glass batches are calculated in support of this contention.

*Glass; Polishing — by means of acids.* O. Parkert. *Sprechsaal*, 1918, 51, 162—163.

WHEN acids are used for polishing glass instead of the customary mechanical methods with abrasive materials, the irregularities in the surface are removed by solution of the glass. Hydrofluoric acid is chiefly used, but other acids (such as sulphuric acid) may be added for subsidiary purposes such as the solution of calcium fluoride formed by the action of the hydrofluoric acid. A solution suitable for

polishing Bohemian lead glass consists of water 1 part, sulphuric acid 2 parts, and hydrofluoric acid 1 part; for other glasses the most suitable solution should be found by trial. The solution should be heated to  $40^{\circ}$ – $50^{\circ}$  C. and maintained at that temperature. The glass is cleaned by immersion for 45 seconds in a mixture of sulphuric acid 12 parts, hydrochloric acid 1 part, and water 100 parts, and then in water at  $35^{\circ}$  C. It is then immersed for 30 seconds in the polishing solution and is afterwards washed thoroughly. If one treatment is insufficient it may be repeated. Fine glass ware may receive a final polish with the aid of tin ash. The glass ware may be etched by covering it with a stencil and exposing it to the gas evolved from the polishing solution. If the glass is not polished after 30 seconds' immersion, more hydrofluoric acid should be added to the solution. Good ventilation should be provided. This method of polishing is applicable to all kinds of glasses except the very finest ware; it is cheap and requires little skill.

—A. B. S.

*Glasses and glazes; Phosphoric acid —.* H. Fritz. *Chem.-Zeit.*, 1918, 42, 422.

GLASSES and glazes were prepared from mixtures of lead oxide, alkalis, lime, barium oxide or zinc oxide, and phosphoric anhydride or a phosphate. When a phosphate was used the products were not clear, but when phosphoric anhydride was used they were mostly clear and occasionally brilliant. In some cases the introduction of 0.15 mol. of  $\text{Al}_2\text{O}_3$  made a turbid glass or glaze clear. Most of the glazes prepared with phosphoric anhydride were subject to "crazing." Brilliant glazes free from crazing were obtained, however, with a mixture of the composition  $(0.1\text{K}_2\text{O}, 0.1\text{CaO}, 0.8\text{PbO}), 0.2\text{Al}_2\text{O}_3, 2\text{P}_2\text{O}_5$ .

—W. P. S.

*Chemical stoneware.* A. Malinowsky. *Amer. Inst. Chem. Eng.*, June, 1918. *Chem. and Met. Eng.*, 1918, 19, 485—487.

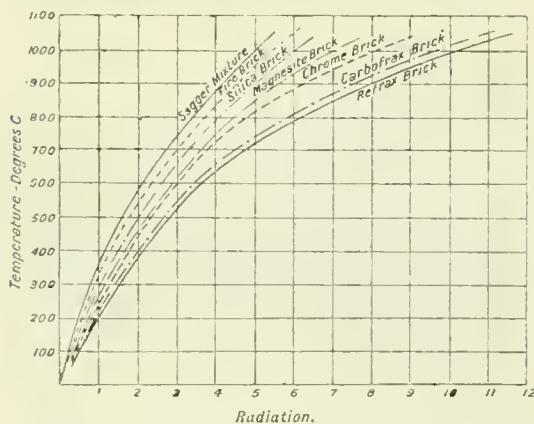
CHEMICAL stoneware is made of a mixture of several clays, selected to produce a body of porcelain-like character. Alternatively, a low-grade fireclay may be used either alone (when the ware should be burned at  $1400^{\circ}$ – $1500^{\circ}$  C.) or in admixture with an impure clay of very low fusing point, or other cheap fluxes such as lime or furnace slag. The mixture should be high in silica but plastic enough to permit the shaping of large articles. It should have a range of  $200^{\circ}$ – $300^{\circ}$  C. between complete devitrification and deformation. Uniformity of composition is essential and is best secured by mixing and blunging the materials with water, screening to separate the coarser particles, and then removing the surplus water in a filter-press. If the coarse particles are not removed, spigots and faucets made of the material will leak. To ascertain the solubility of chemical stoneware, weighed vessels may be filled with concentrated sulphuric acid and heated on a sand-bath at about  $120^{\circ}$  C. for 12 weeks, the acid evaporated being replaced by fresh acid. The vessels are then allowed to cool, washed with water, dried at  $140^{\circ}$  C., cooled, washed, again dried at  $140^{\circ}$  C., allowed to cool in a desiccator for 24 hours, and the loss in weight noted. In two samples tested the losses were 0.074 and 0.098% respectively. Another method consists in grinding a piece of the stoneware until it passes completely through a No. 16 sieve. The material is then screened through a No. 20 sieve and about 100 of the largest pieces left on the sieve are weighed, washed with distilled water, dried at  $110^{\circ}$  C., cooled in a desiccator and weighed. They are transferred to a platinum crucible, a mixture of concentrated nitric acid 10 parts, concentrated sulphuric acid 25 parts and water 65 parts added, boiled for about 4 hours until



all the sulphur trioxide fumes have been driven off, cooled, dilute nitric acid added and again boiled for 10 minutes. The insoluble matter is washed free from acid, dried at 110° C. for five hours, cooled in a desiccator, and the loss in weight noted. In a sample thus tested the loss was 0.56%. Microscopical examination of good chemical stoneware shows that it has a character similar to lava, and that it consists of a refractory material united by a glassy mass. The texture is sufficiently fine for a high polish to be obtained, so that spigots and stoppers may be ground to fit tightly. The chief minerals present are quartz, orthoclase, plagioclase, magnetite, and altered "ferro-magnesia mineral." The quartz grains are surrounded by a thin glassy film, but are otherwise unaltered. The felspar grains are much altered externally and gradually pass into the ground mass. The heat appears to have fractured the grains and to have fused the materials along the cracks. The original "ferro-magnesia" has been chiefly converted into magnetite and a glassy mass. The rate of cooling the ware after burning is important; too rapid cooling will produce brittle ware and too slow cooling will cause too much crystalline matter to be formed. The ware may be cooled rapidly to the freezing temperature of the eutectic (800°—1000° C.), but the subsequent cooling should be very slow.—A. B. S.

**Carborundum refractories.** S. C. Linbarger. Chem. and Met. Eng., 1918, 19, 489—491.

CARBORUNDUM is an important ingredient in refractory materials as it increases their thermal conductivity. It has a low coefficient of expansion, great strength and toughness, and a high specific heat, thermal conductivity, and emissivity at the



temperatures at which crucibles, saggars, and other articles made from it are used. The heat-emissivity curves of various refractories are shown in the accompanying diagram and other properties in the following table:—

Material	Specific heat	Thermal conductivity	Compressive strength, lb. persq. in.
Firebrick . . .	0.192	0.0034	1,050
Sagger mixture...	0.187	0.0033	1,340
Magnesite . . .	0.220	0.0071	4,800
Chrome . . .	0.174	0.0067	3,900
Refrax . . .	0.162	0.0275	12,500
Carbofrax . . .	0.180	0.0243	14,700
Silica . . .	0.191	0.0020	2,300

The form of carborundum most suitable for ceramic purposes ("carbofrax") is made by bonding graded crystallized silicon carbide with a small proportion of refractory clays to secure a dense body of low porosity with about seven times the heat-conducting power of a sagger mixture and

twelve times that of a silica brick, and a load-carrying capacity over ten times as great as that of sagger mixtures, without the same tendency to soften at high temperatures. Hence, saggars made of carborundum need only be one-tenth of the present thickness and so would be lighter and heated much more rapidly than the present ones. Carborundum refractories will also withstand the most sudden changes in temperature and are not readily attacked by the fused ash from coal. "Refrax" and "silfrax" are special products prepared by the action of vapours containing silicon on carbon or silicon carbide in an electric furnace, the final product being pure silicon carbide. Firesand or amorphous carborundum is a mixture of various silico-carbides forming the exterior of the core produced in the manufacture of carborundum. It is highly refractory and although less stable than carborundum at a high temperature has a wide application when bonded with fireclay or water-glass and made into bricks, etc., or applied as a plastic coating to protect furnace linings from the cutting action of impinging flames.—A. B. S.

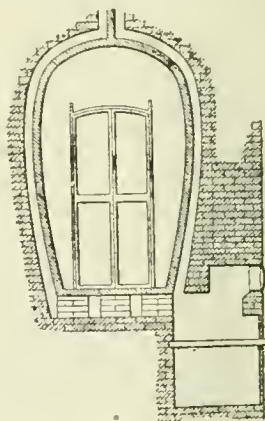
#### PATENTS.

**Glass-furnace.** H. E. De Vaughn, Assignor to W. A. Jones, Morgantown, W. Va. U.S. Pat. 1,274,146, July 30, 1918. Date of appl., Oct. 6, 1916.

A skew block supporting the cap of a glass-furnace has a channel in its under side and a recess leading upward therefrom. Within the recess is a hollow flat supporting device, preferably of metal, which is suspended by bars or other means which extend upwards through passages in the block. The suspension bars are enclosed in tubes through which and through the hollow supporting device a cooling medium is circulated.—A. B. S.

**Poreclain kiln.** W. Mellor, Sebring, Ohio. U.S. Pat. 1,275,285, Aug. 13, 1918. Date of appl., Mar. 30, 1918.

An up-draught muffle oven has several fireboxes on one side, below the sole, connected to lateral sub-



sole flues and to vertical flues which lead to separate chimneys—one for each side of the oven.—A. B. S.

**Kiln.** C. H. Zwermann, Kalamazoo, Mich. U.S. Pat. 1,275,352, Aug. 13, 1918. Date of appl., June 8, 1918.

Two coal-fired tunnel kilns are placed side by side, with the inlet end of one near to the exit end of the other, the intervening wall being relatively thin and provided with a longitudinal flue. Along the outer sides of the tunnel are perforated bag walls,

the bottom of the bags communicating with the flue in the inner wall. Heating flues lead from the top of this outlet flue over the tops of the tunnels, downwardly at the outer sides towards the inlet ends of the tunnels to heat the preheating zone in each tunnel. Discharging and vertical circulating flues, open at top and bottom, for the preheating zones are placed at the outer sides of the tunnels and on opposite sides of the common wall, so as to create a cooling circulation near the exit end of each tunnel and a heating circulation near the charging end.—A. B. S.

*Compartment kiln.* R. H. McElroy, Assignor to The International Clay Machinery Co., Dayton, Ohio. U.S. Pat. 1,275,712, Aug. 13, 1918. Date of appl., Sep. 1, 1917.

EACH chamber in a gas-fired continuous kiln of the transverse arch type is provided with a transverse bag wall along one side, a feather-wall forming a connecting flue, and a perforated floor, portions of the floor adjacent to the bag wall being non-perforated. The connecting flues and the perforations in the floor all communicate with the main flue.

—A. B. S.

*Refractory material.* J. M. Allen, Assignor to Fellhauer-Allen Manufacturing Co., St. Louis, Mo. U.S. Pat. 1,275,354, Aug. 13, 1918. Date of appl., Nov. 10, 1917.

A REFRACTORY material consisting of 48½% of fireclay, 48½% of anthracite coal, and 3% of galena.—A. B. S.

*Clay; Process of hydrating* — J. S. Laird, Ann Arbor, Mich. U.S. Pat. 1,275,705, Aug. 13, 1918. Date of appl., Dec. 24, 1917.

CALCINED clayey material is hydrated and its plasticity is increased by subjecting it to the action of steam under pressure at a temperature above 100° C. whilst working the mass.—A. B. S.

*Crystalline alumina.* U.S. Pat. 1,276,134. See VII.

## IX.—BUILDING MATERIALS.

*Potash from cement dust; Wet process for extracting* — J. G. Dean. Chem. and Met. Eng., 1918, 19, 439—447.

At the works of the South-western Portland Cement Co., Victorville, California, 1 ton of potassium sulphate per day is now being extracted from cement-kiln dust by a process in which the hot kiln gases are saturated with water vapour and then cooled to the lowest permissible temperature before discharge, so that the dust is precipitated in the form of a sludge containing potassium salts in solution. The gases are saturated with water vapour in a spray chamber, and cooled in a condenser-evaporator designed to utilise the heat of the gases for concentrating the weak solutions obtained. Owing to imperfect atomisation of the oil fuel employed, the atmosphere within the kiln is reducing; and the dusty gases contain sulphides, polysulphides, and thiosulphates, in addition to sulphates, of potassium and sodium. The sulphides are converted into sulphates, thiosulphates, and carbonates by reaction with carbon dioxide and water in the spray chamber; and the carbonates into sulphates by passing the filtered liquid through a bed of gypsum, which latter is prevented from dissolving in the alkali thiosulphate by the presence of an excess of carbon dioxide. By evaporating

the solution to crystallisation, a product consisting of potassium sulphate in admixture with a hydrous double thiosulphate of potassium and sodium is obtained which, on being dried and heated to decompose part of the thiosulphate, yields a marketable product containing about 90% of potassium sulphate. The formation of the double thiosulphate may be prevented, and pure potassium sulphate crystallised out from the boiling solutions, by the addition of sufficient sodium sulphate to the liquid at the appropriate stage. By reason of the comparatively low temperature, due to the reducing conditions obtaining within the kiln, only about 45% of the total potash in the charge is volatilised. Oxidising conditions, although producing a higher kiln temperature, would greatly increase the difficulty of handling the solutions and render the separation of the potassium and sodium salts practically impossible. At present the operating cost is nearly covered by the value of the returned dust. Details are given of the general design and operation of the plant.—W. E. F. P.

*Plaster; Importance of the valuation of* — E. Link. Tonind.-Zelt., 1918, 42, 525—526.

HEAVILY adulterated plasters and some plaster substitutes are now on the market in Germany. One of the former examined by the author was found to consist of plaster of Paris 38.59%, calcium carbonate 20.18%, hydrated lime 39.45%, magnesia 0.45%, and insoluble residue 1.12%. It appears to have been made by mixing plaster of Paris with dry slaked lime, the latter having absorbed carbon dioxide from the air. Its composition explains its peculiar behaviour when sprinkled into water, the low strength of the plaster, and its softness when set.—A. B. S.

*Potash recovery.* Bradley. See VII.

## PATENTS.

*Bricks, slabs, columns, and the like; Composition, and manufacture of — and structures erected therewith.* A. T. O. Quick, British Expeditionary Force. Eng. Pat. 119,152, Dec. 1, 1917. (Appl. No. 17,783 of 1917.)

BRICKS, slabs, columns, or the like are made from a mixture of 4 parts of powdered chalk and 1 part of Portland or like cement by gauging with water and allowing to set in moulds. For rapid construction the bricks are made with open-ended passages and when assembled are fixed in position by cement introduced into the passages so as to form keys.

*Bricks; Stepped — for the walls of buildings and acid plants.* W. H. Kenyon, Haslingden, and W. Smith, Accrington. Eng. Pat. 119,087, Sep. 20, 1917. (Appl. No. 13,519 of 1917.)

*Lagging.* Eng. Pat. 118,871. See I.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Selenium and tellurium; Recovery of — in copper refining.* M. H. Merriss and H. T. Binder. Eng. and Min. J., 1918, 106, 442—445.

In the electrolytic refining of crude copper bullion the selenium and tellurium present are concentrated in the anode slimes, from which they are recovered in various ways. (1) The button of noble metals obtained by cupellation of the slimes with lead is fused with sodium carbonate and nitrate; the



"nitre slag," which contains the bulk of the selenium and tellurium, is extracted with hot water, giving a solution of selenite and tellurite. (2) The flue system of the cupellation furnace is equipped with a scrubber or Cottrell apparatus. In both cases it is necessary to saturate the gases with water, and the water, after being circulated repeatedly, forms a cheap source of selenium. The wet sludge from the scrubber is filter-pressed. (3) The flue-dust, or the filter-pressed sludge from (2) is roasted at a low red heat; crystals of selenium dioxide are obtained by cooling the roaster gases in chambers. (4) The flue dust, instead of being roasted, may be extracted with water, selenium dioxide dissolving. The residue is treated with sulphuric acid and bleaching powder, whereby the elementary selenium is oxidised and dissolved. The alkaline solution obtained under (1), on being acidified with sulphuric acid, yields a precipitate of tellurous acid. The acid filtrate, or the solutions obtained under (2) or (4), are reduced with sulphur dioxide to precipitate the selenium. The latter is washed free from acid and digested with boiling water to transform it into the black modification. The dried tellurous acid precipitate is reduced with lampblack or ground charcoal in a crucible furnace and cast into cake or stick form. Alternatively, the tellurous acid may be dissolved in 4 parts of strong hydrochloric acid and the solution precipitated with sulphur dioxide, the solution being diluted but not to such a degree as to re-precipitate tellurous oxide.—W. R. S.

*Zinc; Analysis of commercial* —. L. Bertiaux. Ann. Chim. Analyt., 1918, 23, 161–169, 181–191.

LEAD is determined electrolytically as dioxide, after the sample has been dissolved in a mixture of copper nitrate solution and nitric acid. To determine iron, the metal is dissolved in sulphuric acid, and the ferrous salt titrated with permanganate solution; this gives the iron approximately, but for more accurate work, the iron may be determined colorimetrically in a portion of the titrated solution by means of thiocyanate. Tin is not often present; it may be determined in the usual way as metastannic acid. Antimony is separated from the tin by the oxalic acid method. Copper and cadmium are determined electrolytically, the latter from an alcoholic alkaline cyanide solution. Arsenic is separated by distillation and titrated with iodine solution; the residue from the arsenic distillation may also be used for the determination of the antimony. Sulphur is evolved as hydrogen sulphide which is absorbed in ammoniacal silver nitrate solution, and carbon is determined by oxidation with chromic acid and sulphuric acid, the resulting carbon dioxide being absorbed in potassium hydroxide and weighed.—W. P. S.

*Zinc slags.* O. Mühlhaeuser. Metall u. Erz, 1918, 15, 303–305.

THE amount of slag formed in the retorts during distillation should be small, and its composition should be controlled by mixing the charge in suitable proportions so that it has as little action on the retort as possible. An average slag sample obtained by distilling a mixture of Joplin and Wisconsin ores was found to have an oxygen ratio of 1:2.8, approaching that of a trisilicate. It was very viscous at distillation temperatures and formed an internal coating, the thickest part of which was on the bottom of the retort. The latter was thus protected from corrosion, and the loss of zinc by vapour diffusion decreased.—W. R. S.

*Tin; Crystal structure of* —. A. J. Byl and N. H. Kolkmeier. Chem. Weekblad, 1918, 15, 1077–1078. An application of the Röntgen ray method to the determination of the crystal structure of tin. —A. J. W.

*Flotation; Effect of addition agents in* —. M. H. Thornberry and H. T. Mann. Bull. School of Mines and Met., Univ. of Missouri, Nov., 1917. 48 pages.

A FULLER account of work the chief results of which have been published previously (see this J., 1918, 93 A, 153 A).

*Potash recovery.* Bradley. See VII.

#### PATENTS.

*Chromium; Depositing — upon metals, particularly iron, chemically.* E. E. Burnett, Hayes Town, Middlesex. Eng. Pat. 118,182, Oct. 4, 1917. (Appl. No. 14,336 of 1917.)

IRON or steel may be rendered rust-proof by subjecting the cleaned and grease-free metal to the action of a heated solution of chromium phosphate, preferably containing an excess of phosphoric acid. The solution may be prepared by the action of a 1 to 2% solution of phosphoric acid on chromite or chrome ore.—C. A. K.

*Steel; Method of converting iron ores directly into* —. H. A. Greaves and H. Etchells, Sheffield. Eng. Pat. 118,647, June 12, 1917. (Appl. No. 8366 of 1917.)

A ROTARY inclined tube furnace is arranged in combination with an electric smelting furnace so that the tube furnace discharges into a hopper or shoot, which in turn discharges through the crown of the electric furnace. Connections between the various parts are flexibly luted to provide gas-tight joints, whilst allowing the ordinary manipulation of the furnace. The electric furnace is first charged with ore mixed with powdered carbon and the charge melted, the gases given off during this process passing through powdered or granulated ore in the tube furnace in which partial reduction of the ore takes place. The ore thus enters the electric furnace in a heated and partially reduced condition and powdered carbon is added to effect full reduction, after which refining operations are completed. Owing to the intermittent operation of one furnace it is advisable that two or more refining furnaces should be fed from one tube furnace.—C. A. K.

*Case-hardening.* A. E. Bamfield, Bristol. Eng. Pat. 118,983, Feb. 18, 1918. (Appl. No. 2823 of 1918.)

TO prevent the localised penetration of carbon during case-hardening of steel articles, the cleaned steel is coated with a plastic paste composed of 50% of sodium silicate, 25% of china clay, 18½% of amorphous silica, and 6½% of fine silver sand. An impervious coating is formed during the heating. The paste may be similarly applied to prevent decarburisation during annealing.—C. A. K.

*Pig-iron; Method of producing* —. F. H. Crookard, Birmingham, Ala. U.S. Pat. 1,274,245, July 30, 1918. Date of appl., Apr. 2, 1917.

PIG-IRON low in phosphorus is obtained by smelting steel scrap with suitable quantities of coke and fluxes in a blast furnace.—W. R. S.

*Ferro-silicide; Manufacture of shaped castings of* —. N. G. Pelliot, Assignor to United States Alloys Corporation, New York. U.S. Pat. 1,274,360, July 30, 1918. Date of appl., Dec. 19, 1917.

Ferrous and siliceous materials are heated in an electric furnace with carbonaceous material in such proportions as to obtain ferrosilicon containing 10–16% Si. The molten alloy is poured into a shaped mould, or first into a mixer until sufficient material has been obtained for the casting.

—W. R. S.

*Iron; Process of electrolytically producing* —. A. T. C. Estelle, Hagen, Germany, Assignor to M. F. Wijkstrom, Stockholm, Sweden. U.S. Pat. 1,275,161, Aug. 6, 1918. Date of appl., Nov. 22, 1916.

IRON is produced electrolytically by treating compounds, such as oxides, of iron with caustic alkali or alkali carbonate at high temperatures, and subjecting a slime of the caustic alkali solution and the iron compounds to electrolysis. By mixing with the iron compounds, compounds of other metals, such as cadmium, which can be precipitated under the prescribed conditions, iron alloys may be obtained.—B. N.

*Etching steel or other like plates; Method of* —. W. S. Eaton, Sag Harbor, N.Y. U.S. Pat. 1,275,408, Aug. 13, 1918. Date of appl., Nov. 28, 1916.

THE plate, with some parts protected by a resist, is submerged in an electrolytic bath composed of a chloride solution of such strength that it has little or no corrosive action on the metal. The plate forms the anode in the electrolytic cell, and the voltage of the current used is below the decomposition voltage of the electrolyte, but sufficiently high to dissociate the chlorine from the solution, the etching being done by the gas in a nascent state.

—B. N.

*Steel; Process for hardening* —. J. W. Gilbert, Assignor to D. H. Parker, Windsor, Vt. U.S. Pat. 1,275,242, Aug. 13, 1918. Date of appl., May 8, 1916.

HIGH-SPEED steel is heated slowly to about 700° F. (370° C.), then more rapidly to 1500° F. (815° C.), and at a still more rapid rate to a maximum of 2000°–2500° F. (1090°–1370° C.). The steel is withdrawn and cooled in a metal bath to between 700° and 1500° F. (370°–815° C.), and finally cooled in air to below 200° F. (93° C.).—C. A. K.

*Armour-plates; Method of and apparatus for casting* —. L. Gathmann, Washington, D.C.; O. G. Foley, administratrix. U.S. Pat. 1,276,517, Aug. 20, 1918. Date of appl., Feb. 15, 1916.

MOLTEN metal cast in a mould is caused to cool more regularly by passing a cooling medium horizontally through the lower portion of the metal, and a heating medium through the upper portion. A metallic mould built in sections is provided with a number of horizontal hollow cores supported by the sections, by means of which the heating and cooling are effected.—C. A. K.

*Zinc; Electrolytical production of* —. G. H. Clevenger, Palo Alto, Cal., U.S.A. Eng. Pat. 115,847, May 7, 1918. (Appl. No. 7667 of 1918.) Under Int. Conv., May 7, 1917.

IN the electrodeposition of zinc from a zinc sulphate electrolyte, metals, such as cobalt, are removed

from the electrolyte by precipitation with nitroso- $\beta$ -naphthol, the latter being formed within the electrolyte by the interaction of finely-divided  $\beta$ -naphthol, an acid, and a soluble nitrite, such as sodium nitrite. The electrolyte is subsequently treated with zinc dust to decompose the nitrite or nitrate.

—B. N.

*Zinc; Process of treating* —. C. A. H. de Saulles, New York. U.S. Pat. 1,274,249, July 30, 1918. Date of appl., Nov. 3, 1915.

Zinc containing lead is first melted to allow a large proportion of the lead to separate by gravity. The zinc is then distilled, and the distillate kept in a molten condition until a large quantity has collected.—W. R. S.

*Smelting furnaces and the like*. J. Gaunt, D. Brookfield, and J. Tylor and Sons, Ltd., London. Eng. Pat. 118,750, Nov. 24, 1917. (Appl. No. 17,367 of 1917.)

A HOPPER lined with refractory material and having a hinged cover is attached to a smelting furnace so that the products of combustion pass through and preheat a charge in the hopper, afterwards escaping through a side flue. The base of the hopper is suitably shaped to deflect the hot gases into the preheater. Attachment to the furnace may be made by means of a loose grooved ring and the whole may be lifted away to give access to the furnace.

—C. A. K.

*Roasting furnaces; Mechanical* —. A. Ramen, Helsingborg, Sweden. Eng. Pat. 118,812, Apr. 12, 1918. (Appl. No. 6271 of 1918.)

THE revolving shaft of a mechanical roasting furnace contains a movable and detachable water-supply pipe for each stirring-arm; the pipe at its outlet end is connected to a hollow valve body which corresponds to a valve seat surrounding the water inlet of the circulating channel of the arm, and is adapted to be tightly pressed against the seat. The member for pressing the valve body against its seat may be situated within the furnace shaft or the stirring-arm. The valve body may be mounted on an inner lining, the interior of which communicates with the water supply; the lining has an opening communicating with an outlet in the valve body.

—W. R. S.

*Furnaces for roasting blende, pyrites, or other materials; Mechanical* —. H. C. Bingham, London. Eng. Pat. 118,940, Nov. 13, 1917. (Appl. No. 16,636 of 1917.)

IN a roasting furnace of the superimposed hearth type, the upper surfaces of the hearths are inclined, so that the flow of material under the action of the rabbles is constantly opposed by the inclination of the hearth to a greater or lesser extent according to the degree of mobility of the material treated. In a modification some of the hearths are so inclined, the others being horizontal.—C. A. K.

*Furnace; Electric* — [for sintering tungsten]. C. A. Pfanstiehl, Waukegan, Ill., Assignor to Pfanstiehl Co., Inc., North Chicago, Ill. U.S. Pat. 1,273,920, July 30, 1918. Date of appl., Nov. 19, 1914.

A FURNACE for sintering ingots of compressed metallic tungsten powder at or near its fusing temperature comprises a refractory tube forming a heating chamber, with a pair of water-cooled terminal blocks at the ends. Two parallel rods of ductile metallic tungsten of fibrous structure are interposed between the terminals, so as to form a cradle for supporting and maintaining intimate



contact with the ingot to be sintered. An atmosphere of hydrogen is maintained within the chamber during the sintering operation, and an electric current is passed from one block to the other through the cradle, so as to raise the temperature of the latter to the fusing point of metallic tungsten.—B. N.

*Alloys, and process of making the same.* F. Milliken, New York. Eng. Pat. 118,825, May 17, 1918. (Appl. No. 8289 of 1918.)

AN alloy containing Cu 60–70, Ni 6–9, Fe 4–6, Zn 12–16, Pb 2–3%, and a trace of phosphorus, is claimed to be immune to corrosion by sulphuric acid.—W. R. S.

*Alloy for resistance elements, etc.* W. B. Driver, East Orange, N.J. U.S. Pat. 1,274,250, July 30, 1918. Date of appl., Jan. 2, 1914.

A DUCTILE and malleable alloy of high specific electric resistance contains at least 1% Ti and over 20% Ni. Some examples are given in the following table:—

Ni	Cr	Ti	Fe	Mn	Resistance, microhms per cm. cube
%	%	%	%	%	
75	—	6	19	—	77
68	15	4	11	2	117
65	—	8	27	—	96
65	—	6	19	10	106
60	12	1	24	3	112
60	12	6	19	3	125
40	15	6	39	—	112
30	10	6	54	—	117
30	—	6	64	—	100
	Cu				
80	10	3	—	7	48
75	—	8	—	17	57
45	45	3	—	7	77
94	—	5	1	—	32

—W. R. S.

*Roasting ore; Process for* —. C. H. Fulton, Cleveland, Ohio, Assignor to Metallurgical Laboratories, Inc., Chicago, Ill. U.S. Pat. 1,273,844, July 30, 1918. Date of appl., Aug. 19, 1916.

FINELY divided ore is suspended in a slow current of preheated air and caused to pass into a chamber which is heated to a temperature above the ignition point of the ore. The desulphurised ore is then separated from the exit gases.—C. A. K.

*Cleaning metal articles; Method of* —. C. B. Morey and C. J. Huber, Assignors to Larkin Co., Buffalo, N.Y. U.S. Pat. 1,274,186, July 30, 1918. Date of appl., June 23, 1917.

ARTICLES of gold, silver, etc., are cleaned by immersion in a dilute acid solution of an electrolyte, the metal article being in contact with a more electro-positive metal (e.g. zinc).—C. A. K.

*Copper; Method of producing commercially pure* —. C. S. Bradley, New York. U.S. Pat. 1,275,374, Aug. 13, 1918. Date of appl., Oct. 25, 1913. Renewed Dec. 20, 1917.

A CHLORINE compound of copper (e.g. the trioxychloride  $\text{CuCl}_2 \cdot 3\text{CuO}$ ) is reduced at a red heat by means of carbon and a base (e.g. lime), the chloride of which is fusible but non-volatile at the melting point of copper. The finely divided reduced copper is melted in the fused chloride bath and separated.

—C. A. K.

*Solutions [electrolytes from copper refining]; Process of treating foul* —. F. T. Smith, Jamaica Plain, Mass. U.S. Pat. 1,276,130, Aug. 20, 1918. Date of appl., Apr. 2, 1915.

AN impure electrolyte composed of a solution of acid and salts is electrolysed in the anode chamber of an electrolytic cell containing a soluble anode, whilst an acidified solution of pure copper sulphate flows through the cathode chamber. An oxidising agent and a basic precipitant for iron are added to the anode chamber, and the solution is further treated to remove other impurities and leave only pure copper sulphate which is passed to the cathode chamber.—J. H. J.

*Metals; Extracting — from ores.* C. Biesel, El Paso, Texas. U.S. Pat. 1,276,162, Aug. 20, 1918. Date of appl., Jan. 9, 1917.

ORE is crushed in the presence of a solvent, and the solution separated, the ore being subjected to further grinding with a fresh portion of the solvent. The process is repeated in sequence as great a number of times as required, the impoverished ore being treated with new solvent in each grinding operation.—C. A. K.

*Ores; Process of concentrating* —. R. G. Hall, St. Louis, Mo. U.S. Pat. 1,276,058, Aug. 20, 1918. Date of appl., Sep. 15, 1916.

ORES containing zinc, lead, and other metals are partially roasted, and mixed with a reducing agent and a proportion of sulphur (as sulphide) sufficient to form a matte with the more valuable metals. The charge is heated in a reverberatory furnace, with volatilisation of the zinc and lead, the other metals such as copper, silver, and gold being obtained as a matte, and waste silicious materials being eliminated in the form of slag.—C. A. K.

*Steel and other metals and alloys; Apparatus for the quenching, hardening, tempering, or like heat treatment of* —. H. C. Dickson, Quinton, Eng. Pat. 118,954, Dec. 1, 1917. (Appl. No. 17,793 of 1917.)

*Iron ore; Method of treating* —. F. A. Eustis, Milton, Mass., U.S.A. Eng. Pat. 112,615, Oct. 1, 1917. (Appl. No. 14,132 of 1917.) Under Int. Conv., Jan. 12, 1917.

SEE U.S. Pat. 1,237,765 of 1917; this J., 1917, 1099.

*Steel; Method of and means for annealing [high-speed]* —. J. G. R. Munday, Kingston-upon-Hull. U.S. Pat. 1,277,244, Aug. 27, 1918. Date of appl., Aug. 6, 1917.

SEE Eng. Pat. 106,624 of 1916; this J., 1917, 886.

*Alloys.* British Thomson-Houston Co., Ltd., London. From General Electric Co., Schenectady, N.Y., U.S.A. Eng. Pat. 118,947, Nov. 20, 1917. (Appl. No. 17,095 of 1917.)

SEE U.S. Pat. 1,273,762 of 1918; this J., 1918, 591 A.

*Flux for use in brazing cast-iron and other metals.* A. Figgins, Richmond, and J. H. Nathan, St. Kilda, Victoria, Australia. U.S. Pat. 1,275,412, Aug. 13, 1918. Date of appl., Jan. 18, 1917.

SEE Eng. Pat. 112,500 of 1917; this J., 1918, 127 A.

## XI.—ELECTRO-CHEMISTRY.

*Sodamide and potassamide; Electrolysis of molten* —. L. Wöhler and F. Stang-Laud. *Z. Elektrochem.*, 1918, 24, 261—270.

MOLTEN sodamide and potassamide on electrolysis produce as anode products ammonia and nitrogen but no hydrazine as was expected. These substances melt at 210° and 338° C. respectively, these values being about 60° higher than the values given by Titherley. In the analysis of the amides it is advantageous to moisten with alcohol before adding water to produce ammonia. (See also *J. Chem. Soc.*, Nov., 1918.)—J. F. S.

*High pressures; Production of* — by electrolysis. K. Wilkens. *Chem.-Zeit.*, 1918, 42, 428.

If water is electrolysed in a completely filled vessel, the gases produced can only occupy the space of the decomposed water, that is, in 1 ampère-hour, 624 c.c. of gases must occupy a volume of 0.3354 c.c. and will be under a pressure of 1860 atmospheres. When only nine-tenths of the volume of the vessel is filled with water and the remaining one-tenth occupied by air, the pressure after electrolysis for 1 hr. (with 1 amp.) will be 7 atmospheres; after 19 hrs., 61; after 50 hrs., 269; and after 1000 hrs., 1433 atmospheres.—W. P. S.

*Insulating materials; Electrical* —. Bültemann. *Verbandsmitt. Ver. Dresdner Bez.-Ver. Ing. und Dresdner Elektrotechn. Ver.* (Reprint.) *Z. angew. Chem.*, 1918, 31, Ref., 233—234.

MARBLE or slate when used as insulating plates in electric installations must be polished and impregnated or coated with an enamel varnish. Mica has excellent insulating properties, plates of 1 mm. resisting 40,000 volts or more. Micanite, megotale, micarta, etc., are commercial products prepared from mica flakes and a binding agent such as shellac varnish, etc. Experiments made in America with wood impregnated with asphalt, linseed oil, or paraffin wax have not given satisfactory results. Vulcanite is a good insulator, and when compressed with asbestos fibre forms the so-called "vulcan asbestos." Ebonite formed by vulcanising gutta-percha is not equal to hard rubber (vulcanite) as regards its resistance to temperature. "Vulcan fibre" is a trustworthy insulator when in the dry condition. Cellon varnishes are suitable for coating electric wires, etc. The hard cellon preparations have an insulation resistance of about 10,000 megohms, and plates 1 mm. thick resist up to 30,000 volts. Good insulating powers are also shown by monite, a cellulose product of the viscose type. Other technical products of importance are the phenolic resins, "bakelite," "faturane," and "eswelite." "Faturane" has an insulating resistance of more than 100,000 megohms, and in plates 3 mm. thick resists 25,000—30,000 volts (alternating current). "Eswelite" (sp.gr. 1.2) has an insulating resistance of 8,000,000 megohms. Numerous insulating materials such as "eshalite," "ambroine," "tenacite," "agalite," "australite," "festonite," etc., are composed of asbestos with binding agents.

—C. A. M.

*Bitumens.* Dupré. See IIA.

*Titrating oxalic acid.* Harned and Laird. See VII.

## PATENTS.

*Electrodes for electric furnaces and like purposes.* W. B. Hamilton, H. A. Blackwell, and W. L. Turner. Liverpool. Eng. Pat. 118,690, Sep. 14, 1917. (Appl. No. 13,172 of 1917.)

AN electrode is formed of a hollow container of refractory material, with a tapered block of carbon at, and projecting from, one end. A metallic presser device at the other end is adapted to compress and consolidate a granular or finely-divided mass of graphite within the hollow container, which is supported and reinforced by metal rods. The body of the electrode is rigidly held from a carrier, and the metal presser device is supported in a separate carrier in which it is adjustable. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 7001 of 1905; this *J.*, 1906, 433.)—B. N.

*Electrodes; Manufacture of carbon* —. B. E. D. Kilburn, London. From Det Norske Aktieselskab for Elektrokemisk Industri Norske Industri-Hypotekbank, Christiania, Norway. Eng. Pat. 119,018, Jan. 9, 1918. (Appl. No. 9285 of 1918.)

CARBONACEOUS material is pressed or stamped around a metallic core, and is then baked, first by the heat produced by current passing through the core, and then by current passing through the carbonaceous material of the electrode itself.

—B. N.

*Depolarising material; Volume-stable manganese* —. Void-filled depolarising mass. Non-conducting manganese depolarising electrode. C. Ellis, Montclair, N.J., Assignor to National Carbon Co., Inc., Long Island City, N.Y. U.S. Pats. (A) 1,272,405, (B) 1,272,406, (C) 1,272,407, July 16, 1918. Dates of appl., (A) Oct. 23, (B and C) Oct. 18, 1915.

(A) NORMAL manganese dioxide is heated in an atmosphere of oxygen to a sintering temperature for the preparation of a depolariser for dry batteries of the Leclanché type. (B) The depolariser is in the form of a moulded mass of finely-divided psilomelane, the particles of which are of different sizes, so that the voids are substantially filled. (C) The depolarising electrode is formed of a rod-shaped carbon structure, supporting an annular compressed mass of graphite, and artificial substantially non-conducting manganese dioxide in a slight state of hydration obtained by dehydrating hydrated manganese dioxide up to the point where it becomes substantially non-conducting to electric current, i.e., when it contains 1 mol. H<sub>2</sub>O to 8 mols. MnO<sub>2</sub>.—B. N.

*Depolarising material; Preparation of higher oxide of manganese* —. C. Ellis and A. A. Wells, Montclair, N.J., Assignors to National Carbon Co., Long Island City, N.Y. U.S. Pat. 1,275,666, Aug. 13, 1918. Date of appl., Dec. 6, 1916.

THE precipitate produced by mixing solutions of sodium carbonate and a manganous salt is dried without washing and then ground and exposed to a current of air and water vapour at about 200° C. The resulting manganese dioxide is then washed and dried.—W. H. C.

*Plastic [insulating] composition and process of making the same.* J. P. A. McCoy, Wilkinsburg, Pa., Assignor to Westinghouse Electric and Manufacturing Co. U.S. Pat. 1,274,728, Aug. 6, 1918. Date of appl., July 3, 1914.

TANNINS and tannin-containing materials form efficient binding agents for the manufacture of insulating compositions. Lime, magnesia, or other compounds which form insoluble products with



tannins, may be used as fillers. The waste products of the tanning industry known as phlobaphenes or "reds" may be utilised in this way. With tannins as binding material, moreover, compact, hard moulded products may be prepared from ground cork.

(A) *Furnace; Electric* —. (B) *Furnace for chemical reactions*. (C) *Electric furnace*. (D) *Process of producing aluminium nitride*. (E) *Electric furnace*. M. Shoeld, Assignor to Armour Fertilizer Works, Chicago, Ill. U.S. Pats. (A) 1,274,794, (B) 1,274,795, (C) 1,274,796, (D) 1,274,797, (E) 1,274,798, Aug. 6, 1918. Dates of appl., (A, B, C, D) Jan. 28, 1918, (E) Mar. 6, 1918.

(A) An electric furnace comprises a heating chamber, with an electrically-conductive lining, an outer horizontal ring electrode, and a central vertical electrode, which projects down into the plane of the ring electrode. The material to be treated is fed downwards between the electrodes, and a current of gas is directed upwards through the material. The latter is simultaneously subjected to the action of the gas and the heating effect of the current, the electrodes being of greater electrical conductivity than the lining. (B) The material to be treated is placed in carriers with perforated bottoms, which are caused to slide through an inclined chamber, heated by suitable means, and having a perforated floor. Gas, which has been preheated by the treated material, is passed through the chamber and material, and the gas escaping through the perforations of the carriers and chamber bottom, is used to preheat the material before it enters the furnace. (C) An electric furnace having a conduit for the passage of the material to be treated, is provided with means for heating an intermediate portion only of the conduit. For example, one or more electrodes are arranged in contact with the external portion of the conduit, and form one or more arcs with other adjacent electrodes. (D) A nitrogen-containing gas is uniformly distributed through, and made to travel in the opposite direction to, a body of briquettes, composed of finely-divided carbon, alumina, and a binder. An intermediate zone of the mixture is simultaneously uniformly heated to a temperature of 1800° to 2000° C. by an electric current, e.g., in a furnace of the type described under (A), and the gas is preheated by the treated briquettes, whilst the hot escaping gas is employed to preheat the briquettes as they approach the heating zone. (E) An arc is formed between vertical electrodes, in a heating chamber, lined with carbon, provided with an inclined passage for conveying the material to be treated, and for passing the gas in a direction opposite to the travel of the material. The passage is "off-set" practically vertically, substantially at a point beneath the arc, so that the direction of travel of the material is changed whilst at its highest temperature.—B. N.

*Furnace; Electrical induction* —. O. C. Böckman, Notodden, Norway. U.S. Pat. 1,275,206, Aug. 13, 1918. Date of appl., May 3, 1918.

THE furnace is provided with an induction coil, and a core forming a closed path for the magnetic flux created by the coil. A pivoted member, forming an extension of the core outside the furnace, is arranged in parallel with the ordinary path of magnetic flux, and the path of magnetic flux in the extension may be closed or interrupted, with the object of regulating the magnetic flux in the main core, thus permitting regulation of the furnace temperature independent of the current and magnetic coils.—B. N.

*Electric furnace*. U.S. Pat. 1,273,920. See X.

*Etching*. U.S. Pat. 1,275,408. See X.

*Treating foul electrolytes*. U.S. Pat. 1,276,130. See X.

## XII.—FATS; OILS; WAXES.

*Saponification of citric acid esters and of glycerides; Alkaline* —. J. Pinnow. Z. Elektrochem., 1918, 24, 270–278.

COTTONSEED oil, when treated with alcoholic potash, is converted to the extent of 75% into ethyl esters, whilst a small quantity is directly hydrolysed. The glycerol content of cottonseed oil can be estimated by weighing and the use of an analytical correction. Ditate acids hydrolyse asymmetrical diethyl citrate in two ways with the production of the two monoethyl esters.—J. F. S.

*Crismar test*. Stewart. See XIXA.

### PATENTS.

*Oils, wax, rosins and materials of a like nature; Extraction of — from bone charcoal or other substances containing the same*. J. MacGregor, Glasgow, and G. Scott and Son (London), Ltd., London. Eng. Pat. 118,461, Aug. 30, 1917. (Appl. No. 12,472 of 1917.)

THE material, e.g., bone charcoal or fullers' earth, or the like, is emulsified with water, and the emulsion treated in successive baths of an immiscible solvent tighter than water. After subsidence of the solid particles the supernatant layer of solvent and dissolved oil or the like is decanted.—C. A. M.

*Fats, oils, waxes, and the like; Process of and apparatus for hydrogenating* —. J. B. Walker, Austin, Tex. U.S. Pat. 1,276,290, Aug. 20, 1918. Date of appl., Jan. 6, 1917.

LIQUEFIED fats, etc., are heated and hydrogenated in the presence of a finely divided catalyst. The escaping hydrogen is conducted through a body of coarse catalytic material, whilst simultaneously portions of the oil previously withdrawn from that already treated are sprayed on to this material and allowed to percolate through it into the main body of the oil. Apparatus for this process comprises a closed vessel divided into an upper and a lower compartment by means of a porous diaphragm of coarse catalytic material. The lower compartment contains trays adapted to support catalytic material, whilst the upper compartment contains a fluid-driven motor and a gas pump connected with a gas-circulating conduit and condenser outside the receptacle.—C. A. M.

*Hydrocarbons, their homologues and substitution products; Process of preparing stable aqueous solutions of* —. Schülke und Mayr, and H. Kantorowicz, Hamburg. Ger. Pat. 306,059, July 7, 1912.

SMALL amounts of neutral alkali salts of inorganic or organic acids (e.g., sulphites, oxalates, phosphates, sodium chloride, or potassium cyanide) are added to rosin soaps or sulphonated soaps, to increase by five or six fold their power of rendering soluble in water hydrocarbons (with the exception of halogen derivatives of hydrocarbons of the ethylene series), mineral oils, terpenes, and especially essential oils.—C. A. M.

*Fats and oils; Method of refining* —. S. Morgenstern, Leipzig, Germany. U.S. Pat. 1,275,289, Aug. 13, 1918. Date of appl., July 3, 1914.

SEE Fr. Pat. 473,761 of 1914; this J., 1915, 804.

*Hydrogenation of fats and oils*. J. Dewar, London, and A. Liebmann, Weybridge, Assignors to The Proctor and Gamble Co., Cincinnati, Ohio. U.S. Pat. 1,275,405, Aug. 13, 1918. Date of appl., June 2, 1914.

SEE Eng. Pats. 12,981 and 12,982 of 1913; this J., 1914, 797.

*Oils; Process of obtaining* —. J. Stewart, Philadelphia, Pa., Assignor to E. I. du Pont de Nemours and Co., Wilmington, Del. U.S. Pat. 1,276,822, Aug. 27, 1918. Date of appl., Oct. 27, 1915.

SEE Eng. Pat. 101,959 of 1916; this J., 1917, 557.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Resins; Constituents of* —. II. *Constituents of Sumatra gum benzoin*. H. Lieb and A. Zinke. Monatsh. Chem., 1918, 39, 219–230. (See also this J., 1918, 520 A.)

CONTRARY to the opinion of Lüdy (this J., 1893, 946) the resinol obtainable from Siamese gum benzoin is not identical with that from Sumatra gum benzoin. Whereas the former resin is only partially soluble in hot dilute sodium hydroxide solution, the latter dissolves entirely, although on boiling the solution for an hour colourless needles separate of a levo-rotatory sodium salt of a previously unisolated *l*-benzoresinol, m.pt. 330°–341° C.,  $[\alpha]_D^{25} = -12.88$ , probably of the composition  $C_{20}H_{14}O_4$ . On the addition of a little ether to the alkaline mother liquor there is obtained a deposit of the sodium salt of a dextro-rotatory resinol,  $C_{20}H_{14}O_4 \cdot H_2O$ , crystallising in needles m.pt. 298°–299° C.,  $[\alpha]_D^{25} = +51.60$ , to which the name *d*-sumaresinol is given; it is soluble in aqueous ammonia. *d*-Sumaresinol is isomeric with siaresinol. The benzoresinol described by Lüdy as obtainable from Sumatra gum benzoin was probably merely a mixture of *d*-sumaresinol with a small proportion of *l*-benzoresinol.—D. F. T.

#### PATENTS.

*Paint and method of making same*. F. W. Sperr, jun., Oakmont, Assignor to H. Koppers Co., Pittsburgh, Pa. U.S. Pat. 1,275,778, Aug. 13, 1918. Date of appl., Jan. 18, 1917.

MELTER pitch from tar containing not more than 6% of free carbon is mixed with solvent naphtha at a temperature between 100° and 200° C., the proportion of the naphtha being insufficient to produce "precipitation" of the pitch. The resulting colloidal solution dries readily to form a smooth glossy film.—C. A. M.

*Coating composition*. C. P. Townsend, Washington, D.C., Assignor to General Bakelite Co., New York. U.S. Pat. 1,273,954, July 30, 1918. Date of appl., Dec. 18, 1914.

A BASE for antifoiling paints and varnishes is made by incorporating or chemically combining a salt of copper or other toxic substance with a condensation product of phenol and formaldehyde, or other phenolic condensation product which becomes insoluble and infusible when heated.—C. A. M.

*Phenol-aldehyde condensation product*. T. S. Wennagel, Alt-Rahlstedt, Germany, Assignor to Naaml. Vennoots. Hollandsche Proteïne Maatschappij, Amsterdam, Holland. U.S. Pat. 1,273,967, July 30, 1918. Date of appl., Jan. 22, 1917.

NEUTRAL alkali salts of aromatic hydroxy-acids which are not hydrolytically dissociated are used as condensing agents in the preparation of phenol-aldehyde condensation products.—C. A. M.

*Paint and varnish; Composition for removing* —. C. McAdam, Chicago, Ill., Assignor to Chadeloid Chemical Co., New York. U.S. Pat. 1,274,430, Aug. 6, 1918. Date of appl., June 14, 1915.

CRESOL or other relatively slowly volatile hydroxy derivative of benzol, to act as a varnish-penetrating agent, is emulsified with a loosening agent (grain alcohol, acetone, celluloid, and gum camphor), and a wax (spermaceti).—C. A. M.

*Finishing, cleaning, or polishing composition and process of making same*. H. S. Blackmore, Mount Vernon, N.Y. U.S. Pat. 1,276,481, Aug. 20, 1918. Date of appl., May 20, 1918.

A COMPOSITION for polishing, finishing, etc., is prepared by mixing naphthalene tetrachloride or other halogen compound of naphthalene with a mineral hydrocarbon, and in particular with petroleum the sp. gr. of which is too high for a lamp oil and too low for lubricating purposes under ordinary atmospheric conditions.—C. A. M.

*Impregnating composition and method of making same*. W. V. Lander, Newton, Mass., Assignor to General Indurating Corporation. U.S. Pat. 1,277,322, Aug. 27, 1918. Date of appl., Dec. 20, 1916.

A COMPOSITION for impregnating porous or bibulous materials consists of a solution of petroleum residue and colophony in gasoline.

*Resins; Manufacture of* — from benzol plant residues. H. Koppers Co., Pittsburgh, Assignees of M. Darrin, Wilkinsburg, Pa., U.S.A. Eng. Pat. 112,419, Oct. 1, 1917. (Appl. No. 14,166 of 1917.) Under Int. Conv., Dec. 30, 1916.

SEE U.S. Pat. 1,236,917 of 1917; this J., 1917, 1042.

*Carbon-black*. U.S. Pat. 1,276,487. See IIA.

*Pigments*. Eng. Pat. 118,735. See IV.

*Recovering waste products*. U.S. Pat. 1,274,184. See V.

*Extracting oils, etc.* Eng. Pat. 118,461. See XII.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Rubber; Influence of opening a tapping cut and of pollarding on latex and* —. O. de Vries, Communications of the Central Rubber Stat., Buitenzorg, Java, 1918, 2, 241–255.

WHEN the tapping cut was freshly opened, the amount of latex increased during the first few days as the cut was brought to the usual depth, and reached a maximum of nearly 800 c.c. after a week, subsequently decreasing to 600 c.c. after one month and 500 c.c. after two months; the rubber content decreased from 44.0% on the first day to



35.0% after one week and attained a constant value of 25–26% after the second week, whilst the specific gravity increased from 0.9689 to 0.9920, or from 0.9895 to 0.9950 if compared after dilution to a 15% rubber content. During these changes the tensile strength of the rubber remained constant, but the rate of vulcanisation gradually increased from a period of 145 minutes to 100 minutes, the "slope" increasing from 32.5 to 37; constancy was attained in about four weeks. Pollarding at a height of 3 m. from the ground caused a sudden alteration in rubber content of latex from 26% to 21% and in sp. gr. from 0.9910 to 0.9960 for undiluted latex or 0.9946 to 0.9970 for latex diluted to 15% rubber content, constancy being attained in a few days. The alteration in the qualities of the rubber was within the usual limits of variation for plantation rubber, a slight increase in rate of vulcanisation and a possible slight reduction in tensile strength being observed. Within a month after pollarding the latex production had diminished to one-third.—D. F. T.

*Rubber; Differences in weight in the preparation of sheet and crêpe* —. O. de Vries and H. J. Hellendoorn. Communications of the Central Rubber Stat., Buitenzorg, Java, 1918, 2, 361–400.

THE serum from rubber latex, when slightly milky, does not contain rubber but only proteins and the danger of loss of rubber in milky serum is exaggerated; indeed when a small percentage of rubber is lost in the serum, the value of the rubber lost is often less than that of the additional acetic acid which would be necessary to produce a clear serum. The chief cause of difference in weight of rubber produced is the variation in the content of serum substances. Excluding matured slab rubber, the difference in the weight of crêpe rubber produced in various ways may amount to 1%, whilst for sheet rubber it may be 3%. With modern methods, involving standardised procedure such as a standard concentration of rubber in latex before coagulation, and rolling on the day after coagulation, the difference generally does not exceed  $\pm 0.5\%$ . The moisture content of rubber is higher the greater its relative weight, i.e., the greater its content of serum substances. By diluting the latex before coagulation or especially by soaking the sheets in water, the proportion of serum substances in the rubber is diminished, and the subsequent air-dried rubber shows a decreased moisture content.

—D. F. T.

[*Rubber*] *coagulum; Maturing of* —. O. de Vries. Communications of the Central Rubber Stat., Buitenzorg, Java, 1918, 2, 213–240.

THE results of Eaton and his co-workers (this J., 1916, 715, 1046; 1917, 1217) and of Campbell (this J., 1917, 604) as to the increased rate of vulcanisation of matured or "slab" rubber are confirmed, but the actual rate is found to be far from uniform for matured rubber prepared on different estates or under different conditions. The tensile strength is only slightly superior to that of sheet rubber, but the great superiority of sheet to ordinary crêpe in strength as reported by Eaton appears to be out of accord with the observations of other workers. Whether the greater strength of vulcanised matured slab rubber is an intrinsic characteristic of the rubber or is merely due to the shorter period of heating necessary for vulcanisation remains yet to be decided. The "slope" (Schidrowitz, this J., 1915, 842; Whitby, this J., 1916, 495) is smaller and therefore the resistance to stretching at greater elongations is better for slab rubber and the viscosity of the solutions is higher; on the other hand, the hardening of the coagulum and consequent difficulty in rolling, the slow dry-

ing of the crêped mass, and the frequent poor colour are disadvantages. During maturing of the coagulum the decomposition of serum substances causes a loss in weight of 2–2½%. Natural variation in the composition of the latex has a marked influence on the rate of vulcanisation of the matured rubber, and dilution of the latex also gives rise to a more slowly vulcanising rubber with a decreased "slope," but unaltered tensile strength and viscosity. If the coagulum is kept under water, the resulting rubber has approximately the same rate of vulcanisation as ordinary crêpe, whilst soaking in water after maturing for several days in the air also causes a reduction in the rate, the effect in both cases being probably due to the extraction of serum substances or decomposition products of these. If freshly rolled crêpe is kept in water for several days, it undergoes alteration comparable with the effect of maturing, possibly because the harder crêpe does not yield its serum substances so readily to the water. The addition of sodium bisulphite to latex retards the changes caused by maturing, but even with 1 grm. per litre of latex the effect is only small.—D. F. T.

*Vulcanisation without sulphur by Ostromyslenski's method*. E. Bunschoten. Kolloid. Zeits., 1918, 23, 25–31.

IN his experiments on the vulcanisation of rubber by means of such substances as mono-, di-, and trinitrobenzene, Ostromyslenski made no quantitative measurements of the physical properties of his products. In the experiments here reported the progress of vulcanisation was followed by breaking-stress and extension tests. Preliminary experiments in which raw rubber was heated with nitrobenzene and *m*-dinitrobenzene at 147° C. for 30, 60, or 90 minutes gave negative results, and even in presence of such organic accelerators as "acceleren" (*p*-nitrosodimethylaniline) and "vulcazite" (a condensation product of ammonia and acetaldehyde) no vulcanisation occurred. Different metallic oxides, however, were found to act as catalysts towards rubber and nitro- or *m*-dinitrobenzene, and in all the subsequent experiments lead oxide was used. Experiments were made on two varieties of rubber, a quick vulcanising smoked sheet with a vulcanisation coefficient of 4.5, and a slow vulcanising first latex crêpe with a coefficient of 2.6. Nitrobenzene was found to have poor vulcanising properties, *m*-dinitrobenzene giving much better results. In both cases the smoked sheet was vulcanised by the organic agent more quickly than the crêpe, just as when sulphur is used. There are exceptions, however, cases being known in which the sample which is vulcanised the more rapidly by sulphur is affected less readily by dinitrobenzene. The time factor is important, for if heating is continued beyond a certain length of time the physical properties of the sample deteriorate. Thus 100 parts of smoked sheet heated with 8 parts of lead oxide and 4 parts of *m*-dinitrobenzene at 147° C. showed the best results after 10–15 minutes. The velocity of vulcanisation is also increased by increasing the proportion of catalyst and of vulcanising agent within certain limits. Between 137° and 157° C. the velocity of the reaction increases markedly with rising temperature. The quality of the rubber vulcanised with *m*-dinitrobenzene deteriorates rapidly; after a month a sample had become quite soft, although it was still insoluble in benzene. The samples also become gradually coated with crystals of dinitrobenzene. Attempts to prepare hard rubber by prolonged heating of raw rubber with lead oxide and dinitrobenzene failed. A soft leather-like substance was obtained which gradually extruded crystals of *m*-nitraniline. Attempts were made to vulcanise rubber by heating with inorganic

oxidising agents such as arsenic acid, potassium persulphate, bleachlag powder, potassium bichromate, permanganate, chlorate, nitrate, and ferricyanide, and manganese dioxide. In each case vulcanisation appeared to commence but the products were unsatisfactory, although better than those obtained by use of mono- or dinitrobenzene without the addition of lead oxide. The addition of *m*-dinitrobenzene to a benzene solution of rubber increases the viscosity of the solution in the dark at ordinary temperatures, and more so at 65° C. Under the influence of light, however, the viscosity rapidly falls. It is concluded that the vulcanising action of dinitrobenzene is due to an acceleration of polymerisation of the rubber. Sulphur acts in the same manner, but at the same time forms a chemical or physical compound.—E. H. R.

## PATENT.

*Sheet-rubber; Manufacture of* —. W. Seward. Toronto, Canada. U.S. Pat. 1,274,091, July 30, 1918. Date of appl., Feb. 27, 1918.

A SHEET of dried pure crêpe rubber is united, by rolling, with a sheet of washed, dried, and broken down rubber, and the composite sheet is vulcanised.—C. A. M.

## XV.—LEATHER; BONE; HORN; GLUE.

*Hide-powder; Adsorption by* —. V. Kubelka. Kolloid. Zeits., 1918, 23, 57—68.

As a result of experiments on the removal of hydrogen chloride from aqueous solution by hide powder, it has been found that the hide substance retains its absorbing capacity unchanged for at least two years when the powder is preserved in a carefully stoppered bottle. The equilibrium between the hide powder and the acid solution is rapidly attained and in the case of pure hydrochloric acid solutions the quantity of acid in the hide is independent of the concentration of the acid in the aqueous solution. For acids of concentration  $N/100$  to  $N/1$ , the quantity of hydrogen chloride fixed by 1 gm. of dry hide powder is found to be 0.739 millimol. In presence of considerable quantities of sodium chloride, hydrogen chloride is removed more easily from the aqueous solution and under these conditions the amount of acid in the hide powder depends on the concentration. From solutions saturated with respect to sodium chloride, the quantity of hydrogen chloride absorbed by 1 gm. of hide powder increases from 0.773 millimol. in the case of 0.008  $N$  acid to 1.655 millimol. in the case of 0.4%  $N$  acid. The graphic representation of the actual results suggests that the phenomenon is of complex character in that the absorption curve consists of two distinct branches. The first portion of the curve appears to reach a maximum corresponding with the absorption of 1 gm.-mol. of  $HCl$  per 977 grms. of hide powder and it is supposed that this indicates the formation of a definite compound between the hide substance and the acid. The number 977 would thus represent the molecular weight of the hide collagen. The second branch of the absorption curve is of the normal adsorption type and it is inferred that the compound formed is capable of removing further quantities of hydrogen chloride from aqueous solutions containing sodium chloride in virtue of its adsorptive faculty.

—H. M. D.

*Gelatin and glue; "Jelly value" of* —. A. W. Clark and L. DuBois. J. Ind. Eng. Chem., 1918, 10, 707—708.

DEFINITE quantities of the sample (*e.g.*, 1, 2, 3, 4, etc. grms.) are placed in separate test-tubes, 10 c.c. of cold water is added to each, the mixtures are

stirred for some hours, then heated in a boiling water-bath, and afterwards cooled to 0° C., the test-tubes being corked during the cooling. The mixtures are then heated very slowly and observations are made as to whether the contents of a tube are liquid or solid. Usually, it is advisable to make the observation at 10° C. If, for instance, the 3, 4 and 5% mixtures are liquid at 10° C., whilst the 6, 7, and 8% mixtures are solid, the sample is said to have a comparative jelly strength of 6%.—W. P. S.

## PATENTS.

*Leather substitute and process for making the same.* R. B. Respass, New York. U.S. Pat. 1,276,113, Aug. 20, 1918. Date of appl., Nov. 26, 1917.

A COMPRESSED sheet of fibrous material is saturated with a binding agent consisting of rubber, balata, naphtha, fish glue, sodium silicate, sulphur, and linseed oil, then dried, and again pressed.—W. P. S.

*Proteids [fertilisers]; Recovery of — from waste liquors of the hide-treating art.* C. L. Peck, Assignor to The Dorr Co., New York. U.S. Pat. 1,274,763, Aug. 6, 1918. Date of appl., Mar. 2, 1918.

AN EFFLUENT containing proteins is separated from the waste liquors from the treatment of hides, *e.g.*, spent lime liquors, and the proteins are precipitated and preserved from putrescence by means of waste liquors from the tanning process. The resulting material may be used in the manufacture of fertilisers, etc.—C. A. M.

*Colouring matter.* Eng. Pat. 118,735. See IV.

*Plastic compositions.* U.S. Pat. 1,274,728. See XI.

## XVI.—SOILS; FERTILISERS.

*Soil classes; Chemical criteria, crop production, and physical classification in two* —. J. S. Burd. Soil Sci., 1918, 5, 405—419.

DATA are given of the mechanical analyses and chemical analyses by the fusion method, and by extraction with hydrochloric acid (sp. gr. 1.115), and with 1% citric acid respectively, as well as the crop production under controlled conditions, of fourteen soils of which five belong to a single series of one soil type and eight to different series of another type. Seasonal averages of the water extractions of these soils are also given. The results obtained by the water extraction and to a lesser extent by the citric acid extraction method are shown to be in general accord with the soils' crop-producing powers, whereas the complete analysis and hydrochloric acid extraction method are worthless as criteria of present production or probable endurance except in the case of highly silicious soils. The great variations in crop yield and in the analytical figures for the water extractions within a given series and soil type indicate that physical classification into types is inadequate as a means of predicting probable yields or determining fertiliser requirements.—W. G.

*Soil; Chemical effects of calcium oxide and calcium carbonate on the —. I. The effect on soil reaction.* D. R. Hoagland and A. W. Christie. Soil Sci., 1918, 5, 379—382.

CALCIUM oxide when first added to a soil produces a high concentration of hydroxyl ions, which decreases within a few days and continues to decrease



slowly over a considerable period, but the treated soils still show a greater intensity of alkalinity even after several months as compared with the untreated soils or those to which calcium carbonate has been applied. If a considerable excess of calcium oxide is used, certain soils may maintain such a high hydroxyl ion concentration as practically to inhibit nitrification.—W. G.

*Soil; Chemical effects of calcium oxide and calcium carbonate on the —. II. The effect on water-soluble nutrients in soils.* A. W. Christie and J. C. Martin. *Soil Sci.*, 1918, 5, 383–392.

SEVEN soils of three different types were examined to find the effect of applications of calcium oxide or carbonate on their water-soluble nutrients. In certain cases the concentration of the soil solution was materially increased by the application of lime, the effect of calcium oxide being more pronounced than that of the carbonate. The effect of lime on soils depends in part upon the previous treatment of the soil, particularly with respect to the concentration of the soil solution. A greater effect was observed in soils recently cropped or having a low initial concentration. The application of lime increased the water-soluble potassium in only two cases and in two soils caused a depression. Soluble magnesium was increased in four soils and decreased in one. Soluble sulphates were increased in four soils, but only two soils showed any significant increase in phosphates.—W. G.

*Soils; Partial sterilisation of —.* G. Truffaut. *Comptes rend.*, 1918, 167, 433–436.

FIELD experiments with various vegetable crops using carbon bisulphide or calcium sulphide as antiseptics have shown marked increased crop production due to partial sterilisation. The carbon bisulphide was applied at the rate of 500 kilos. per hectare, whilst the most satisfactory rate for calcium sulphide was 250–300 kilos. per hectare applied in February or March. Satisfactory results were also obtained by using a mixture of equal parts by weight of calcium sulphide and naphthalene.

—W. G.

*Carbon dioxide; Production of — by moulds inoculated into sterile soil.* R. S. Potter and R. S. Snyder. *Soil Sci.*, 1918, 5, 359–377.

TYPICAL soil moulds when inoculated into sterilised soil grew with a vigour equal to or nearly equal to the growth induced by inoculation with the entire soil flora, the vigour of growth being measured by the evolution of carbon dioxide. The presence of calcium carbonate in the soil had no marked effect on the growth of moulds. Two methods of sterilisation were used, namely by heating in an autoclave for three hours at 10 lb. pressure, three times at intervals of 48 hours, which treatment caused an increase in the nitrate, ammonia, and soluble non-protein nitrogen; and by heating in an Arnold steriliser at 99° C. with flowing steam six times for 1½ hours on alternate days, which treatment caused an increase in the ammonia and soluble non-protein nitrogen, but a decrease in the nitrate nitrogen. Moulds caused a decrease in the amount of nitrates in all cases, a decrease in the amount of soluble non-protein nitrogen in nearly every case, and but very little change in the amount of ammonia.

—W. G.

*Cane sugar [sucrose]; Inversion of — by soils and allied substances and the nature of soil acidity.* F. E. Rice and S. Osugi. *Soil Sci.*, 1918, 5, 333–358.

SOILS of many kinds are capable of inverting sucrose, this power being a property of the mineral

portion as well as of the organic matter of the soil. The effect is undoubtedly due to acid which may be present in one of four forms, viz., a slight amount in certain soils, soluble in the sugar solution; acids ordinarily easily soluble, but in the soil strongly adsorbed on the soil particle surfaces; an insoluble acid, such as silicic acid; acids liberated as a result of the presence in the soil solution of neutral salts of which the bases are more strongly adsorbed than the acids, which latter are thus left free to cause inversion of the sugar. The inversion of sugar by soils is chiefly a property of the insoluble portion, since aqueous extracts had little or no inverting power; the inversion increased with the amount of soil in contact with the sugar solution; long continued and repeated extraction of the soils with water or the sugar solution did not greatly reduce their inverting power. Many soils showed inverting action on sugar in a solution which was neutral or in some cases alkaline after contact with the soil. It was found that silicate minerals in aqueous suspension when submitted to direct electric current acquired inverting power and soils under similar treatment had their soil acidity increased. The authors consider that the measurement of the power of a soil to catalyse the inversion of sucrose is probably the only method which can measure the acidity bound up with the solid phase of the soil.

—W. G.

*Fertiliser from the effluent from potassium chloride works.* W. Hüttner. *Chem.-Zeit.*, 1918, 42, 434–435.

THE effluent is introduced into a tank containing quicklime, the latter being covered with the liquid; heat is developed and the whole is converted into a powdery mass which consists of magnesium oxide, magnesium and calcium oxychlorides, small quantities of potassium chloride, sodium chloride, calcium sulphate, etc. Its fertilising value depends chiefly on the presence of the magnesium oxide.

—W. P. S.

#### PATENTS.

*Fertilising method and material.* W. O. Snelling, Pittsburgh, Pa. U.S. Pat. 1,274,343, July 30, 1918. Date of appl., Apr. 17, 1915.

A CARTRIDGE containing an explosive material surrounded by finely divided calcium phosphate is placed in the soil and is then fired.—J. H. J.

*Fertiliser; Process of producing a phosphoric acid — containing urea.* E. Lie, Odda, Norway. U.S. Pat. 1,275,276, Aug. 13, 1918. Date of appl., May 6, 1918.

A CYANAMIDE, superphosphate, and water are mixed together, with or without the addition of an acid anhydride, and allowed to react until the nitrogen of the cyanamide is largely converted into urea compounds.—J. H. J.

*Calcium cyanamide; Process of improving the grade of —.* G. E. Cox, Niagara Falls, N.Y., Assignor to American Cyanamid Co., New York. U.S. Pat. 1,275,535, Aug. 13, 1918. Date of appl., Dec. 3, 1917.

A SUPPORTING form containing a paper sleeve is introduced into a cyanamide furnace. The sleeve is filled with finely ground calcium carbide and covered with a heat insulating cover. The form is then removed, and the carbide converted into cyanamide as usual.—J. H. J.

*Fertiliser.* P. Kessler, Kleefeld. Ger. Pat. 307,244, May 16, 1917.

LIME sludge containing finely-divided carbon which has been formed at a high temperature, *c.g.*, the sludge obtained in the manufacture of ammonia from crude calcium cyanamide, is converted into a dry powder by mixing with quicklime. The finely-divided carbon is readily oxidised in the soil.

—L. A. C.

*Fertiliser from peat and process of making the same.* N. Testrup, London, and T. Rigby, Dumfries. Assignors to Wetcarbonizing, Ltd., London. U.S. Pat. 1,277,155, Aug. 27, 1918. Date of appl., July 29, 1915.

SEE Eng. Pat. 18,888 of 1914; this J., 1916, 133.

*Recovery of proteids [fertilisers].* U.S. Pat. 1,274,763. See XV.

## XVII.—SUGARS; STARCHES; GUMS.

*Starch in potatoes; Effect of frost and decay upon the —.* H. A. Edson. J. Ind. Eng. Chem., 1918, 10, 725–726.

FROZEN and decayed potatoes yield, in many cases, normal quantities of good quality starch, but the mechanical difficulties in the recovery of starch from decayed pulp are sometimes greater than usual.—W. P. S.

*Laundry starch.* Reinke. See VI.

*Inversion of sucrose.* Rice and Osugi. See XVI.

### PATENTS.

*Glucose; Manufacture of —.* C. Hopkinson, Bradford. Eng. Pat. 118,651, July 3, 1917. (Appl. No. 9509 of 1917.)

THE friable hydrocellulose waste obtained from the carbonisation of materials containing mixed animal and vegetable fibres is dissolved in the proportion of 1 part of carbonised waste to 3 or 4 of 65–70% sulphuric acid; the mixture is allowed to stand for 3–4 hours, then diluted to give a 1–2% solution of sulphuric acid, boiled for 1 hour, neutralised with calcium carbonate, filtered, and evaporated.

—J. F. B.

*Vegetable glue or adhesive.* R. W. Tunnell, Philadelphia, Pa. U.S. Pat. 1,275,799, Aug. 13, 1918. Date of appl., Dec. 22, 1917.

A BLEND of high- and low-grade starch of the same kind (*e.g.* 20% of high-grade and 80% of low-grade tapioca) is treated with barium hydroxide, or other alkaline-earth hydroxide, in the proportion of, say, 0.1 to 0.25% of the total weight of starch.—C. A. M.

## XVIII.—FERMENTATION INDUSTRIES.

*Sisal waste; Utilisation of — for the production of alcohol.* Tropical Life, 1917, 13, 155. Bull. Agric. Intell., 1918, 9, 988.

AFTER the fibre has been extracted from sisal there remains much waste. At Yncatan, whence sisal was first exported, alcohol has been made from the waste; the leaves from which the alcohol was made contained 12% of sugar, but sometimes, after a long

dry season, the sugar content reached 14%. Experiments in British East Africa also showed that it is possible to prepare alcohol from sisal; the plants in this case contained 3% of sugar. A yeast was isolated from sisal plants which fermented a dextrose solution but was killed quickly in sisal extract, no doubt owing to the large amounts of organic acids present. After neutralisation with sodium carbonate and the addition of a small quantity of dextrose, the extract did not kill this yeast but no fermentation took place.—W. P. S.

*Alcoholic fermentation; Arrest of — in the aldehyde stage. Experimental confirmation of the acetaldehyde pyruvic acid theory.* C. Neuberg and E. Reinthur. Biochem. Zeits., 1918, 89, 365–414.

NEUBERG (this J., 1911, 379, 1179; 1913, 803) has shown that yeast can convert pyruvic acid into carbon dioxide and acetaldehyde, and that the latter substance readily undergoes reduction to ethyl alcohol during yeast fermentations. According to the theory set forth by the authors, only one molecule of aldehyde should be formed from one molecule of sugar, the other part of the molecule acting as acceptor for the hydrogen. By fermenting sugar in the presence of disodium sulphite, aldehyde in the amount equivalent to 73.45% of that required by this theory can be isolated. (See also J. Chem. Soc., 1918, i., 517.)—S. B. S.

*Catalase of bacteria.* Jacoby. Biochem. Zeits., 1918, 89, 350–354.

PROTEUS bacteria were grown on media containing, in addition to inorganic salts, only sodium aspartate and sodium lactate. An active catalase could be obtained from the cultures by precipitation with sodium chloride, or with magnesium or ammonium sulphate. These experiments form part of an attempt to prepare a pure enzyme. (See also J. Chem. Soc., 1918, i., 517.)—S. B. S.

*Potash, etc., from kelp.* Higgins. See VII.

*Separating oxalic from tartaric acid.* Bau. See XX.

*Acetic acid and acetone.* Hibbert. See XX.

### PATENT.

*Beverages; Manufacture of dry extracts of partly-fermented —.* H. Heuser, Chicago, Ill. U.S. Pat. 1,275,254, Aug. 13, 1918. Date of appl., Mar. 12, 1917.

PARTLY-FERMENTED wort containing live yeast is dehydrated at a temperature which will not injure the yeast; the dry compound may be converted into a beverage by dilution with water.—W. P. S.

## XIXA.—FOODS.

*Milk; Certain changes caused by bacteria in some of the nitrogenous constituents of —.* G. C. Supplee. J. Dairy Sci., 1917, 1, 312–319. Bull. Agric. Intell., 1918, 9, 990–991.

IN an investigation on changes in the nitrogenous evident, the following species of bacteria were change in the physical appearance of the milk was evident, the following species of bacteria were used:—*B. lactis acidii*, *Mic. albidus*, *Ps. liquefaciens*, *B. lactis viscosus*, *B. mycoides*, "Acid peptoniser" (species unknown), *B. bulgaricum*, *B. coli*



*communior*, *B. aerogenes*, *B. prodigiosus*, *Ps. pyocaneus*, and *B. subtilis*. All these organisms, except two, caused a decrease in the casein fraction; with some, there was an increase in the albumin fraction, with others a decrease in the albumin fraction and little or no decrease in the casein fraction.—W. P. S.

*Milk; Determination of cow manure in —. Moisture content and solubility of cow manure.* G. B. Taylor. *J. Dairy Sci.*, 1917, 1, 303–312. *Bull. Agric. Intell.*, 1918, 9, 991.

WHEN the quantity of manure present in the milk is large, it can be determined fairly accurately by a chemical method, but in the majority of milks the amount of manure is too small to be determined chemically. The manure examined contained 83% of water. Air-dried manure contained 6% of water; 5% of the remaining solid matter was soluble in milk; this would indicate that only 11% of air-dried manure dissolved in milk. Nearly 85% of fresh cows' manure dissolved in milk, 83% of this being water. Manure, in whatever condition, was less soluble in milk than in water.—W. P. S.

*Butter; Influence of salt on the changes taking place in storage —.* R. M. Washburn and A. C. Dahlberg. *J. Dairy Sci.*, 1917, 1, 114–126. *Bull. Agric. Intell.*, 1918, 9, 996–997.

It is generally believed that salt improves the keeping quality of butter, but it has been observed that unsalted butter in commercial cold storage keeps as well as, or better than, salted butter. Investigations carried out by the authors show that salt, exclusive of its antiseptic property, hastened the deterioration of butter; when stored at  $-15^{\circ}\text{F.}$  ( $-25^{\circ}\text{C.}$ ), unsalted butter kept as well as salted butter. The bacteria decreased more rapidly at  $-15^{\circ}\text{F.}$  ( $-25^{\circ}\text{C.}$ ) in unsalted butter than in salted butter and increased more rapidly at  $58^{\circ}\text{F.}$  ( $15^{\circ}\text{C.}$ ). The acidity of unsalted and salted butters increased uniformly at  $-15^{\circ}\text{F.}$  ( $-25^{\circ}\text{C.}$ ) but at  $58^{\circ}\text{F.}$  ( $15^{\circ}\text{C.}$ ) the increase was greater in the unsalted butter. Moisture was lost from the salted butter but not from the unsalted butter kept at  $-15^{\circ}\text{F.}$  ( $-25^{\circ}\text{C.}$ ).—W. P. S.

*Butter; Relation of Oidium lactis and Penicillium to the keeping qualities of —.* W. B. Combs and C. H. Eckles. *J. Dairy Sci.*, 1917, 1, 347–355. *Bull. Agric. Intell.*, 1918, 9, 992–993.

Mould spores do not germinate or grow in butter; the growth of moulds on butter is due to contamination from outside and does not proceed readily. The taste of cream is considerably affected by the growth of *Oidium lactis* and *P. chrysogenum* when the cream is sweet, but mould growth on sour cream does not produce an objectionable flavour; the growth of these moulds on cream has a detrimental effect on the butter made from the cream. Abnormal flavours which develop in the butter are due to enzymes which are secreted by moulds in the cream. Pasteurisation greatly improves the keeping quality of butter made from mouldy cream but it does not check completely the action of the enzymes produced.—W. P. S.

*Butter, lard, etc.; Crismer test for the detection of foreign fat in —.* A. W. Stewart. *J. State Med.*, 1918, 26, 312–315.

INFORMATION similar to that furnished by the Valenta value can be obtained by a modification of Crismer's test (this J., 1895, 1069). Three c.c. each of filtered fat and absolute alcohol are pipetted into a test-tube and heated over a small flame with continuous stirring with a thermometer until

the mixture is clear. The tube is then removed from the flame and the contents stirred until turbidity appears. The temperature at which this occurs constitutes the Crismer number. Butter fat gives a result varying from 50.5 to 57, whilst margarine has a value over 65 if it is composed of animal fat, and under 50 if prepared from vegetable fat. The Crismer test yields more consistent results than can be obtained by the Valenta method, because absolute alcohol of constant quality can readily be obtained, whereas the commercial glacial acetic acid required for the Valenta test differs considerably in its moisture content from time to time (see Fryer and Weston, this J., 1918, 64a). The fat must be free from moisture and quite clear, and can generally be so obtained by filtration in a hot water oven through a dried filter paper. The Crismer test may also be applied to lard, the normal value for which is 76 to 77. The Crismer values of other fats are as follows:—Sesame oil 67.5, almond oil 64, cottonseed oil 61.5, arachis oil 57.5, olive oil 56, cacao butter 47, tallow 34.5, palm oil 22, coconut oil 15 to 19.5, palm kernel oil 13.5. Rape oil is characterised by its insolubility at the boiling point of the mixture.—H. W. B.

*Egg; Chemical and physical constants of the oily matter of the —.* F. Schaffer and L. Meyer. *Mitt. Lebensmittelunters. u. Hyg.*, 1918, 9, 135–136. *Bull. Agric. Intell.*, 1918, 9, 995.

THE nature of the solvent has a considerable influence on the physical and chemical constants of the oil extracted from egg yolk; petroleum spirit extracts the oily matter alone, whilst chloroform extracts the cholesterol and a portion of the lecithins as well. Results obtained by the authors show that the refractive index of the oil lies within the limits recorded in literature; the iodine value is, on an average, 74 to 75. The phosphorus content of the lecithins diminishes gradually during storage, being 1.27% (as  $\text{P}_2\text{O}_5$ ) in the case of fresh eggs and 0.8% after storage for 2 years.—W. P. S.

*Foods; Detection of decomposition in —.* K. Brauer. *Chem.-Zeit.*, 1918, 42, 421–422.

INCIPIENT decomposition in sausages, preserved foods, etc., may be detected by inoculating dextrose-bouillon with a small portion of the sample, incubating at  $38^{\circ}\text{C.}$  for 24 to 48 hrs., and noting whether or not gas is produced during this period. If there is formation of gas, organisms capable of decomposing the food are present; further investigation by sub-cultures will, if desired, indicate the kind of organism. The dextrose medium employed is prepared by dissolving 1 gm. of meat extract in 100 c.c. of water, adding 1 gm. of peptone and 0.5 gm. of sodium chloride, sterilising the solution, then adding 1 gm. of dextrose, introducing portions of the solution into a series of Einhorn fermentation tubes, and again sterilising for a short time.—W. P. S.

*Ergotinine.* Wolter. *See* XX.

#### PATENTS.

*Wheat for milling purposes; Preparatory treatment of —.* J. S. Remington. *Grange-over-Sands, Lancs. Eng. Pat.* 118,431, July 28, 1917. (Appl. No. 10,897 of 1917.)

THE wheat is washed with a 10% solution of potassium or sodium carbonate, then centrifuged, and dried in a current of cold (preferably refrigerated) air.—W. P. S.

**Bread; Manufacture of leavened** —. H. A. Kohman, Pittsburgh, Pa., Assignor to Ward Baking Co., New York. U.S. Pat. 1,274,898, Aug. 6, 1918. Date of appl., Feb. 15, 1918.

A MEAL-LIKE product containing diastatic and proteolytic enzymes, produced by the action of *Aspergillus oryzae* on grains, seeds, maize, legumes, etc., is added to the flour, yeast and other ingredients of dough, and the mixture is fermented.—W. P. S.

**Soya bean milk; Manufacture of — and the complete utilisation of by-products.** W. J. Melhuish, Poole, Dorset. Eng. Pat. 118,535, Dec. 10, 1917. (Appl. No. 18,279 of 1917.)

SOYA beans are immersed in water at 60° C., allowed to cool in the water for 24 hrs., then drained and ground; 16 lb. of the meal is mixed with 100 lb. of water containing 140 grains of potassium phosphate, and the mixture is stirred and heated at 95° C. for 45 mins. The mixture is then passed through a filter-press, the press-cake being afterwards transferred to a hydraulic press to obtain a feeding cake and separate the oil. The liquid from the filter-press is centrifuged, heated under reduced pressure, and stirred with the addition of suitable oils (sesame, earthnut, coconut, etc.), then cooled to 36° C., treated with a culture, salts and acids (e.g., butyric acid) are added, and after the lapse of sufficient time for the culture to grow, the whole mixture is sterilised at 70° C., cooled to 16° C., and stored. The separator slime may be mixed with the foots obtained in clarifying the soya bean oil, and the mixture used as a soap base.—W. P. S.

**Whey extract for use as a food and process of producing the same.** H. H. Senior, East Stour, Dorset. Eng. Pat. 118,874, Aug. 28, 1917. (Appl. No. 18,354 of 1917.)

WHEY is heated at about 190° F. (88° C.) for 1½ hrs., then cooled, and the coagulated substances are separated, drained, dried slowly and ground to a fine powder. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 23,127 of 1909; this J., 1910, 1327.)—W. P. S.

**Milk; Process of making a new food product of —.** P. W. Turney, Portland, Oreg. U.S. Pat. 1,274,218, July 30, 1918. Date of appl., Mar. 22, 1917.

FRESH milk is curdled by an enzyme, the action of which is allowed to continue until the precipitate becomes flocculent, when the temperature is rapidly lowered to prevent further enzyme action. The low temperature is maintained until the precipitate is required to be used.—J. H. J.

**Milk; Process of treating** —. N. D. Nielsen, Elyria, Ohio. U.S. Pat. 1,274,748, Aug. 6, 1918. Date of appl., Feb. 11, 1918.

MILK at 40° F. (4° C.) is introduced into a vessel provided with a jacket heated at 212° to 220° F. (100° to 105° C.); the milk is stirred until its temperature is 110° to 160° F. (43° to 71° C.) and then cooled in the same vessel while the stirring is continued.—W. P. S.

**Milk and the like; Process of treating** —. N. D. Nielsen, Elyria, Ohio. U.S. Pat. 1,274,750, Aug. 6, 1918. Date of appl., Mar. 22, 1918.

MILK or other liquid is sterilised at 145° F. (63° C.) by placing it in a jacketed vessel and introducing a heated condensable gas into the jacket; a cooling medium is then admitted to the jacket so that the

gas is condensed and a partial vacuum created in the jacket. After 30 mins., a cooling medium is again admitted to the jacket so as to cool the milk to 36° F. (2° C.), the cooling medium is then withdrawn, and the condensable gas again introduced and condensed, so that the liquid is then contained in a vacuum-jacketed vessel.—W. P. S.

**Milk and other liquids readily decomposed by heat** [e.g., blood, serum, must, etc.]; Sterilisation of —. O. Lobeck, Leipzig. Ger. Pat. 306,924, Sep. 30, 1913.

THE liquid is spread out by means of a current of air or other gas on the surface into very thin layers on a suitable support and exposed for a short time to a sterilising temperature.—L. A. C.

**Foods and other products; Drying of** —. L. P. Bowler, A. Paterson, and L. S. Levy, London. Eng. Pat. 118,911, Oct. 2, 1917. (Appl. No. 14,251 of 1917.)

THE material to be dried, such as fruit, vegetables, meat, fish, copra, coal, etc., is placed on trays arranged in a closed chamber through which a current of dry heated air is drawn by means of a pump. The air is heated by passing through a chamber containing a steam coil and then enters a chamber fitted with air filters and also containing drying substances, such as calcium chloride; this filter chamber is provided with a heat-insulating jacket. After traversing the heating coil the steam may be conducted to a jacket surrounding the drying chamber. Dehydrating substances may be placed in the air-heating chamber and in the drying chamber, and also arranged between the drying chamber and the pump.—W. P. S.

**Fruit; Apparatus for drying** —. T. W. W. Forrest, Oakland, Cal. U.S. Pat. 1,275,547, Aug. 13, 1918. Date of appl., Apr. 1, 1918.

THE fruit is dried on an endless conveyor constructed of open-mesh material and mounted on a removable frame fitted in a vacuum chamber. Heating devices and means for operating the conveyor are provided in the chamber, and the inlet and outlet passages for the fruit are fitted with valves.—W. P. S.

**Eggs; Dried — in powder form.** E. S. Spencer and F. F. Kemp, London. Eng. Pat. 119,007, Apr. 17, 1918. (Appl. No. 6502 of 1918.)

WHOLE eggs are dried until the water content is reduced to 5%; egg yolks are dried separately in a similar manner. Five parts by weight of the dried whole egg is then mixed with 2 parts of the dried yolks. The resulting product is miscible with water and contains protein 42.6, fat 49.0, ash 3.4, and water 5%.—W. P. S.

**Vegetable proteid substances; Process of manufacturing** —. S. Satow, Sendai, Japan. U.S. Pat. 1,275,308, Aug. 13, 1918. Date of appl., Nov. 25, 1916.

VEGETABLE substances are freed from oil, extracted with water, the extract is clarified, and the soluble proteins then precipitated by the action of lactic ferment.—W. P. S.

**Proteid matter from corn [maize]; Process of obtaining** —. J. Takamine, New York, and S. Satow, Sendai, Japan. U.S. Pat. 1,275,324, Aug. 13, 1918. Date of appl., Nov. 25, 1916.

MAIZE is steeped in water, crushed, the germs are removed, and the mass is ground with water; after



separating bran and starch, the substances remaining in the liquid are separated in the form of a "meal" and transferred to a "liquid solution," and the proteins are precipitated from this solution.  
—W. P. S.

*Food products of starchy material; Method of preparing* —. F. G. Lorenzen, Assignor to Kellogg Toasted Corn Flake Co., Battle Creek, Mich. U.S. Pat. 1,275,711, Aug. 13, 1918. Date of appl., Mar. 30, 1918.

STARCHY material is subjected to the action of steam for about 2–4 hrs., first at a pressure of less than 2 lb. per sq. in., then at a higher one (10–20 lb. per sq. in.), the globules being thereby gelatinised. They are then separated, dried at 100°–110° C., and subsequently subjected to a sharp dry heat, whereby they are puffed.—A. B. S.

*Cotton-seed flour and process of making the same.* G. A. Baumgarten, Schulenburg, Tex. U.S. Pat. 1,276,477, Aug. 20, 1918. Date of appl., Sep. 21, 1915.

COTTONSEED is freed from bran, and cooked under steam-pressure for substantially 1 hour 40 mins. at about 238°–240° F. (114.5°–115.5° C.), or for a sufficient length of time at a sufficient temperature to rupture the oil-cells. This cooking process may be effected in a series of cookers the steam-pressure in which is successively reduced. The oil and disintegrated coating of the oil-cells are separated by expression, and the residual cake is dried, ground, and bolted to produce flour.—C. A. M.

*Food; Substitute highly-nitrogenous — and process of producing the same.* N. Moskovits and K. A. Jacobsen, Assignors to Moskovits Mor és Fia, Nagy-Várád, Hungary. U.S. Pat. 1,277,336, Aug. 27, 1918. Date of appl., Feb. 26, 1916.

SEE Eng. Pat. 100,135 of 1916; this J., 1916, 1128.

## XIXB.—WATER PURIFICATION; SANITATION.

*Water purification; Baryta as substitute for soda in* —. F. Hundeshagen. Z. öffentl. Chem., 1918, 24, 159–167.

IN the softening of boiler water containing calcium sulphate, the use of barium chloride results in the precipitation of barium sulphate which does not form a crust or corrode the boiler. If barium carbonate is used, calcium sulphate is converted into barium sulphate and calcium carbonate. The process leads to some loss of barium carbonate which is used in excess, but if the precipitant is added to the hot water, only 5–10% in excess need be used and the precipitation of calcium is complete. Barium hydroxide reacts with calcium sulphate to form barium sulphate and calcium hydroxide. The latter is objectionable on account of its tendency to form a hard crust on the hottest parts of the boiler. In the presence of calcium bicarbonate, barium sulphate and calcium carbonate are precipitated. If the calcium sulphate is in excess, a little calcium hydroxide is formed, but this can be removed with barium carbonate or with sodium carbonate. Baryta water precipitates magnesium hydroxide from magnesium salts. Barium aluminate acts as a precipitant in a similar manner to barium hydroxide. Formulae and examples are given for calculating the amount of precipitant to be used for various combinations of hardening salts.—J. H. J.

*Catalase.* Jacoby. See XVIII.

*Anthrax.* (1) Kelser. (2) Berg. See XX.

### PATENTS.

*Softening water.* E. Edser, S. Tucker, and Minerals Separation, Ltd., London. Eng. Pat. 118,668, Sep. 3, 1917. (Appl. No. 12,624 of 1917.)

THE water to be softened is treated with tri- or disodium phosphate and caustic soda, or other suitable reagents, to precipitate the hardening salts. The precipitate produced is formed into a froth by addition of sodium oleate and violent agitation with air. On allowing the water to come to rest, the precipitate floats on the surface. Iron salts also may be removed from water by this method.

—J. H. J.

*Water; Apparatus for heating and deoxidising* —. F. N. Speller, Pittsburgh, Pa. U.S. Pat. 1,274,099, July 30, 1918. Date of appl., Apr. 29, 1918.

THE apparatus consists of a water heater containing a series of thin plates of steel or iron for deoxidising the water.—J. H. J.

*Filtering apparatus [for water].* C. S. Smith, Brooklyn, N.Y. U.S. Pat. 1,276,129, Aug. 20, 1918. Date of appl., May 18, 1914. Renewed Jan. 10, 1918.

A TANK is fitted with transverse partitions forming galvanic couples, between which are layers of a filtering medium of carbon and aluminium. The water to be filtered is forced upwards through the tank under pressure, and the residue descends to the bottom.—J. H. J.

*Softening water; Method of and apparatus for* —. T. R. Duggan, Assignor to The Permutit Co., New York. U.S. Pat. 1,276,629, Aug. 20, 1918. Date of appl., Oct. 23, 1916.

A WATER-SOFTENING bed of exchange-silicates containing lime and magnesia is regenerated after use by passing an upward current of sodium chloride solution through the bed as long as lime and magnesia continue to be removed, drawing off the contaminated solution at the surface of the bed after a single passage, and flushing out the residue of the solution.—J. H. J.

*Tetra-iodo-hexamethylenetetramine [antiseptic] composition.* K. G. Falk and K. Sugiyama, Assignors to G. B. Pegram, New York. U.S. Pat. 1,275,162, Aug. 6, 1918. Date of appl., June 23, 1917.

A COMPOSITION, for use as an antiseptic, healing, or sterilising agent, comprises 1% of tetra-iodo-hexamethylenetetramine diffused uniformly throughout a liquid solvent or vehicle, such as collodion.—B. N.

*Antiseptic and germicidal tablet.* A. Rogers, Assignor to C. T. Davis, Brooklyn, N.Y. U.S. Pat. 1,276,119, Aug. 20, 1918. Date of appl., Apr. 2, 1918.

THE tablet consists of potassium mercuric iodide with a sufficient quantity of a stabilising agent, such as an iodide, to prevent the formation of insoluble mercuric iodide, and with a sufficient quantity of sodium chloride to act as a binder and to give a solution of the tablet a tonicity approximating to that of the blood.—J. H. J.

*Preserving organic materials by the use of X-rays; Apparatus for* —. D. C. Gillett, Tampa, Fla. U.S. Pat. 1,275,417, Aug. 13, 1918. Date of appl., Apr. 14, 1917.

THE apparatus consists of an outer chamber impervious to X-rays and an inner chamber with one or more of its sides pervious to X-rays. At the pervious side of the inner chamber is a chamber containing several X-ray tubes connected to a source of electric current.—J. H. J.

*Water; Apparatus for purifying* —. L. Linden, London, and W. J. Stewart, Belfast. U.S. Pat. 1,276,234, Aug. 20, 1918. Date of appl., July 9, 1915.

SEE Fr. Pat. 474,907 of 1914; this J., 1915, 1112.

*Sterilising milk, etc.* Ger. Pat. 306,924. See XIXA.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Ergotinine; Detection of* —. L. Wolter. Chem.-Zeit., 1918, 42, 446.

ERGOT of rye contains about 0.12% of ergotinine, about two-thirds of which can be separated in the amorphous condition and about one-third in the crystalline form by Tanret's method. The potassium mercuric iodide reagent for alkaloids gives a distinct reaction with an alcoholic solution of 1 part of ergotinine in 1,240,000. Tanret's reaction (yellowish-red zone changing to violet and then to blue), with sulphuric acid added to the solution in ether, alcohol, or ethyl acetate, is characteristic of ergotinine. In Rosenthaler's modification sulphuric acid is added to a solution of the alkaloid in glacial acetic acid which has previously been treated with a trace of ferric chloride, whilst Keller applies the test by adding strong sulphuric acid containing a trace of ferric salt to a solution of ergotinine in ethyl acetate. The reaction may be rendered more sensitive by using a trace of hydrogen peroxide in place of ferric chloride as the oxidising agent, and is then capable of detecting 0.01 mgrm. as against 0.02 mgrm. by Tanret's original test. It is essential that the solution of the alkaloid should not be too concentrated, or misleading colorations will be obtained. (See also J. Chem. Soc., Nov., 1918.)

—C. A. M.

*Eserine.* J. Herzig and H. Lieb. Monatsh. Chem., 1918, 39, 285–292.

THE micro-method for the determination of imino-methyl groups in eserine and eseroline indicates the presence of one more such group than the ordinary macro-method. If, in the ordinary method, the proportion of hydriodic acid to substance is increased, e.g. by taking 0.1 gm. of eserine to 29 c.c. of hydriodic acid, the result indicates three methyl groups in agreement with the micro-method. It is possible that the third difficultly removable alkyl group may be an ethyl radicle, and not methyl. (See also J. Chem. Soc., 1918, 1., 504.)—D. F. T.

*Scoparin.* J. Herzig and G. Tiring. Monatsh. Chem., 1918, 39, 253–267.

SCOPARIN, the yellow substance present in *Spartium scoparium*, when treated with diazomethane, can be made to yield a dimethyl and a trimethyl derivative, whereas with silver oxide and methyl iodide a heptamethyl derivative is obtainable. Scoparin, therefore, contains at least seven hydroxyl groups. It also contains a methoxyl group. The composition

of these products indicates a formula  $C_{22}H_{22}O_{11}$  for scoparin (compare Goldschmidt and von Hemmel-mayr, this J., 1895, 63; see also J. Chem. Soc., 1918, 1., 503).—D. F. T.

*Symptomatic anthrax (blackleg); Methods of immunisation against* —. R. A. Kelsner. J. Agric. Res., 1918, 14, 253–262.

THE medium found to be the best for growing the bacillus of symptomatic anthrax in order to produce a true toxin was a dextrose-peptone bouillon, of which the peptone was prepared as a solution direct from pigs' stomachs. Flasks were filled with this medium up to the neck, and several c.c. of a recently isolated, virulent culture of the bacillus added to the bottom of each. After 10 days' growth, the culture was filtered through a Berkefeld filter and the filtrate preserved with chloroform. This product was found to possess valuable immunising properties against blackleg.—J. H. J.

*Symptomatic anthrax (blackleg) toxin; Concentration of* —. W. N. Berg. J. Agric. Res., 1918, 14, 263–264.

TOXIN, prepared as described in the preceding abstract, was placed in Petri dishes and frozen. The dishes and contents were then placed in evacuated desiccators and maintained at  $-9^{\circ}\text{C}$ . for 1–2 days, when the toxin had evaporated to a paste. —J. H. J.

*Oxalic acid; Separation of* — from tartaric acid. A. Bau. Chem.-Zeit., 1918, 42, 425–426.

THE solution, containing not more than 0.2% of oxalic acid, is treated with an excess of boric acid (at least 0.25 mol. of boric acid per mol. of tartaric acid is required to prevent the precipitation of the latter) and one-fifth of its volume of calcium acetate reagent. The mixture is kept in an ice-chest for about 40 hrs., the precipitated calcium oxalate then collected, washed, ignited, and the resulting calcium oxide titrated with N/10 hydrochloric acid. One litre of the reagent dissolves 3.42 mgrms. of calcium oxalate and 1 litre of wash-water dissolves 4.64 mgrms.; the volumes of the filtrate and wash-water must be noted, and the corresponding correction applied to the result. The calcium acetate reagent is prepared by dissolving 330 grms. of sodium acetate in 300 c.c. of water, and adding 500 c.c. of this solution to 25 grms. of calcium chloride dissolved in 500 c.c. of 50% acetic acid; the mixture is kept in an ice-chest for 24 hrs. and then filtered. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Acetic acid and acetone; Industrial developments relating to the manufacture of* —. H. Hibbert. Chem. and Met. Eng., 1918, 19, 397–400.

ON the outbreak of war the demand for acetic acid and acetone rose to such an extent that the output of the wood-distillation industry was totally inadequate to meet the requirements. Working in conjunction with the British Government, the U.S. Industrial Chemical Co. erected a large plant which is now producing acetic acid by the oxidation of alcohol obtained from molasses by the quick fermentation process. The plant has an output of 70,000 lb. of acetic acid (calculated at 100%) per day. The oxidation of the alcohol to acetic acid is effected in generators filled with beech shavings. It is most probable that this process will not be profitable under normal conditions. Acetone is also produced in Toronto and in the United States by the Fernbach process, in which starch from maize or other grain is fermented by a special ferment, which resolves the carbohydrate into a mixture



of butyl alcohol and acetone. The output of acetone at the Toronto plant is 100 tons per month, and at the United States plant nearly 250 tons. A process has been worked out and put into operation in Canada (output 56,000 lb. of 100% acetic acid per day) which is based on the German patents for the manufacture of acetic acid from acetylene. When acetylene gas is passed through a solution of a mercury salt in dilute acid, it combines with the water yielding acetaldehyde, which is readily converted by atmospheric oxygen into acetic acid. The process is important as a potential source ultimately of cheap acetic acid, and of acetaldehyde for probable new industrial uses. In Germany and in Switzerland large electrical installations have been erected for the manufacture of alcohol and acetic acid from calcium carbide. The wood-distillation industry is threatened with dangerously keen competition in the future. Development on scientific lines, and research for the exploitation of untapped sources of revenue in the acetone, crude acetone oils, and wood tar are imperative.—S. S. A.

*Acetol; New reaction for* —. O. Baudisch. *Biochem. Zeits.*, 1918, 89, 279—280.

A DILUTE solution of acetol in water containing sodium hydroxide is boiled for a few minutes with *o*-aminobenzaldehyde. The mixture is cooled, acidified, and then made alkaline again with sodium bicarbonate. A fluorescent solution is obtained from which 3-hydroxyquinoline can be extracted by ether; on distilling off the ether it is obtained as a white residue, which gives a deep red colour with ferric chloride in alcoholic solution. The alcoholic solution also gives a brilliant blue fluorescence on dilution with water. These two reactions are characteristic. (See also *J. Chem. Soc.*, Nov., 1918.)—S. B. S.

*Potash, etc., from kelp.* Higgins. See VII.

*Citric acid esters.* Pinnow. See XII.

#### PATENTS.

*1,8-Dihydroxyanthranol; Manufacture of* —. *Manufacture of 1-hydroxyanthranol. Manufacture of 1-hydroxy- and 1,8-dihydroxyanthranol.* *Farbenfabr. vorm. F. Bayer and Co. Ger. Pats.* (A) 296,091, Oct. 30, 1915, (B) 301,452, Feb. 20, 1916, and (C) 305,886, Apr. 6, 1917.

(A) 1,8-DIHYDROXYANTHRANOL, which is a valuable remedy for psoriasis and other skin diseases, is obtained by the reduction of 1,8-dihydroxyanthraquinone by zinc and acid. (B) 1-Hydroxyanthraquinone is reduced by zinc and acid, yielding 1-hydroxyanthranol, of value as a remedy for psoriasis. (C) Instead of the hydroxyanthraquinones as in (A) and (B), their alkyl ethers may be used. These are readily obtained by boiling 1-nitro- or 1,8-dinitroanthraquinone with alcoholic alkali.

*Compound of lime and tannic acid; Manufacture of a — soluble with difficulty in dilute acids [gastric juice].* Knoll und Co., Chem. Fabr., Ludwigshafen. *Ger. Pat.* 306,979, Nov. 22, 1914.

BASIC calcium tannate is heated for some time at a high temperature; the compound so produced is insoluble in the gastric juice, but is decomposed in the intestine to lime and tannic acid.—L. A. C.

*Dentifrice.* U.S. Pat. 1,275,779. See V.

*Aqueous solutions of hydrocarbons.* *Ger. Pat.* 306,059 See XII.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

*Gelatin [photographic plates]; Reticulation of* —. S. E. Sheppard and F. A. Elliott. *J. Ind. Eng. Chem.*, 1918, 10, 727—732.

THE immediate cause of reticulation of the surface of photographic negatives is the production of restricted tangential dilation, partially arrested, during swelling and drying of the gelatin film. It may be produced by the combined action of both a swelling or softening agent (hot water, potassium iodide) and a hardening agent (chromic acid, mercuric iodide). Reticulation generally starts in one or more regions of the plate and spreads, and it seems that in its earliest stages it involves something like the nucleation of a crystallising solution. In the ordinary development of a negative it is probable that incipient reticulation is proceeding, foreign nuclei being available; different developers and treatments affect the "graininess" of the developed image, and alterations in the swelling equilibrium by changing from an acid to an alkaline medium in operations subsequent to development are liable to increase any incipient reticulation.—W. P. S.

#### PATENT.

*Photography; Substance for use in — and method of making the same.* A. de Brayer, Paris. U.S. Pat. 1,277,048, Aug. 27, 1918. Date of appl., Apr. 20, 1917.

SEE Eng. Pat. 105,920 of 1917; this J., 1918, 224 A.

## XXII.—EXPLOSIVES; MATCHES.

*High explosives; Initial priming substances for* —. G. B. Taylor and W. C. Cope. U.S. Bureau of Mines, Tech. Paper 162, 1917. 32 pages.

Tests were made to ascertain the minimum weights of various primers required to detonate completely trinitrotoluene and tetryl (tetranitromethylaniline), the "sand test" (this J., 1916, 1132) being used to ascertain the degree of detonation of the nitro-compound. Tests made with mixtures of mercury fulminate and various oxygen carriers did not disclose any single property of the oxidiser as having a direct relationship with the priming efficiency of the mixture. Oxidisers varying widely in specific gravity, heat of formation, percentage of available oxygen, character of the non-volatile product of explosion, and general chemical relationship, yielded about equally efficient mixtures. In general, salts of the oxyhalogen acids made efficient mixtures; oxides and nitrates of heavy metals yielding the free metal on decomposition made especially efficient mixtures; heavy metal oxides which are reduced to lower oxides were ineffective. Mercury fulminate was found to be more efficient when uncompressed than when compressed. It seemed also that the sensitiveness of trinitrotoluene to detonation decreased with the degree of compression. The efficiency of primers containing lead azide increased with the pressure of loading. The priming efficiencies of nitrodiazobenzene perchlorate (Herz, Fr. Pat. 450,897; this J., 1913, 627) and hexamethylene-triperoxide-diamine (Von Girssewald, this J., 1912, 1058; 1913, 992) were found to be high, 0.10 gm. and 0.15 gm. of these substances respectively being equal to 0.30 gm. of lead azide or 0.35 gm. of mercury fulminate for detonating trinitrotoluene. The "sand test," in which the ability to break up quartz sand in a "sand bomb" is measured, showed no

parallelism between quantity of sand crushed, and priming efficiency as measured by the relative minimum weights required to cause detonation, but appeared rather to be a test for "strength," the values being in nearly the same order as Berthelot's "characteristic products" ( $QV_0$ , where  $Q$  is the heat of explosion and  $V_0$  the calculated volume of gaseous products of explosion at  $0^\circ\text{C}$ . and 760 mm. from 1 gm. of explosive). The authors consider that the effectiveness of a detonator in practice depends upon explosive energy, that may be separated into an intensity factor ("quickness," depending upon acceleration of the explosive decomposition), and a capacity factor, "strength." The "sand test" may be regarded as a measure of "strength," and the lead-plate test, and the shattering effects on glass tubes, as tests of "quickness." The behaviour of silver acetylide, which although an inefficient primer yet markedly increases the effectiveness of mercury fulminate, is explained by its low "strength" and its great "quickness." Emphasis is laid on the value of the "sand test" for controlling the manufacture of a given priming composition, as although "quickness" is probably the more important of the two factors, this may be regarded as constant for a given material, and the "strength" then becomes a measure of the efficiency of a particular sample.—T. St.

*Detonator compounds containing mercury fulminate; Analysis of —.* P. Nicolardot and J. Boudet. *Ann. Chim. Anal.*, 1918, 23, 192—194.

A portion of the sample is digested with ammonium sulphide solution, first for 2 hrs. in the cold and then for 1 hr. at  $60^\circ\text{C}$ . The mercury is thus converted into mercuric sulphide, which is collected and weighed. The filtrate from the mercuric sulphide is heated with ammonium sulphite and the precipitated antimony sulphide is collected and weighed. The solution from the antimony sulphide is evaporated, and the alkalis weighed as sulphates. A separate portion of the sample is tested for the presence of nitrates and chlorates. The mercuric and antimony sulphides are volatilised after being weighed, since they may contain traces of copper, zinc, or iron; any powdered glass in the sample will be found in the mercuric sulphide precipitate. —W. P. S.

#### PATENT.

*Explosive.* F. G. L. Johnson, London, Assignor to Palmer-Perchlorate Powder Co. of Canada, Ltd., Montreal, Canada. U.S. Pat. 1,276,537, Aug. 20, 1918. Date of appl., Sep. 21, 1916.

SEE Eng. Pats. 14,866 of 1915 and 1480 of 1916; this J., 1916, 1236.

### XXIII.—ANALYSIS.

*Vegetable colouring matter [of myrtle-berry juice]; Use of — as indicator.* C. Marini. *Annali Chim. Appl.*, 1918, 10, 32—36.

The berries of the myrtle (*Vaccinium myrtillus*, L.) contain a red colouring matter which has been shown to be identical with cyanocyanin, the red colouring matter of wine. The slight differences in the behaviour of the two colouring matters, on which are based methods of detecting myrtle-berry juice in wine, are due to the greater proportion of chlorophyll as an impurity in the colouring matter of grape juice. The colouring matter obtained by extracting myrtle berries with alcohol consists of two components, one of which is of a glucosidal character, and is converted into the other by hydrolysis with acids. It is a weak acid and forms

green salts, and may therefore be used as a sensitive indicator in alkalimetry. Red and green test papers may be made from a 90% alcoholic extract of the berries in a manner analogous to that used in the preparation of turmeric paper. (See also J. Chem. Soc., 1918, 1, 518.)—C. A. M.

*Microchemical reactions of metals with rubidium and caesium chlorides.* J. Vermande. *Pharm. Weekblad*, 1918, 55, 1131—1134.

Double chlorides of caesium and rubidium with mercury, silver, copper, bismuth, antimony, tin, lead, nickel, cobalt, cadmium, iron, zinc, manganese, and magnesium have been prepared. They are analogous to the corresponding double salts of potassium and sodium. It was not found possible to prepare double chlorides with arsenic, aluminium, calcium, strontium, barium, potassium, sodium, or lithium.—A. J. W.

*Molybdenum [as ammonium molybdate]; Recovery of — [from residues].* S. Malowan. *Chem.-Zeit.*, 1918, 42, 410.

To recover molybdic acid used as a precipitant in phosphorus determinations, the liquors are precipitated with sodium or calcium phosphate; the yellow precipitate, after being washed and dried, is heated with excess of strong sulphuric acid until solution is complete, and the acid colourless. The cold liquid is poured into 8—10 times its volume of water and the molybdenum precipitated with excess of potassium ferrocyanide. The precipitate is filtered off after 3 hours, and washed with dilute ferrocyanide solution till free from sulphuric acid; it is then dried, separated from the filter, and ignited at a dark red heat to incipient fusion. The cold mass is leached twice with hot water, after which the molybdic acid is extracted with ammonia. The colourless solution is filtered and evaporated to dryness, the residue treated with a little water and hydrogen peroxide and the solution boiled to oxidise any lower oxides. The resulting solution of ammonium molybdate is diluted to a sp. gr. of 1.09 at  $17^\circ\text{C}$ . (i.e., 10%).—W. R. S.

*Determining benzene, etc., in gases.* Davis. See IIa.

*Tar analysis.* Weiss. See III.

*Indigo analysis.* Heinisch. See IV.

*Logwood extracts.* Savini. See IV.

*Oxalic acid.* Harned and Laird. See VII.

*Analysis of spent oxide.* Wentzel. See VII.

*Zinc analysis.* Bertiaux. See X.

*Sodamide and potassamide.* Wöhler and Stang-Lund. See XI.

*Crismer test.* Stewart. See XIXa.

*Decomposition in foods.* Brauer. See XIXa.

*Ergotinine.* Wolter. See XX.

*Separating oxalic from tartaric acid.* Bau. See XX.

*Acetol reaction.* Baudisch. See XX.

*Detonator compounds.* Nicolardot and Boudet. See XXII.



## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

- Akt.-Ges. Kummeler und Matter. Evaporation of liquids. 17,387. Oct. 24. (Switzerland, Feb. 26.)  
 Battersby and Dunlop. Apparatus for separating materials of different densities. 16,782. Oct. 15.  
 Bibb. 16,809. *See* II.  
 Boby, Ltd., and Jennings. Apparatus for separating dust, etc., from gases. 17,462. Oct. 25.  
 Bradley. 17,277. *See* XI.  
 British Thomson-Houston Co. (General Electric Co.). Producing a high vacuum. 17,454. Oct. 25.  
 Bynoe. Drying apparatus. 17,043. Oct. 18.  
 Carpenter. Heating stills, etc. 17,121. Oct. 19.  
 Carruthers and Co., Gourlay, Johnstone, and Young. Filters. 17,308. Oct. 23.  
 Chambers. Grinding-machinery. 17,369. Oct. 24.  
 Fletcher. Conversion of water pastes into oil pastes. 17,493. Oct. 26. (Australia, Aug. 15.)  
 Hewett. Drying fruit, chemicals, etc. 17,157. Oct. 21.  
 Jaslier. Drying apparatus. 16,729. Oct. 14. (Fr., Oct. 20, 1917.)  
 Joubert. Furnaces. 17,124. Oct. 19. (Fr., Oct. 19, 1917.)  
 Leendertz. Process for briquetting refuse. 16,826. Oct. 15. (Holland, Oct. 25, 1917.)  
 Morison. Condensing plant. 16,818. Oct. 15.  
 Mumford. Decolorising and purifying agents, and methods of making same. 16,757. Oct. 14.  
 Peck. Filter apparatus. 17,256. Oct. 22.  
 Pinkney. Mixing-machines. 17,293. Oct. 23. (U.S., Oct. 30, 1917.)  
 Riley. Method for sampling. 16,780. Oct. 15.  
 Soddy. Process for improving or restoring absorptive power of charcoal for gases. 17,071. Oct. 18.  
 Steiger. 16,758. *See* VIII.  
 Sturtevant Mill Co. Pulverising mills. 17,175. Oct. 21. (U.S., Feb. 23.)

#### COMPLETE SPECIFICATION ACCEPTED.

- 14,785 (1917) and 140 (1918). Mauss. Centrifugal separators for separating solid and liquid mixtures. (119,706.) Oct. 23.

### II.—FUEL; GAS; MINERAL OILS AND WAXES: DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

- Ballingall and Dempster. Adaptation of dust, etc., arresting means to plant for manufacture of water gas. 16,711. Oct. 14.  
 Bibb. Manufacture of briquettes. 16,809. Oct. 15.  
 Boby, Ltd., and Jennings. 17,462. *See* I.  
 Kirpotschnikoff and Klasson. Utilisation of peat as fuel. 17,125. Oct. 19.  
 May. Coal-cakes and process of making same. 17,003. Oct. 17.  
 Soc. de Chimie et Catalyse Industrielles. Production of rich gases by means of petroleum residues. 16,820. Oct. 15. (Fr., Feb. 20.)

Soddy. 17,071. *See* I.

Wade (Barrett Co.). Manufacture of bituminous material. 16,847. Oct. 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 13,697 (1917). McClelland. Electrically producing a gas suitable for internal combustion engines, etc. (119,885.) Oct. 30.  
 14,031 (1917). Aiton. Gas generators. (119,676.) Oct. 23.  
 14,541 (1917). Tissier. Carbonisation of wood. (110,360.) Oct. 23.  
 14,725 (1917). Pearse. Distillation of carbonisable materials. (119,700.) Oct. 23.  
 15,134 (1917). Bradley. Retorts for distilling or carbonising coal, carbonaceous and other materials. (119,911.) Oct. 30.  
 15,396 (1917). Glover, West, and West's Gas Improvement Co. Gas producers. (119,723.) Oct. 23.  
 17,613 (1917). Dunstan. Purification of cracked oils. (119,751.) Oct. 23.  
 2372 (1918). Westwood. Treatment of heavy fuel oils preparatory to burning. (119,977.) Oct. 30.

### IV.—COLOURING MATTERS AND DYES.

#### APPLICATIONS.

- Brass. Manufacture of colours and dyes. 16,939. Oct. 17.  
 Ellis (Chemical Works, formerly Sandoz). Production of blue sulphur colorants. 16,761. Oct. 14.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

- Andrew, and D. R. Cotton Mills. Treatment of canvas, etc. 17,119. Oct. 19.  
 Carter, Davis, and Foster. Extraction or decortication of ramie, hemp, jute, etc. 17,014. Oct. 18.  
 Cavanaugh. Soil-proof fabric. 16,724. Oct. 14.  
 Gray and White. Separator for paper pulp. 17,130. Oct. 21.  
 Kanegafuchi Boseki Kabushiki Kwaisha. Treatment of silk fibres, etc. 16,839. Oct. 15.  
 Lacy and Slight. Impregnating articles of mill-board, etc., to render them waterproof. 17,165. Oct. 21.  
 MacDonald, and Spicer and Sons. Vulcanised or parchmentised fibre. 17,501. Oct. 26.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 11,825 (1917). Thompson (Plante). Fabric-dyeing processes. (119,881.) Oct. 30.  
 17,721 (1917). Calico Printers' Assoc., Clegg, Farnworth, and Roberts. Printing fabrics and yarns. (119,947.) Oct. 30.  
 499 (1918). Livesey and Haworth. Machines for printing yarn. (119,773.) Oct. 23.  
 1592 (1918). Dudley. Dyeing machine. (113,094.) Oct. 23.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

- Amber Size and Chemical Co., and Weygang. Production and use of compositious possessing alkaline properties. 16,825. Oct. 15.

Ashcroft. Manufacture of anhydrous magnesium and/or calcium chlorides. 16,701. Oct. 14.

Ashcroft. Production of anhydrous magnesium chloride or anhydrous carnallite. 16,860. Oct. 16.

Ashcroft. Manufacture of oxides or salts of tungsten, or metallic tungsten. 17,313. Oct. 23.

Ashcroft. Manufacture of oxides or salts of metals, or of metals, or of non-metals, from ores, residues, etc. 17,314. Oct. 23.

Ashcroft. Manufacture of aluminium or its oxides or salts. 17,315. Oct. 23.

Barron. Compressed Epsom salts. 16,733. Oct. 14.

Calvert. Production of cyanogen compounds, ammonia, etc. 17,167. Oct. 21.

Dutt and Dutt. Manufacture of magnesium sulphate. 17,076. Oct. 18.

Dutt and Dutt. Manufacture of sodium carbonate and alumina. 17,077. Oct. 18.

Dutt and Dutt. Manufacture of potassium carbonate and alumina. 17,078. Oct. 18.

Dutt and Dutt. Manufacture of potassium salts. 17,079, 17,080, and 17,081. Oct. 18.

Haslup. Fixation of atmospheric nitrogen and production of ammonia, etc. 17,364. Oct. 21.

Haslup. Fixation of atmospheric nitrogen in a blast furnace. 17,365. Oct. 21.

Kestner. Filling material for Glover towers, etc. 17,065. Oct. 18.

Parkes and Pearson. Manufacture of nitrogen acids from ammonia. 16,827. Oct. 15.

Pickard and Shaw. Manufacture of nitric acid. 16,884. Oct. 14.

Quain. Apparatus for producing ozone. 17,017. Oct. 18.

Rockefeller Institute. 17,171 and 17,172. See XX.

Welch. Producing sulphuric acid. 17,226. Oct. 22.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,711 (1917) and 3152 (1918). Llewellyn, Spence, and Spence and Sons. Production of aluminous compounds. (119,924.) Oct. 30.

1211 (1918). Baker. Recovery of ammonium chloride from ammoniacal liquor with the extraction of cyanides, etc. (119,971.) Oct. 30.

### VIII.—GLASS; CERAMICS.

#### APPLICATIONS.

Pike. Magnesite refractories. 16,742. Oct. 14.

Steiger and Steiger. Gas-fired shaft kilns for burning or roasting non-sintering substances. 16,758. Oct. 14.

Vergniaud. Manufacture of crucibles. 17,253. Oct. 23.

Vergniaud. Drying crucibles artificially. 17,254. Oct. 23.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

De Bas. Production of artificial stone. 17,329. Oct. 23. (Holland, Apr. 2, 1917.)

Lea. Apparatus for mixing cement aggregates, etc. 16,856 and 16,857. Oct. 16.

Smith. 17,143. See XIII.

Steiger. 16,758. See VIII.

Wade (Barrett Co.). 16,847. See II.

#### COMPLETE SPECIFICATIONS ACCEPTED.

749 (1918). Chance and Hunt, Holley, and Webb. Acid-resisting cement. (119,966.) Oct. 30.

992 (1918). Surle, and Suiresstone, Ltd. Insulating materials for cold storage. (119,783.) Oct. 23.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Ashcroft. 17,313, 17,314, and 17,315. See VII.

Blair and Clapp. Manufacture of iron and steel. 17,509. Oct. 26.

Boving. Treatment and reduction of ores. 17,227. Oct. 22.

Cooper Co. Alloy. 16,804. Oct. 15. (U.S., Oct. 15, 1917.)

Harvey, and Harvey Gas Furnace Co. Regenerative or recuperative gas furnaces for heating or melting metals. 17,068. Oct. 18.

Haslup. 17,365. See VII.

Lang. Zinc cyanide process of extracting gold from quartz, etc. 16,985. Oct. 17.

Morrison and Thompson. Coating surfaces of metal with lead or its alloys. 16,695. Oct. 14.

Munden and Shilton. Crucible furnaces. 16,937. Oct. 17.

Price. Crucible furnaces for melting aluminium, etc. 17,151. Oct. 21.

Shedlock. Manufacture of alloys and electrodes therefor. 16,739. Oct. 14.

Soc. Lorraine des Anc. Etabl. de Diétrich et Cie. Measuring hardness of bodies. 17,328. Oct. 23. (Fr., Nov. 12, 1917.)

Stevensen. Amalgam of mercury. 17,438. Oct. 25.

Williams. Crucible furnace. 16,772. Oct. 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,531 (1917). Procter. Soldering mixtures. (119,688.) Oct. 23.

803 (1918). Tylor and Sons, and others. Furnaces for smelting and refining metallic swarf, etc. (119,779.) Oct. 23.

8874 (1918). Carpmacel (A.G. f. Autogene Aluminium Schweissung). Autogenous welding of aluminium or its alloys. (120,005.) Oct. 30.

10,379 (1918). Greenwood and Batley, and Clegg. Annealing furnaces. (119,841.) Oct. 23.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Bradley. Electrical treatment of gases. 17,277. Oct. 23. (U.S., June 21, 1916.)

Cave. Alkaline accumulators. 17,338. Oct. 23. (Fr., Nov. 28, 1917.)

Gerrard and Newbery. Electrodes. 16,762. Oct. 14.

Oldham and Oldham. Galvanic batteries. 17,258. Oct. 22.

Shedlock. 16,739. See X.

#### COMPLETE SPECIFICATION ACCEPTED.

13,697 (1917). McClelland. See II.



### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

- Fletcher. 17,493. *See* 1.  
 Holzapfel. Manufacture of coating or preservative compositions. 17,375. Oct. 24. (Norway, May 3.)  
 Ligterink. Linoleum substitute. 17,471. Oct. 25.  
 Nevello. Paints. 17,228. Oct. 22.  
 Smith. Aircraft dope. 17,101. Oct. 19.  
 Smith. Varnish for wood preservatives. 17,143. Oct. 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 15,022, 15,023, and 15,024 (1917). Morris and Nevill. Manufacture of white pigments. (119,711, 119,712, 119,713.) Oct. 23.  
 12,236 (1918). Rogers. Manufacture of oil pastes from wet precipitated pigments and separation of free water therefrom. (118,289.) Oct. 30.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 4998 (1917). Bennett and Mellowes. Substitutes for indiarubber, vulcanite, etc. (119,878.) Oct. 30.  
 14,965 (1917). Watkins. *See* XV.

### XV.—LEATHER: BONE; HORN; GLUE.

#### APPLICATIONS.

- Bendixen. Tanning fish skins. 17,486. Oct. 26. (U.S., Dec. 29, 1914.)  
 Blanc. Manufacture of tanning liquors. 16,968. Oct. 17. (Fr., Sep. 17, 1917.)  
 Edwards, Kay, and Ward. Treatment of hides, skins, etc. 17,475. Oct. 26.  
 Mumford. Manufacture of glue, etc. 17,185. Oct. 21.  
 Sayer. Leather substitute. 17,431. Oct. 25.

#### COMPLETE SPECIFICATION ACCEPTED.

- 14,965 (1917). Watkins. Consolidation of leather scrap or refuse with crude or waste rubber. (119,902.) Oct. 30.

### XVI.—SOILS; FERTILISERS.

#### COMPLETE SPECIFICATION ACCEPTED.

- 7843 (1918). Furse. Manufacture of manure from refuse. (119,834.) Oct. 22.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATIONS.

- Chemical Works, formerly Sandoz. Manufacture of ferments, etc. 17,193. Oct. 21. (Switzerland, Oct. 20, 1917.)  
 Kanegafuchi Boseki Kabushiki Kwaisha. Production of proteolytic enzymes. 16,840. Oct. 15.

- Scott. Process for brewing beer, etc. 16,876. Oct. 16.

#### COMPLETE SPECIFICATION ACCEPTED.

- 7660 (1918). Boulard. Brewing of beer. (119,833.) Oct. 23.

### XIX.—FOODS: WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

- Alcock, Brander, and Wagstaff. Treatment of frozen meat. 16,749. Oct. 14. (Australia, Sep. 5.)  
 Cullen. Manufacture of egg substitute. 17,399. Oct. 24. (New Zealand, Nov. 2, 1917.)  
 Greville. Food products. 17,221. Oct. 22.  
 Hewett. 17,157. *See* I.  
 Imray (Soc. Chem. Industry in Basle). Manufacture of vitamin preparations. 16,830. Oct. 15.  
 Leendertz. 16,826. *See* I.  
 Nicholson. Treatment of sewage sludge, etc. 16,740. Oct. 14.  
 Quain. Sterilisation of water, etc. 17,048. Oct. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

- 14,752 (1917). Callebaut. Regenerating damaged cereals, seeds, and flour, especially maize. (110,364.) Oct. 23.  
 15,624 (1917). Sleeman. Flour or meal. (119,923.) Oct. 30.  
 12,499 (1918). Mapleton's Nut Food Co., and Mapleton. Food preparation. (120,018.) Oct. 30.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

- Imray (Soc. Chem. Industry in Basle). 16,839. *See* XIX.  
 Rockefeller Institute for Medical Research. Arseical compounds 17,172 and 17,173. Oct. 21. (U.S., Jan. 28 and June 12.)

#### COMPLETE SPECIFICATION ACCEPTED.

- 573 (1918). Myddleton. Manufacture of *p*-oxyphenylarsinic acid. (119,964.) Oct. 30.

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATIONS.

- Bloch and Renwick. Colour-sensitised photographic materials. 16,845 and 16,846. Oct. 15.  
 Kelley. Photographic films. 17,191. Oct. 21.  
 Marchant and Co. Photographic printing. 16,755. Oct. 14. (Australia, Sep. 11, 1917.)

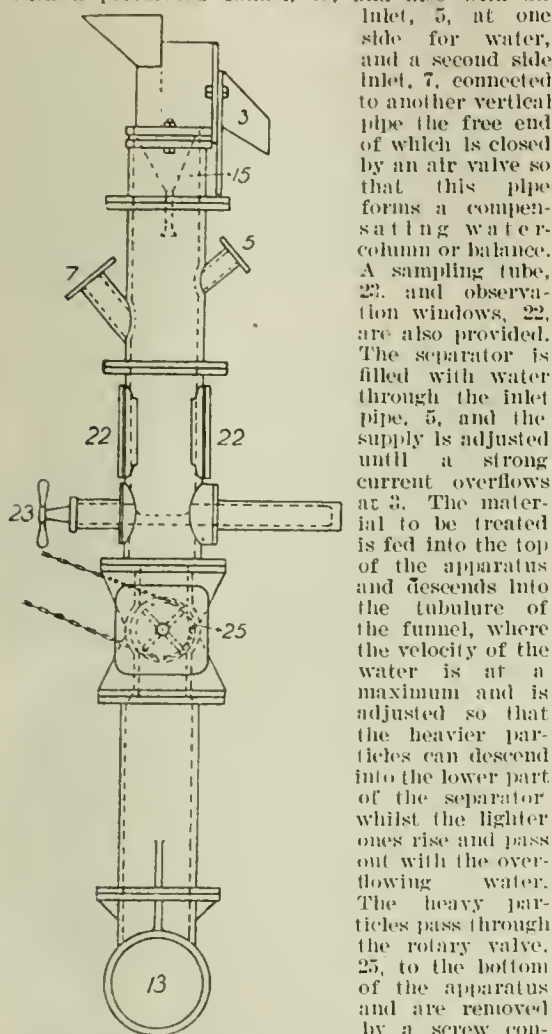
#### COMPLETE SPECIFICATION ACCEPTED.

- 18,639 (1916). Watkins. Photomicrography. (119,876.) Oct. 30.

**I.—GENERAL ; PLANT ; MACHINERY.**

*Hydraulic separation as applied to the recovery of fine coals, shales, and clays.* J. M. Draper. Trans. Ceram. Soc., 1917-18, 17, 213—225.

The hydraulic separator designed by the author consists of a vertical tube, provided near the top with a perforated funnel, 15, and also with an



inlet, 5, at one side for water, and a second side inlet, 7, connected to another vertical pipe the free end of which is closed by an air valve so that this pipe forms a compensating water-column or balance. A sampling tube, 23, and observation windows, 22, are also provided. The separator is filled with water through the inlet pipe, 5, and the supply is adjusted until a strong current overflows at 3. The material to be treated is fed into the top of the apparatus and descends into the tubulure of the funnel, where the velocity of the water is at a maximum and is adjusted so that the heavier particles can descend into the lower part of the separator whilst the lighter ones rise and pass out with the overflowing water. The heavy particles pass through the rotary valve, 25, to the bottom of the apparatus and are removed by a screw conveyor, 13.

When dirty coal is led into the apparatus any shale or heavy mineral particles pass to the screw conveyor; the coal and fine clay pass out with the water and may be separated by passing this portion of the product over a No. 100 sieve, the clay and coal dust passing through the sieve and the remainder of the coal being washed down the sieve into a receiver. When a clay is fed into the apparatus the heavier impurities (pyrites, etc.) are separated and pass into the conveyor. A preliminary treatment with sieves is desirable where the material is composed of widely different sizes, the various grades being then dealt with in independent separators placed side by side.

—A. B. S.

*Extraction of liquids; Apparatus for — in towers filled with Raschig's rings.* F. Raschig. Z. angew. Chem., 1918, 31, 183—185.

The introduction of Raschig's rings (Eng. Pat. 6288 of 1914 and Ger. Pat. 292,622; this J., 1914,

907; 1916, 916) overcomes certain objections to the application of the counter-current principle to processes in which a liquid is purified by treatment with another liquid immiscible with it. The apparatus consists of an iron cylinder, 8 m. high and 60 cm. diam., in which a screen is fixed 1 m. from the base. On this screen rests a column (6 m. in height) of Raschig's rings (25 mm. diam.). An inlet tube (2 m. length and 30 cm. diam.) is fixed in the middle of the cylinder at the top, and serves to admit the heavier liquid, whilst the lighter liquid is introduced through a long upright tube which enters the cylinder just below the screen. The heavier liquid sinks ultimately to the bottom of the chamber below the screen, whence it passes out through a long upright tube, of such a height that the heavier liquid in this tube balances the column of the mixture in the cylinder. The lighter liquor overflows through an outlet in the side of the cylinder near the top. The runnings are always clear, any emulsion or other deposit settling on the rings, which are cleaned when necessary, by emptying the apparatus and running in a suitable solvent. This apparatus may be advantageously applied to the purification of mineral oils by treatment with sulphuric acid, the recovery of aniline from weak solutions by extraction with benzene, the extraction of acetic acid with ether, and of alcohol from dilute solutions with benzene.—S. S. A.

*Decolorisation; Determination of the minimum quantity of decoloriser required for complete — by the adsorption formula.* T. Tadokoro. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 405—411.

In calculating the amount of a decolorising agent required for complete decolorisation by means of the adsorption formula,  $\log \frac{x}{m} = \log K + \frac{1}{n} \log C$ , five or more different quantities of the decolorising agent were used, and the results plotted,  $\log \frac{x}{m}$  being taken on the ordinate and  $\log C$  measured on the abscissa. The point where a line connecting these five or more points cut the ordinate gave the value of  $\log \frac{x}{m}$  in the case of  $\log C=0$ ;  $\frac{x}{m}$  indicates the ratio between the quantity of decolorising agent and the total amount of colouring matter completely absorbed, and from this ratio the value of  $m$  may be calculated.—C. A. M.

*Electrostatic precipitation.* Eschholz. See XI.

**PATENTS.**

*Furnaces; Regenerative —.* J. R. C. August and H. V. Hooson, Halifax. Eng. Pat. 119,495, 26.9.17. (Appl. 13,817/17.)

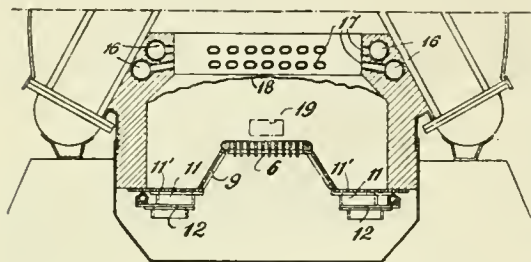
The furnace gases pass through parallel horizontal flues, arranged in zigzag form, to the uptake, and the secondary air is preheated by passing in the reverse direction through flues alternating with the gas flues. The secondary air is controlled by dampers immediately before meeting the combustible gas. (Reference is directed, in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 7210 of 1899, 186 of 1902, 6924 of 1911, and 101,983; this J., 1916, 1254.)

—W. F. F.



**Furnaces.** A. J. M. A. R. van der Does de Bijle, The Hague. Eng. Pat. 115,644, 9.5.18. (Appl. 7813/18.) Int. Conv., 2.5.17.

COAL is fed on to a non-perforated "dead" grate and is moved forward by a reciprocating plunger, 19, on to a grate, 6, of normal form, and thence forward to a perforated plate, the total area of the air passages of which is about one-tenth of those of the normal grate. This grate area is connected, along its full length, on both sides, by



ribbed plates, 9, to perforated plates, 11', at a lower level. At the rear end of the plates 11', recesses, 11, with closure plates, 12, are provided for the discharge of ash. Supplementary air passages, 16, are provided in the setting, with jets, 17, directed on to the top of the fire, 18.—W. F. F.

**Furnaces; Gas heated** —. H. J. Yates, Birmingham, S. N. and E. R. Brayshaw, Manchester. Eng. Pat. 119,553, 16.10.17. (Appl. 14,963/17.)

THE furnace is provided with a number of transverse passages beneath the floor, each having a gas burner at one end, and projecting upwards at its farther end some distance into the furnace. The hot gases, after passing through the furnace, pass through passages at the floor level into a vertical exhaust chamber above the ends of the transverse passages containing the burners. The gases pass upwards through this chamber to the exhaust, and the air supply for the burners passes downwards through pipes in the chamber and is thereby preheated.—W. F. F.

**Furnace.** W. J. Bagot, Bridgewater, Mass., Assignor to The Stanley Works, New Britain. Conn. U.S. Pat. 1,277,356, 3.9.18. Appl., 24.3.17.

ARTICLES which have been heated on a furnace hearth are pushed forward into an inclined shoot formed by a number of hollow spaced inclined members filled in with hollow blocks so as to form a flat inclined surface. The discharging edge of the hearth is supported by a transverse member which is braced by the hollow inclined members. A cooling medium is circulated through the hollow blocks and members, and is discharged through the inclined surface.—W. F. F.

**Drying apparatus.** L. B. Clarke, London. Eng. Pat. 119,156, 6.12.17. (Appl. 18,093/17.)

THE trays which support the material have deflector plates secured to the underside, so that when several trays are piled in a stack they may be inserted into or withdrawn from the drying chamber as one unit. Additional baffle-plates are provided, so that the direction of the air through the trays may be reversed.—W. H. C.

**Dryer.** L. C. Reed, Dallas, Tex. U.S. Pat. 1,276,957, 27.8.18. Appl., 21.2.18.

THE materials are passed successively in alternate directions through a superposed series of jacketed horizontal pipes provided with internal conveyor

worms and connected at alternate ends. A heating medium is passed through the jackets from a manifold at one side and discharged into a manifold at the other side of the series of pipes.—W. H. C.

**Mills for crushing, grinding, and mixing colours, chemicals, drugs, ores, and the like; Means for covering in and discharging** —. T. Breakell, Wirksworth, Derby. Eng. Pat. 119,367, 4.1.18. (Appl. 227/18.)

A GRINDING mill in which an eccentric vertical pestle is rotated by frictional contact with a rotating mortar, or *vice versa*, is enclosed in a casing of which the base forms a bed-plate for the mortar, and the removable cover contains the vertical bearing for the pestle. The cover may be made in two parts, one of which can be lifted off for feeding material or for inspection. An outlet opening in the bottom of the mortar is closed by a conical stopper of the same material, rotating with it and supported in a footstep bearing carried by a pivoted lever. The lever may be operated during rotation of the mortar, to withdraw the stopper.—W. F. F.

**Gases and/or vapours; Means for carrying out catalytic or contact reactions between** —. C. S. Roy, and T. Morson and Son, Ltd., London. Eng. Pat. 119,639, 26.6.18. (Appl. 10,536/18.) Add. to 116,151.

IN the apparatus described in the principal patent (this J., 1918, 397 A), the gauze catalyst is mounted in a number of separate rings each having pins projecting radially at opposite ends of a diameter. The pins engage in slots in short inner tubes which fit into the reaction tube so that the rings may be rotated till their planes are at any desired angle with the axis of the tube. The area of catalyst in the path of the gases is thereby varied. All the rings may be connected by links so that they may be operated simultaneously by external means.

—W. F. F.

**Mixing or stirring means.** N. D. Nielsen, Elyria, Ohio. U.S. Pat. 1,276,254, 20.8.18. Appl., 22.3.18.

A VERTICAL cylindrical tank has a saucer-shaped bottom with a central opening to which a valved elbow pipe is attached. A vertical shaft passes through a stuffing-box at the angle of the elbow pipe and extends upwards into the tank. A propeller having a diameter of less than one-third that of the tank is fixed to the upper end of the shaft so that the blades are partly above and partly below the line of junction between the vertical side and the bottom of the tank. The shaft is driven by gearing from below and as the propeller rotates it drives the liquid in the tank partly vertically upwards and partly horizontally outwards, causing thorough mixing.—W. H. C.

**Liquids; Apparatus for separating** —. Method of separating associated liquids. C. W. McKibben, Houston, Tex. U.S. Pats. (A) 1,276,386 and (B) 1,276,387, 20.8.18. Appl., 24.11.17 and 22.1.18.

(A) EMULSIONS of liquids of different sp. gr. and electrical resistance are separated by means of an apparatus consisting of a vertical vessel serving as a conduit for the emulsion and provided with means for separating it. Below the conduit is a settling chamber for the heavier liquid, provided with electrodes connected with an electro-responsive device whereby a valve is automatically opened when the electrical resistance of the contents of the chamber reaches a predetermined point. (B) Associated liquids are heated to a temperature below the b.pt. of the mixture, and made to pass through

a path between two active electrodes at sufficient velocity to prevent re-entanglement of the nascent streams formed between the two electrodes. Means are provided for the automatic reduction of the effective force of the applied current when the streams begin to form.—C. A. M.

*Fire-extinguishing and fire-proofing composition.* G. E. Ferguson, New York, Assignor to Pyrene Manufacturing Co. U.S. Pat. 1,276,742, 27.8.18. Appl., 12.12.14.

An aqueous solution of potassium carbonate, trisodium phosphate, and borax.—W. E. F. P.

*Refrigerating-machine.* F. G. Keyes, Boston, Mass., Assignor to National Automatic Refrigerator Co. U.S. Pat. 1,277,085, 27.8.18. Appl., 25.4.17.

An intermediate condenser is connected with a distilling chamber and a refrigerating chamber, the walls of the latter being composed of Monel metal or other poor conductor of heat.—W. H. C.

*Low temperatures; Production of —.* P. Langer, Aix-la-Chapelle. Ger. Pat. 307,359, 19.5.14.

In a refrigerating apparatus, the gas or vapour, after passing through one or more expansion nozzles, and through the refrigerating tubes, in which it absorbs heat, is passed gradually into a wider tube, the gradual decrease in velocity causing a corresponding increase in temperature and pressure. The gas or vapour is then led through a cooler and, by means of a compressor, the pressure is again brought to its initial value. After passing through a further cooler, the gas or vapour is led back to the expansion nozzles.—L. A. C.

*Centrifugal liquid machine.* B. R. Wright, Poughkeepsie, Assignor to The De Laval Separator Co., New York. U.S. Pat. 1,277,676, 3.9.18. Appl., 15.12.13.

The machine consists of a rotary bowl provided with a central vertical receiving passage. A stem projects upwards within and spaced from the inner wall of the receiving passage, and a screw conveyor is mounted in the annular space between the stem and the receiving tube. Vents are provided within the radius of discharge from the bowl for the escape of air or gases tending to retard the flow of the liquid through the apparatus.—J. H. P.

*Cooling liquids or exposing them to air; Apparatus for —.* A. Smallwood, London. Eng. Pat. 119,571, 23.11.17. (Appl. 17,265/17.)

*Separating solids from liquids; Process and apparatus for —.* P. T. Sharples, West Chester, Pa., U.S.A. Eng. Pat. 119,288, 27.9.18. (Appl. 13,965/17.)

SEE U.S. Pat. 1,232,104 of 1917; this J., 1917, 917.

*Distillation of liquids; Method and apparatus for —.* O. Stalhane and O. O. Kring, Ludvika, Sweden. U.S. Pat. 1,277,659, 3.9.18. Appl., 20.12.16.

SEE Eng. Pat. 110,821 of 1916; this J., 1917, 1262.

*Tunnel furnaces.* Eng. Pat. 119,366. See VIII.

## II.A.—FUEL; GAS; MINERAL OILS AND WAXES.

*Coal; Diffusion of oxygen through stored —.* S. H. Katz. U.S. Bureau of Mines. Tech. Paper 170, 1917, 47 pages.

The effect of the size of the coal pieces and the proportion of voids in a coal pile, on the rate of diffusion of oxygen into the pile has been deter-

mined. The method consisted of interposing a layer of weathered coal, composed of pieces of known size, between an atmosphere of air on one side and nitrogen on the other. By analysing the gases periodically on both sides, the progress of diffusion was noted. The apparatus consisted of a vertical cylinder made in two halves, the upper half fitting into a water lute carried by the bottom half and thereby forming a closed vessel. The cylinder contained three compartments, one above the other—the middle one carrying the coal on a wire mesh support. The capacity of the upper and lower compartments was about 37 litres (8½ galls.) each, the depth of the coal usually being 8 inches. The sizes of coal ranged between 1 to 2 inch cubes and pieces passing through a sieve of 40-mesh (per linear inch) and remaining on 60-mesh. Tests were also made with coal of all sizes from a storage pile. The results indicate that the principles stated in Fick's law of diffusion are applicable to the diffusion of oxygen through the atmosphere in the voids of broken coal. If the proportion of voids in a mass of coal pieces is varied, the time required for a definite diffusion of oxygen increases approximately in inverse proportion to the percentage of voids. In storing coal in the air, factors other than diffusion may be accelerated or retarded by changes in voids; but if diffusion alone be considered, mixing the sizes of coal will reduce the oxygen entering the pile by diffusion and thus reduce oxidation from this source.—T. F. E. R.

*Coal; Carbonisation of —.* W. Savage. Chem. and Met. Eng., 1918, 19, 579—582.

In the manufacture of metallurgical coke a satisfactory product has been obtained in the beehive oven from coals containing up to 32% of volatile substances, whilst with by-product ovens the best results are given by harder coals with 23 to 28% of volatile substances. Coal containing more than 4% of moisture gives inferior results. In Smith's process (U.S. Pat. 1,177,727 and 1,224,424 of 1915; this J., 1916, 625; 1917, 636), an intermediate soft semi-coke containing 16 to 20% of volatile matter is made into briquettes with hard pitch under pressure and carbonised at suitable temperatures. The product, termed "carbo-coal," has been tested by several railway companies and by the U.S. Navy, and found to burn more readily than coke. With a rate of combustion of 27 lb. per sq. ft. of grate area per hour, it evaporated 12.8 lb. of water per lb. of fuel; and with 100 lb. combustion rate, 8.5 lb. of water. In working the process any grade of bituminous coal is reduced to about 12-mesh size, and fed continuously through a cylindrical retort containing agitators rotating at about 4 revolutions per hour. The retort, which is of refractory material, is heated to about 900° F. (480° C.) by the combustion of the gases evolved in the process, and, after distillation for about an hour, the material is cooled in a hopper, and then conveyed to crushing and pulverising machinery. The finely-divided semi-coke is incorporated with several per cent. of pitch in a mixing machine heated by direct steam, and the hot mixture is transferred to a briquetting machine of the roller type. The resulting briquettes are conveyed while hot to the carbonising retorts. As a rule only 4 to 5% of volatile matter is left in the residual "carbo-coal," this being sufficient to allow the product to burn readily in an open fireplace. From 2000 lb. of coal thus treated, 1440 lb. of "carbo-coal" briquettes are obtained and 560 lb. of gases and vapours, of which 200 lb. are permanent gases used for heating the retort. The vapours consist of 20 lb. of ammonia, etc., and 250 lb. of tar oils including benzol, toluol, naphthalas, "motor spirits," creosote



oils, tar acids, pitch, etc. The tar obtained in the primary distillation of the coal has sp. gr. 1 to 1.06. It contains no naphthalene, anthracene, or phenol, but is rich in oils, tar acids, and cresols. The following table shows the liquid products yielded by one ton of coal in comparison with the amounts of by-products from the coke oven:—

	Distillation temperature	By-product coke oven		Carbo-coal, first distillation		Carbo-coal, second distillation	
		galls.	%	galls.	%	galls.	%
Light oil ..	Up to 77	0.27	3.47	1.58	6.60	0.003	0.05
Middle oil ..	77—110	0.44	5.85	3.29	13.70	0.036	0.60
Cresosote oil ..	110—132	0.78	10.37	3.11	12.95	0.126	2.10
Heavy oil ..	132—182	1.26	16.81	8.88	37.00	2.485	41.42
Pitch ...	—	4.66	62.18	6.90	28.75	3.290	54.83
Loss ...	—	0.09	1.32	0.24	1.00	0.060	1.00
		7.50	100.00	24.00	100.00	6.000	100.00

—C. A. M.

*Coal-gas manufacture: Technical scale tests on efficiency of — in Koppers' chamber ovens.*  
K. Bunte and E. Terres. J. Gasbeleucht., 1918, 61, 423—436, 445—450.

A SERIES of efficiency tests were carried out in 1912 at the Vienna-Leopoldau Gas Works on the Koppers system of chamber ovens. The plant consisted of 72 chamber ovens in ranges of 9. During the tests 5 ranges were working. Each chamber was 10.3 m. long and had a breadth of 540 mm. at the discharging end, tapering to 480 mm. at the charging end. Four regenerators were built under each oven for heating the cold producer gas and secondary air by heat from the waste gases. The ovens were supplied with producer gas from an external generating plant consisting of 12 Kerpely-Marischka producers, five of which were at work. The water jacket steam boilers on these producers generated all the necessary steam. The volume of producer gas made was calculated on a carbon balance sheet obtained from analyses. It was shown by one test that 11.17 kilos. of coke was burned in the producer during the carbonisation of 160 kilos. of coal and also that 663 calories had to be supplied as producer gas to the ovens to carbonise one kilo. of coal. The following thermal balance sheet was obtained for a 1-day test on the producers.

<i>Energy supplied to the producer:—</i>		%
Heat of combustion of the coke ...	96.97	
Heat in steam supplied under the grate ...	3.03	
	100.00	
<i>Energy subtracted from producer:—</i>		%
Calorific value of gas ...	79.13	
Sensible heat in dry gas ...	0.46	
Latent and sensible heat in moisture in gas ...	1.80	
Heat used in evaporating feed water ...	15.56	
Heat in unburnt combustible matter in clinker ...	0.71	
Heat in unburnt combustible matter in flue dust ...	0.20	
	97.86	
Heat lost by radiation and conduction (by difference) ...	2.14	
	100.00	

The percentage composition of the producer gas was:—CO<sub>2</sub>=3.65, O<sub>2</sub>=0.2, CO=29.12, H<sub>2</sub>=9.88, CH<sub>4</sub>=0.2, N<sub>2</sub>=56.95. The thermal balance sheet for the ovens showed that the heat doing useful work in the ovens amounted to 71.1% of the heat of combustion of the coke used in the producers, whereas the producer gas itself contained 81.6% of the heat of combustion of the coke used in its production. The difference represents heat lost in the waste gases and by radiation from the ovens.

—T. F. E. R.

*Gas economics, with special regard to carbonisation and the treatment and disposal of residual products.* J. Dickson. Scottish Junior Gas Assoc. Gas J., 1918, 144, 187—189.

THE author deals with the steaming of coal charges during carbonisation and the necessary precautions to be observed in order to obtain success. By steaming vertical retorts of the continuous-intermittent type a make of 16,193 cub. ft. gas per ton has been obtained. After oil washing the gas had a calorific value of 450—460 B.Th.U. and the composition H<sub>2</sub> 50.8, CH<sub>4</sub> 19.7, unsaturated hydrocarbons 2.2, CO 14.1, O<sub>2</sub> 0.8, CO<sub>2</sub> 5.6, and N<sub>2</sub> 6.8%. The coal passes the pasty stage sooner with steaming. A survey is given of some recent progress in gas works in coke manufacture, boiler firing and fuel economy, ammonia recovery, tar dehydration, and benzol recovery.—T. F. E. R.

*Naphthalene; Estimation of — in coal gas.* H. G. Colman. Gas J., 1918, 144, 231—232.

THE author describes additional precautions or modified methods to be employed in estimating naphthalene in coal gas by the method previously described by Colman and Smith (this J., 1900, 128). The gas is passed through a series of four wash-bottles, the first of glass throughout containing a 10% solution of citric acid to retain any ammonia in the gas. The second wash-bottle contains 100 c.c. of N/20 picric acid, the third 50 c.c. of the same solution, and the last is left empty to retain spray. The gas must have no access to indiarubber during or prior to its passage through the train (see this J., 1900, 813). The picric acid solution is standardised with N/10 caustic soda solution, using lacmoid or phenolphthalein as indicator. The gas (about 10 cub. ft.) is passed through the apparatus at a rate not exceeding 1 cub. ft. per hour, the volume being corrected to 60° F. (15.5° C.), and 30" pressure. After passage of the gas, the contents of the third and fourth bottles of the train are washed into the second bottle, which is then closed by a tight-fitting rubber stopper fitted with glass stopcock or glass device answering the same purpose. The wash-bottle is evacuated until the naphthalene picrate rises to the surface. The stopcock is closed and the contents of the bottle heated to boiling with occasional shaking, and then allowed to cool with occasional shaking. The volume of the cold mixture is measured, the naphthalene picrate is separated by filtration through a dry filter paper, the first few c.c. of the filtrate being rejected, and 100 c.c. of the filtrate is titrated with N/10 NaOH solution. If  $v$  represents the difference between the volume of N/10 NaOH required to neutralise the total volume of N/20 picric acid solution originally taken, and that required for the neutralisation of the total volume of picric acid solution after heating and filtering, then: Grains of naphthalene per 100 cub. ft. =  $\frac{v \times 19.75}{\text{volume of gas passed}}$ .

—J. S. G. T.

*Gas; Relative efficiency in use of different grades and compositions of* —. First Report of Research Sub-Committee appointed by the Gas Investigation Committee, Inst. of Gas Eng., 9.5.18, with supplementary matter 9.8.18. Gas J., 1918, 144, 235–249.

The report deals with the relative efficiencies of various grades of gas in existing appliances for use with gas, no adjustments additional to those furnished by the makers being, in general, introduced.

*Ring burners.* Gases varying in calorific value from 530 B.Th.U. gross and 480 net to 281 B.Th.U. gross and 214 net were employed. The experiments with ring burners are the most complete of the series. The following conclusions, amongst others, are drawn: (a) Unless interfered with by very poor aeration or too close proximity of the containing vessel, within wide limits, the efficiency of a burning ring is almost independent of the rate at which the gas is burnt. (b) Flame contact is essential for high efficiency. (c) The distance of the bottom of the vessel, in which water is being boiled, from the burner requires careful adjustment in order to secure maximum efficiency. (d) Provided suitable adjustments are made, the efficiency is practically independent of the composition, content of inert gases, or calorific value of the gas. (e) A wide variation in grade or composition of gas can be made with reference to other uses, without impairing the efficiency of ring burners now in use.

*Upright low-pressure incandescence lighting burners.* Experiments were carried out with gases varying in calorific value from 615 B.Th.U. gross, 552 net, to 314 B.Th.U. gross, 285 net. The candle power developed in a horizontal direction was alone measured. The different consumptions of the burner were adjusted by varying the pressure at the injector of the burner. The main conclusions derived are:—(a) With gases of about 450 B.Th.U. gross and below, the control possible to obtain by varying the consumption and aeration was not sufficient to give satisfactory illumination. (b) With the burner used, a gas of about 550 B.Th.U. gross maximum to 500 B.Th.U. net minimum gave the best performance, both as regards efficiency and total illumination. (c) The maximum attainable illumination was found at about 525 B.Th.U. gross.

*Gas fires.* The whole series of experiments with gas fires is not yet complete. The calorific value of the gases employed ranged from 559 B.Th.U. gross, 503 net, to 385 B.Th.U. gross, 347 net. The same fire was not used throughout the series of tests. The main conclusions are as follows:—(a) With each gas fire, there is a certain consumption which for the gas used gives the maximum radiant efficiency. (b) If the radiants are heated to less than half their height the efficiency suffers severely. (c) Existing gas fires vary very considerably in their ability to adapt themselves to gases of different grades.—J. S. G. T.

*Motor fuels; Valuation of* —. H. Moore. Automobile Eng., Sep., 1918, 245–248.

The methods at present in vogue for the analysis of motor spirits are not fully satisfactory for the testing of mixed fuels such as will probably be extensively used in the future. The conditions existing in the engine and in the carburettor through which the fuels have to pass are the main factors in deciding the tests to which a motor spirit should be subjected. *Specific gravity* has little value as an indication of the quality of a motor spirit. The *viscosity* of all common fuels is very small, but this value may become important with future fuels. Heavy oils for Diesel engines may

require suitable preheating to maintain the viscosity within reasonable limits. The *cold test* is important for fuel mixtures containing solid hydrocarbons in solution. For aero-engine fuels it is essential that the temperature at which solids separate shall be very low. The *coke test* consists in volatilising the fuel in a platinum crucible and coking any residue remaining. Some of the heavier coal-tar distillates present in mixed fuels sold as “substitutes” yield appreciable quantities of coke. The *fractional distillation test* gives considerable information as to the ingredients present in a fuel mixture. *Latent heat of vaporisation* is an indication of the cooling effect produced in the carburettor, and of the desirability or otherwise of hot air intake. *Iodine and bromine values* give information as to the method of preparation of petroleum spirits, but are not a direct indication of the behaviour of a fuel in the engine. The *ultimate analysis* indicates the nature of the products present in the fuel, and allows of the calorific value and the weight of air required for combustion being calculated. The *temperature of spontaneous ignition* of a fuel (this J., 1917, 109–112) determines the limits of compression pressure which may be employed. When these limits are exceeded, pre-ignition takes place, and knocking and inefficient running result. The most careful consideration is necessary in preparing mixed fuels to ensure that the mixtures will stand normal compression pressures without pre-igniting. *Vapour pressure* determinations give direct information as to the probable behaviour of fuels as regards easy starting and “flexibility.” Benzol and good quality petrols have high vapour pressures, and the former also possesses the property of raising the ignition point of mixtures. It is therefore advantageous to mix benzol with heavy petroleum fuels (which, consisting mainly of aliphatic hydrocarbons, have low ignition points), and to employ light petrol for “livening” heavy fuels of coal-tar origin, such as solvent naphtha, which consists mainly of aromatic hydrocarbons (largely xylol) and has a comparatively high ignition point. The *explosive range* must be considered in adjusting carburettor devices, so that the throttle scale may fall within the range, and the lower limit requires consideration in connection with vapour pressure for fuels, such as alcohol, which are only explosive in mixtures containing comparatively high percentages. *Velocity of flame propagation* is not easy to determine; it affects the correct angle of spark advance at any given speed. Fuels which possess slow speeds of flame propagation, and, therefore, require greater angles of spark advance, can withstand slightly higher compression pressures than fuels of equal ignition point but possessing higher velocities of flame propagation. *Calorific values* (per weight) of motor spirits of any particular class do not vary greatly.—T. St.

*Paraffin wax; Heat of fusion of* —. G. von Kozlecki and S. von Pilat. Chem. Umschau, 1917, 71. Chem.-Zeit., 1918, 42, Rep., 147.

The heat of fusion of paraffin wax was determined by the lowering of the solidifying point on adding a compound (naphthalene, dimethylaniline, phenanthraquinone) of known molecular weight. It was found that the latent heat of fusion ranges from 38.9 to 43.9, increasing with rise of specific gravity, solidifying point, and molecular weight.

*Hydraulic separation.* Draper. See I.

*Gas purification masses.* Müller. See VII.



## PATENTS.

*Liquid fuel burners.* Burdons, Ltd., W. M. and M. M. Burdon, Bellshill, Lanark. Eng. Pat. 119,376, 18.1.18. (Appl. 1042/18.)

THE air supply for the burner passes through a pipe in the discharge flue of the furnace to preheat it to a temperature above the flash point of the oil, and thence to the rear of the burner nozzle where it is rotated and agitated by a helix and a small rotary fan. The front of the nozzle contains a short concentric inner tube into which a supply of heavy pitch or tar oil, or tar, is drawn by the air suction. The oil is vaporised and mixed with air in the central tube, and air passes also through the surrounding annular space. The nozzle is slightly smaller at the outlet, so that the inner conical jet of combustible mixture, and the outer hollow conical jet of air impinge at a point in front of the nozzle. The air supply pipe may be provided with an expansion joint, and may be replaced by two pipes to provide a greater heating surface. Modified forms of burner nozzles are described and also the application of the apparatus for heating a gas producer.—W. F. F.

*Alcohol; Solidified* —. A. Strobl, New York. U.S. Pat. 1,277,149, 27.8.18. Appl., 20.7.15.

SOLIDIFIED alcohol burning without soot is composed of ethyl alcohol 60 parts, methyl alcohol 40 parts, and sodium stearate 2–3 parts.—W. F. F.

*Chamber ovens or retorts for producing gas and coke or fuel; Heating flues of* —. C. W. Simpson, Leeds. Eng. Pat. 119,413, 12.4.18. (Appl. 6230/18.)

INCLINED gas retorts are arranged parallel to one another in a setting, and are heated by inverted U-shaped flues arranged between each pair of retorts. One limb of each flue is provided with a gas burner at the bottom, and all the burners between one pair of retorts are fed by an inclined gas conduit parallel to the retorts below the flues, so that the gas is preheated. All such gas conduits are supplied by a transverse flue in front of the setting. Preheated air is supplied to each burner from an inclined air flue parallel to the inclined gas conduit, and the combustion products pass in reverse direction through return flues parallel and adjacent to the gas conduit and air flue, and thence to a regenerator. An inspection opening is provided at the top of each of the inverted U-shaped flues, through which the burners, which are fitted with renewable and interchangeable nozzles, are accessible.—W. F. F.

*Coal; Calcining* — at high temperatures [in an electric furnace]. W. Dyrssen, New York. U.S. Pat. 1,277,707, 3.9.18. Appl., 27.5.18.

COAL is fed continuously downwards through an electric furnace from which air is excluded, the electrodes being arranged at an angle with one another converging towards the centre. The electrodes are adjusted longitudinally to vary the length of the arc, so that the voltage required for varying conditions of the charge is approximately constant.—W. F. F.

*Illuminating gas; Furnace for the production of* — in vertical retorts or chambers. Chamottefabrik Thonberg A.-G., Thonberg. Ger. Pat. 307,267, 19.2.16.

THE charge of a vertical retort for the production of illuminating gas is dropped, when evolution of gas is complete, into an extension of the retort built in the furnace walls and of sufficient size to

receive the whole of the charge. The hot coke heats up the surrounding brick work in which are passages for the air used in the furnace, and is then drawn off into a receiver through a gas-tight device consisting of a rotating drum with one or more openings.—L. A. C.

*Ammonia recovery producer gas process and apparatus therefor.* A. H. Lynn and N. E. Rambush. London. Eng. Pat. 119,049, 13.6.17. (Appl. 8461/17.)

HOT producer gas is passed through a washer to remove dust and cool it to a temperature not materially below 80° C., and is then passed upwards through a tubular reflux condenser. The admission zone of the condenser is kept at about 80° C., if necessary by external means, and the discharge zone of the condenser is kept at about 40° C. so that steam and tar are condensed and the ammonia passes out with the gases. The ammonia may then be absorbed by acid at normal temperature. In a modification a number of superposed tubular condensers may be used, having a free space for mixing the gases, between the tube-plates of each pair. The gas passes through the tubes, which are surrounded by films of cooling water. The heat transferred to the cooling water may be recovered for preheating the air blast for the producer, by passing the air around the tubes. Any ammonia contained in the condensed water and tar may be recovered by heating it with or without lime or other alkali.—W. F. F.

*Gas producers.* A. H. Lynn and N. E. Rambush. London. Eng. Pat. 119,207, 17.5.18. (Appl. 8258/18.)

IN a gas producer having a revolving grate, in which the portion on which the charge rests is provided with inclined helical lifting surfaces alternating with abrupt shoulders of considerable depth (see Eng. Pat. 15,467 of 1915; this J., 1917, 379), the shoulders are made sloping in a series of steps so as to make the descent of the charge less abrupt. The air blast is delivered through the spaces between the steps, which may be formed of a series of interlocked bars.—W. F. F.

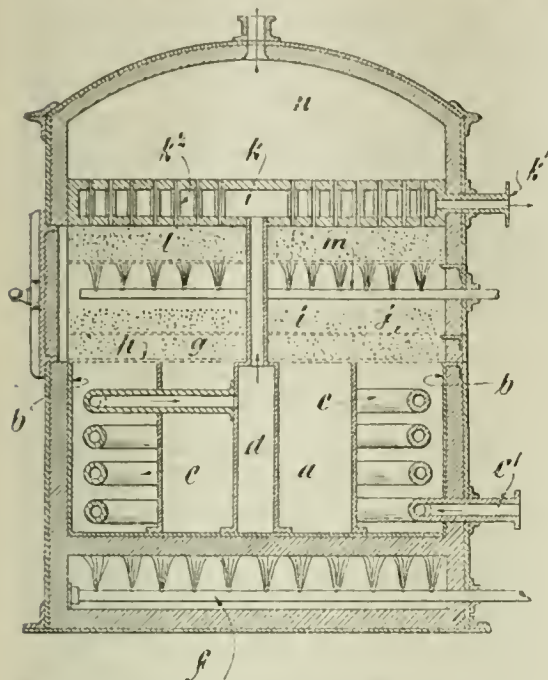
*Hydrocarbon oils; Process of treating* — and the product. T. Payne, Detroit, Mich., U.S.A. Eng. Pat. 119,066, 6.9.17. (Appl. 12,802/17.)

COMMERCIAL grades of kerosene, coal oil, etc., are cooled under increasing pressure, and at successive stages saturated with different combustible gases at the same pressure and temperature as the oil. The temperature and pressure are then restored to about atmospheric. The product can be utilised in place of gasoline in internal combustion engines. For example, kerosene may be caused to absorb, in this manner, in successive stages (1) about 5 vols. of coal gas at 0° C. and 6 atmospheres pressure, (2) about 8 vols. of Pintsch gas at -18° C. and 8 atmospheres, or about 3 vols. of acetylene at -26° C. and 12 atmospheres, and (3) 2 vols. of hydrogen at -35° C. and 15 atmospheres.—T. St.

*Oil gas; Apparatus for producing* —. W. Pickard, Rochdale, and D. R. Dobson, Bolton, Lancs. Eng. Pat. 119,399, 15.3.18. (Appl. 4588/18.)

AN inner chamber, *a*, containing crude liquid hydrocarbon is surrounded by a chamber, *b*, containing water acidulated with sulphuric acid. A heating coil, *c*, is fed by the pipe, *c*<sup>1</sup>, and exhausts into the central pipe, *d*, chamber, *k*, and discharge pipe, *k*<sup>1</sup>. Hydrocarbon vapour and steam generated by means of the burner, *f*, pass upwards through two layers of iron filings, *g*, *i*, supported on per-

forated trays, *h*, *j*, and then through a layer, *l*, of finely powdered charcoal and iron filings maintained at a red heat by a burner, *m*. The resulting



permanent gas of low calorific value passes through porcelain tubes, *k*<sup>2</sup>, to the collecting chamber, *n*, and thence for use as required.—W. F. F.

*Hydrocarbons; Process of producing* — H. H. Mandle, New York. Eng. Pat. 119,284, 27.9.17. (Appl. 13,955/17.)

HEAVY hydrocarbon oil such as crude petroleum, distillate, or residue, is mixed with water in any desired proportion by means of pumps drawing from separate tanks, the stroke of one of the pumps being variable. The mixture is sprayed into the upper end of a tortuous cracking tube in a heated chamber, and passes over a catalyst consisting of a packing of woven nickel-chromium-steel wire at the lower end of the tube. The cracked vapour is then passed through condenser pipes in the oil supply tank, whereby the oil is preheated and part of the vapour is condensed. The condensed liquid is collected in another tank and returned to the main tank for further treatment. The uncondensed vapour is then passed through a similar condenser in the water supply tank, whereby the water is preheated and a further portion of the vapour condensed. The uncondensed gas passes to a storage reservoir. The products include gasoline, benzene, toluene, and other light aromatic hydrocarbons.—W. F. F.

*Hydrocarbon oils; Method of decomposing* — A. E. Alexander, London. From A. B. Adams, New York. Eng. Pat. 119,485, 5.7.17. (Appl. 9713/17.)

WITHIN a furnace are three superimposed coils of piping. Water is forced through the top coil, and the steam generated is passed into the bottom of a closed tank containing hydrocarbon oil, thereby forming an emulsion of oil and steam which is forced by air pressure upwards through the middle or "subgenerator" coil, on leaving which it passes down a pipe outside the furnace, through an

atomiser, and into the upper end of the lowest or "generator" coil. At the lower, outlet end of this coil there is a metallic catalyst (preferably platinum, palladium, or nickel) in the form of a spirally wound coil of wire gradually decreasing in diameter until it merges into a point directed reversely to the flow of the gases. By the action of the catalyst the water vapour is decomposed and the hydrogen formed, together with free hydrogen produced by decomposition of the oil, is caused to combine with hydrocarbons, forming compounds of low boiling point. The apparatus may also include a condenser, a separator, a scrubber, and suitable means for storing the liquid and gaseous products. When it is desired to produce a maximum of fixed gas, the generator is heated to about 1600° F. (about 875° C.) and the pressure is reduced to a minimum; when a maximum of hydrocarbon compounds of low boiling point is required, the pressure is increased, preferably to about 250 lb. per sq. in., and the temperature reduced to about 1100° F. (about 595° C.). For heating the furnace, a portion of the emulsion of oil and steam is forced out of the storage tank into a burner of special construction. The mixture enters the burner through a circular coil of pipe heated by the flames of the burner. The vapours generated are led into an expansion box and then to the burner tip. Air for combustion is also admitted to the burner tip. At the outlet end of the circular coil of pipe and within the expansion chamber are coils of catalytic metal similar in construction to and for the same purpose as those at the outlet end of the generator coil.—L. A. C.

*Oil-distilling apparatus.* J. B. Edwards, Jersey City, Assignor to Tide Water Oil Co., Bayonne, N.J. U.S. Pat. 1,277,884, 3.9.18. Appl., 19.9.16.

IN the upper part of a pressure still for the pressure cracking of petroleum or its higher boiling fractions is a disc valve mounted in a vertical guide. The valve is normally held open by suitable means, which can be released by a device operable at a distance from the system, the pressure within the still then forcing the valve against the outlet.

—J. A. C.

*Paraffin; Extracting — from oils.* J. S. Hussey, Martinez, Cal. U.S. Pat. 1,277,935, 3.9.18. Appl., 22.1.17.

MINERAL oil is introduced into a vertical cylinder in which there is a hollow rotating shaft fitted with a hollow arm provided with a number of openings near the bottom of the vessel, and a "gathering or skimming" arm at the surface of the oil. Air is forced through the shaft into the hollow arm and through the openings into the oil in the form of small bubbles, around which the paraffin collects and is carried to the surface, whence it is removed by the upper arm.—L. A. C.

*Hydrocarbons [paraffin]; Process for separating — [from still residues].* R. R. Rosenbaum, Chicago, Ill. U.S. Pat. 1,278,923, 3.9.18. Appl., 13.12.17.

To separate paraffin from the residue of the distillation of crude oil, the residue is heated and intimately mixed with finely divided absorbent material. The mixture is then cooled by agitation in contact with cooling surfaces and treated with a solvent which does not dissolve the paraffin but dissolves other hydrocarbons present. After removing the solution from the solid residue, the paraffin is separated from the porous absorbent material.—L. A. C.



*Petroleum and other hydrocarbon oils* [; Refining —]. H. W. Knottenbelt, Brussels. U.S. Pat. 1,277,605, 3.9.18. Appl., 3.7.16.

See Fr. Pat. 411,780 of 1910; this J., 1910, 933.

*Gas furnaces.* Eng. Pat. 119,553. See I.

*Hydrogen manufacture.* Eng. Pat. 119,591. See VII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Efficiency of different grades of gas.* See IIa.

*Explosibility of wood charcoal.* Wöhler. See VII.

*Distillation of automobile tyres.* Dubosc. See XIV.

### PATENTS.

*Hydrocarbon materials; Apparatus for the extraction of — [from shale].* A. M. Boyle, Assignor to Crane Shale Oil Corporation, Reno, Nev. U.S. Pat. 1,276,866, 27.8.18. Appl., 29.12.17.

A VERTICAL retort for treating shale is heated by means of a steam jacket and is fitted with a vertical rotating screw agitator which raises the shale in the centre of the retort and allows it to fall down the space between the agitator and the walls. Means are provided for releasing steam confined within the peripheral sweep of the agitator, and for drawing off the gases and vapours from the top of the retort. Shale is fed into a supply container from the bottom of which it is conveyed into the retort along a tubular screw conveyor inclined upwards, the upper end of the conveyor leading into a vertical shoot down which the shale passes into the retort.—L. A. C.

*Hydrocarbon materials; Process of extracting — from shale and similar earthy material.* A. G. Crane, Assignor to Crane Shale Oil Corporation, Reno, Nev. U.S. Pat. 1,276,879, 27.8.18. Appl., 4.10.17.

FINELY-DIVIDED shale or other earthy material is mixed with a neutral, granular, non-absorbent material (e.g., sand) and while thoroughly agitated, treated with dry steam in a suitable container. The mixture is then conveyed to another container in which it is heated to a higher temperature and under a pressure greater than that of the atmosphere. The vapours generated at each stage are withdrawn and condensed.—L. A. C.

*Lignite, peat, wood, etc.; Distillation of —.* Bertzit Ges.m.b.H., Munich. Ger. Pat. 306,956, 21.3.16.

THE material is partially dried (to a moisture content of 10 to 20%) by a counter current of moderately moist air prepared by mixing the waste heating gases arising from the furnace with cold air. Peat and other very moist fuels are first air dried to a moisture content of about 50%.—L. A. C.

## III.—TAR AND TAR PRODUCTS.

*Phenol and the cresols; Compounds of — with pyridine.* H. F. W. Skirrow and T. V. Binmore. J. Amer. Chem. Soc., 1918, 40, 1431–1442.

THE effect of excess of phenol and excess of pyridine on the dissociation of pyridine phenoxide, postulated as  $C_6H_5OH.C_5H_5N \rightleftharpoons C_6H_5OH + C_5H_5N$ , has been studied by the cryoscopic method and it

has been found that phenol depresses the dissociation much more strongly than does pyridine. The dominant equilibrium might therefore be  $(C_6H_5OH)_2.C_5H_5N \rightleftharpoons 2C_6H_5OH + C_5H_5N$ , but a study of this dissociation on similar lines showed that the effect of excess of phenol on the apparent dissociation was only greater than that of excess of pyridine with comparatively high additions of these. An explanation of these results and of the extraction results obtained previously (Hatcher and Skirrow; this J., 1918, 51 A) is based on the following equilibria:  $C_6H_5OH.C_5H_5N \rightleftharpoons C_6H_5OH + C_5H_5N$  and  $C_6H_5OH.C_5H_5N + C_6H_5OH \rightleftharpoons (C_6H_5OH)_2.C_5H_5N$ . It is shown that the difference between the effect of addition of excess of phenol and of excess of pyridine on the extraction experiments tends to disappear as the dilution of the benzene solution is increased.—H. W.

*Chlorinated toluene; Rapid analysis of mixtures of —.* H. A. Lubs and A. B. Clark. J. Amer. Chem. Soc., 1918, 40, 1449–1453.

THE sample (about 0.5 gm.) is placed in a sealed tube with water (10 c.c.) and briskly shaken in a bath of boiling water for two hours; the contents of the tube are washed into a small assay flask, and the tube is rinsed with water (free from carbon dioxide) and then with alcohol if any benzoic acid remains. The solution is then titrated with alkali in the presence of thymolsulphophthalein according to the authors' method (see page 718 A). The percentage of benzotrichloride ( $x$ ) is calculated from the benzoic acid formed; the percentages of benzal chloride ( $y$ ) and benzyl chloride ( $z$ ) are derived from the following relations:  $x + y + z = 100$ ;  $0.545x + 0.434y + 0.281z = \% \text{ side chain chlorine}$ . The method is very suitable for plant control work and, if certain precautions are taken, will give a high degree of accuracy.—H. W.

*Toluene-o- and -p-sulphonamides; Freezing point curve of mixtures of —. Composition of mixtures of toluene-o- and -p-sulphonic acids.* P. V. McKie. Chem. Soc. Trans., 1918, 113, 799–803.

THE estimation of the relative amounts of toluene-o- and -p-sulphonic acids in a mixture of the two acids cannot be satisfactorily performed by means of the freezing point curve of the mixed sulphonyl chlorides (Holleman and Caland, this J., 1911, 1202) for several reasons, particularly on account of the flatness of the curve in the eutectic region. The fusion curve of the sulphonamides, however, is satisfactory. It lies within a convenient range of temperature, namely  $110^\circ$ – $156^\circ$  C., and the eutectic point ( $110.25^\circ$  C.; 61.25% of the *p*-sulphonamide) is extremely sharply defined. The melting points, not the freezing points, of the mixtures were determined.—C. S.

*Carbonisation of coal.* Savage. See IIa.

*Naphthalene in gas.* Colman. See IIa.

### PATENTS.

*Aromatic hydrocarbons; Process of oxidising — and the lower oxidation products thereof. Process of oxidising aromatic hydrocarbons having a plurality of side chains, or consisting of a plurality of rings and the lower oxidation products of such hydrocarbons.* The Seldon Co., Pittsburgh, Pa., and H. D. Gibbs, Cherrydale, Va., U.S.A. Eng. Pats. (A) 119,517, and (B) 119,518, 1.10.17. (Appls. 14,150 and 14,151/17.)

AROMATIC hydrocarbons containing one or more side chains or consisting of a number of rings are oxidised by treatment in the form of vapour at a suitable temperature with an oxygen-containing

gas (e.g. air) in the presence of a catalyst comprising an oxide or mixture of oxides of metals of the fifth or sixth group of the periodic classification, i.e. antimony, bismuth, chromium, tungsten, uranium, or, preferably, molybdenum, and vanadium. The oxides may be in a coarse or fine state of division, and the gas and vapour mixture may be passed over or through the catalyst. In the case of hydrocarbons containing one side chain, the temperature employed lies between 200° and 500° C. (preferably between 350° and 450° C.); in the case of hydrocarbons containing a number of side chains or consisting of a number of rings, the temperature varies between 250° and 600° C. The quantity of oxygen employed should be, preferably, twice that required by theory. Examples: (a) Toluene vapour mixed with air is passed over vanadium oxide ( $V_2O_5$ ) between 350° and 450° C., and the resulting mixture, consisting chiefly of benzaldehyde, together with some benzoic acid, unconverted toluene, and water, is separated by suitable means; (b) a mixture of naphthalene vapour and air is passed over vanadium oxide at 350°–550° C. (preferably about 450° C.), or over molybdenum oxide ( $MoO_3$ ) at about 500° C., resulting in the formation of phthalic anhydride, phthalic acid, 1,4-naphthoquinone, benzoic acid, and water; oxidation of xylene vapour in a similar manner results in the formation of methylbenzaldehydes, phthalaldehydes, benzenedicarboxylic acids, toluic acids, benzoic acid, and benzaldehyde; oxidation of anthracene vapour (preferably at about 500° C.) results mainly in the formation of anthraquinone. —L. A. C.

Hydrocarbons. Eng. Pat. 119,284. See IIa.

#### IV.—COLOURING MATTERS AND DYES.

*Fuchsin synthesis; Explanation of Heumann's* —. H. E. Fierz and H. Koechlin. *Helv. Chim. Acta*, 1918, 1, 218–226.

In the original Heumann reaction (Ger. Pats. 66,511 and 68,976), in which carbon tetrachloride, aluminium chloride, and a tertiary amine, for example dimethylaniline, react to produce Crystal Violet or an analogous product, the authors claim that the initial product of the reaction is the ketochloride,  $CCl_2[C_6H_4.N(CH_3)_2]$ , because a small quantity of Michler's ketone is obtained by adding the reaction product to ice-water. When acetanilide is used in place of dimethylaniline, a 60% yield of 4,4'-diaminobenzophenone is obtained after hydrolysing the product formed by the addition of ice-water. In a similar way *o*-chloroacetanilide yields 3,3'-dichloro-4,4'-diaminobenzophenone and dimethyl-*m*-toluidine yields 2,2'-dimethyl-4,4'-tetramethyldiaminobenzophenone. (See also *J. Chem. Soc.*, Dec., 1918.)—C. S.

*Quinonoid dyes; Formulation of so-called* —. H. E. Fierz and H. Koechlin. *Helv. Chim. Acta*, 1918, 1, 210–218.

The authors propose to apply, with certain modifications, Werner's views of the structure of inorganic substances to quinonoid dyes and to formulate these as complex compounds. Thus fuchsin chloride is represented by the formula,  $[(NH_2.C_6H_4)_3.C]Cl$ , and similar formulae are proposed for dyes of the diphenylmethane, azine, thiazine, oxazine, and xanthene classes. (See also *J. Chem. Soc.*, Dec., 1918.)—C. S.

*Sulphur dyestuffs; Determination of sulphur that produces sulphuric acid in* —. W. Zänker and K. Schnabel. *Färber-Zeit.*, 1918, 29, 26–29.

SULPHUR dyestuffs, when exposed to the air in a finely divided, moist condition, become acid with sulphuric acid. The rate of formation of acid is

increased by rise of temperature. In presence of alkali the formation of acid is favoured and reaction accelerated. Various dyestuffs were tested both in the commercial form and after purification by dissolving 1 grm. of dyestuff and 2 grms. of pure sodium sulphide in 250 c.c. of water, boiling for a short period, pouring the solution into 3 litres of water acidified with acetic acid, allowing to settle, washing the residue six times by decantation with water acidified with acetic acid, collecting the dyestuff on a hardened filter, and drying at 100° C. For the estimation of the acid the dyestuff was mixed with 50 c.c. of N/10 caustic soda, evaporated to dryness, and heated for one hour at 110° C. to 120° C. The labile sulphur was thus converted into sulphuric acid, the amount of which was determined by back titration, using as indicator phenolphthalein, or litmus upon writing paper. The following table shows the results obtained:—

Dyestuff	Percentage of pure dyestuff in commercial article	Percentage of sulphur giving sulphuric acid		Percentage of sulphuric acid formed	
		Pure dye	Commercial dye	Pure dye	Commercial dye
Thioxin Black G O.O.O.	69.62	9.43	6.56	28.93	20.14
Sulphuroil Black ...	46.2	13.09	6.05	40.14	18.55
Katigene Deep Black.					
Immedial Brilliant	56.98	11.07	6.31	33.96	19.35
Carbon F.G. ...	82.52	8.86	7.32	27.19	22.44
Immedial Black N.N.G.	52.72	12.81	6.75	39.29	20.72
Katigene Black F.G. ...	39.32	17.91	7.03	54.95	21.56
Katigene Brilliant					
Black F.G. ...	70.66	9.60	6.78	29.46	20.82

These figures do not show the sulphuric acid produced on dyed materials, but simply the acid produced under the above conditions.—L. L. L.

*Morindone.* J. L. Simonsen. *Chem. Soc. Trans.*, 1918, 113, 766–774.

THE glucoside, morindin, has been extracted from the root bark of *Morinda citrifolia* and very carefully purified. It has the composition,  $C_{26}H_{34}O_{14}$ , and from its properties and those of its acetyl derivative there would appear to be no difference between morindin obtained from *M. citrifolia* and that from *M. umbellata* (compare Perkin and Hummel, this J., 1894, 940, 1054; Oesterle and Tisza, 1908, 441; Perkin, 1908, 766). Morindone, obtained by hydrolysis of the glucoside, appears to be either a hydroxymethylanthranidin or a hydroxymethylchrysazin.—C. S.

*Pyrrole Blacks.* A. Angeli. Atti R. Accad. Lincei, 1918, 27, I., 209–212.

LIKE hydrogen peroxide in acetic acid (see this J., 1917, 210), most oxidising agents convert pyrrole into highly coloured products, mostly black or brown, some of these being readily soluble in alkali and some insoluble. Thus, the action of potassium bichromate on an acetic acid solution of pyrrole produces a black precipitate, which may be produced on a cotton fabric and is very fast to soap and light. This colouring matter is apparently a chromium lake.—T. H. P.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Vegetable fibrous materials; Scheme for the analytical investigation of* — and the cellulose prepared from them. C. G. Schwalbe. Verein deut. Chem., Sep., 1918. Z. angew. Chem., 1918, 31, 193–194.

THE time is ripe for a coördinated re-investigation on a uniform basis of all the standard raw



materials of the textile and cellulose industries, in order that the proximate composition of these may be definitely fixed, the variations due to environment, etc. defined, and the results utilised for the classification and evaluation of new or little known materials. The author discusses the question of the selection of the analytical "constants" of most general significance and of the standardisation of methods and apparatus. These questions should be settled by a committee of recognised standing, and data should be accumulated on the agreed basis. The scheme of analysis proposed by Cross and Bevan and utilised by other investigators is regarded as not fully adapted to the light of modern knowledge: it is considered to be unnecessarily long and to contain factors of undetermined significance. The abbreviated scheme recommended by the author comprises determinations of ash, moisture, fat, wax and resin, cellulose, furfural, methylfurfural, and methoxyl. The pentosans are satisfactorily represented by the furfural value, but certain standardisation of methods and apparatus is required to ensure concordant results. The lignin may be taken to be represented by the methoxyl value, but it is possible that this requires correction by the results found for methylfurfural which is supposed to represent methylpentosans.

—J. F. B.

*Celluloid; Analysis of* —. A. Hervé. Caontchoue et Gutta Percha, 1918, 15, 9601—9604.

The rasped celluloid may be extracted in a Soxhlet apparatus, preferably with chloroform, and the camphor determined by difference. The nitrocellulose residue, however, obstinately retains some of the solvent, even after drying to constant weight, and a correction should be made for the retained chloroform by saponification of the residue by alcoholic potassium hydroxide and determination of the chloride produced. Alternatively the camphor may be determined by the cryoscopic method in benzene solution. For the extraction of the camphor, 2—5 grms. of the celluloid, in small pieces about 1 mm. thick, is placed in a distillation flask with 200 c.c. of water and 2—3.5 grms. of potassium hydroxide. The camphor is distilled over and solidifies in the condenser; the operation is generally complete when 150 c.c. of distillate has passed over. The water is exhausted by shaking twice with 5 c.c. of pure benzene. This benzene is used for washing down the condenser, and is collected together with five other washings in a flask of about 100 c.c. The benzene solution of camphor is dried by anhydrous copper sulphate and filtered into a 50 c.c. flask. The filter is washed with dry benzene and the solution made up to the mark. The cryoscopic apparatus used is a modification of that of Raoult, with a Beckmann thermometer graduated in 0.01° C. Cooling is effected by the evaporation of carbon bisulphide in a current of air, and the tube containing the camphor solution is placed in an intermediate jacket containing alcohol. The camphor solution is stirred during the operation and the thermometer is read with a lens. The solution is slowly supercooled, a crystal of pure benzene is dropped in, and the point to which the thermometer rises and then remains constant for one minute during solidification is taken as the freezing point. The result is calculated from a table prepared from observations with solutions containing known quantities of pure camphor. The accuracy of the method has been verified, and with the lens the thermometer can be read to 0.001° C. Methylacetanilide (mannol) in admixture with acetanilide may be estimated cryoscopically in the same way, using pure aniline as the solvent.—J. F. B.

*Celluloid; Stabilisation of [nitrocellulose for manufacture of]* —. A. Colassi. Caoutchouc et Gutta Percha, 1918, 15, 9604—9605.

NITROCELLULOSE intended for the manufacture of celluloid should undergo a process of stabilisation with hot water if subsequent troubles are to be avoided, since even the most thorough washing with cold water is not sufficient to eliminate the unstable forms of nitrocellulose of a low degree of nitration. The material on coming from the hydroextractors or nitrating pots is ground in a hollander, and is afterwards repeatedly washed with successive changes of hot water until it is sufficiently purified to pass the heat test for stability. Two grms. of the nitrocellulose is placed in a test-tube and heated in a paraffin bath at 130° C.; a satisfactory material should not decompose with formation of brown fumes in less than an hour at this temperature. The occurrence of white points in celluloid is sometimes due to the presence of particles of nitrocellulose of a high degree of nitration; these white points are insoluble in alcohol and camphor, but may be recognised by their gelatinisation in acetone.

—J. F. B.

*Cellulose and nitrocellulose.* Hervé. See XXII.

#### PATENTS.

*Woven fabric; Material for — and process of manufacturing it [from palm leaves].* K. Miyake, Tokyo, Japan. Eng. Pat. 119,428, 7.6.18. (Appl. 9422/18.)

The fresh leaves of the coconut palm are boiled in water for a short time, then torn apart into upper and lower halves. The halves are then torn into straps or bands of suitable width, which are boiled for 1—2 hours in a 5—8% solution of sodium carbonate. The material is washed and steeped in a bleaching solution containing 1—3 lb. of sodium peroxide, 1—2 lb. of potassium oxalate, and 50—100 grms. of sulphuric acid to 100 lb. of water. The treated strips roll up on drying, and form smooth, semi-transparent threads suitable for weaving into various articles, e.g. hats, mats, bags, etc.

—J. F. B.

*Textile product [; Water-repellent —] and process of making same.* W. L. Franck, Philadelphia, Pa. U.S. Pat. 1,277,057, 27.8.18. Appl. 9.3.17.

A SOFT, pliable, water-repellent material is produced by treating textile fibre with a mixture of chromium sesquioxide and glucose. The bath is prepared by dissolving 4 parts of sodium bichromate in water and adding 8 parts of commercial hydrochloric acid, 2 of glucose, 3 of "sal soda" (sodium carbonate), 2 of sodium thiosulphate, and 2 of sodium sulphite.

—J. F. B.

*Yucca and the like; Process of preparing and extracting vegetable fibre from —.* A. G. Brown, Assignor to Yusi Fibre Co., Los Angeles, Cal. U.S. Pat. 1,277,185, 27.8.18. Appl. 24.12.17.

GREEN yucca leaves are passed between crushing rollers, and the crushed material is boiled, passed between corrugated rollers, then between differential smooth rollers, and the soft wet material treated in a scutching machine.—J. F. B.

*Fabric; Soil-proof — and process of making the same.* L. J. Cavanaugh, Swampscott, Mass. U.S. Pat. 1,277,695, 3.9.18. Appl. 20.7.17.

TEXTILE fabric is first saturated with a fixing agent, e.g. a solution of a metallic salt, then dried, impreg-

nated with a highly volatile non-solvent of dissolved cellulose, e.g. petroleum spirit mixed with a small proportion of oil, and finally coated with a preparation of dissolved cellulose.—J. F. B.

*Wool-scouring liquors; Recovery of potassium and sodium compounds, fats, and other valuable ingredients from —.* F. E. Underwood and B. F. Ilston, Jamestown, N.Y. U.S. Pat. 1,277,784, 3.9.18. Appl., 1.6.17.

WOOL-SCOURING liquor is treated with an alkaline-earth salt, such as magnesium sulphate, and heated at about 350° under a pressure of about 125 lb. per sq. in., in order to separate all the fats in the form of insoluble soaps or in conjunction with such soaps. The liquid is separated and evaporated for the recovery of potassium and sodium salts, and the insoluble soap is treated with an acid to liberate the fat and regenerate the magnesium sulphate for further use.—J. F. B.

*Celluloid; Manufacture of unflammable —.* II. Dreyfus, Basle, Switzerland. Eng. Pat. 114,304, 25.2.18. (Appl. 3336/18.) Int. Conv., 22.3.17.

IN the manufacture of celluloid or films having a basis of cellulose acetate, the proportion of softening agents incorporated with the cellulose acetate is varied according to the relative viscosity of the latter. For example, with a cellulose acetate having a relative viscosity of 8–12, triacetin equivalent to 10–15% of the weight of the cellulose acetate may be incorporated, whereas with an acetate having a viscosity of 35–45 on the same scale, the proportion of triacetin may be increased to 40–50%. —J. F. B.

*Cellulosic material; Method of treating threads of —.* C. E. Vanderkleed, Collingswood, N.J., and J. E. Brewer, Norristown, Pa., Assignors to W. M. Field, Haverford, Pa., H. J. Verner, Bryn Mawr, Pa., and F. Heidlberg, Jersey City, N.J. U.S. Pat. 1,269,340, 11.6.18. Appl., 5.8.16.

A LAYER of cellulose is deposited upon threads of cellulosic material, or upon fabrics made of such threads, by passing them through a cuprammonium solution of cellulose and then subjecting them to the action of acid. Copper and ammonium salts are removed by washing.—F. C. T.

*Paper; Etchable coated — for the production of backings or surfaces for printing illustrations.* J. Miln, Toronto, Canada. Eng. Pat. 119,368, 4.1.18. (Appl. 250/18.)

PAPER is coated with a composition prepared with 200 lb. of "blanc fixe," 580 lb. of china clay, and 65 galls. of water mixed with a solution containing 125 lb. of gelatin, 2 lb. of glycerin, and 12 oz. of aluminium acetate in 25 galls. of water. The coated paper is printed with a medium capable of resisting the etching liquid, and then treated for a short time with a dilute solution of bleaching powder. The acidity of the aluminium acetate causes the formation of hypochlorous acid which destroys the binding qualities of the gelatin in the exposed portions of the coating, so that the materials of the coating can be washed away, leaving the printed device in relief.—J. F. B.

*Paper making machines [; Suction-boxes of —].* W. A. Aitken, Gravesend. Eng. Pat. 119,594, 14.1.18. (Appl. 832/18.)

IN a Fourdrinier paper machine the suction-boxes are mounted on a carrier-frame, pivoted at the centre on one side of the machine and oscillated in a horizontal plane over a limited path on the

other side of the machine. The oscillating motion is imparted by a slow-speed reciprocating gear operating on the carrier-plate remote from the pivoted plate. The movement of this plate is regulated by a roller-pin situated in a curved slot having a radius centred at the pivot. The swinging oscillation of all the suction-boxes simultaneously enables the wire to guide freely and keep to its running centre.—J. F. B.

*Paper; Method and means for finishing — [to produce a cloth finish].* H. J. Guild, Assignor to Eastern Manufacturing Co., Bangor, Me. U.S. Pat. 1,277,714, 3.9.18. Appl., 9.12.16.

BELTS of suitable textile material are fed between two rollers, the paper under treatment lying between the belts. One of the rollers is of a hard material, and the other is of a material sufficiently yielding to bring about a slight relative motion between the paper and the belts while under the pressure of the rollers, but at the same time has sufficient compressive rigidity to ensure that each side of the paper receives an impression of the textile belt with which it is in contact.—L. A. C.

*Paper; Process and means for producing all-wood —.* A. H. Lefebvre, Watertown, N.Y. U.S. Pat. 1,277,737, 3.9.18. Appl., 16.2.17.

WOOD is ground to produce a mixture of fine fibrous material and partially reduced material, the pulp from the grinder is passed through a scrubbing tank and then through a hydrostatic gravity separator, in which the foreign matters are separated; the cleaned material is then screened and the portions which are rejected by the screens are treated in a refiner to produce a relatively coarse fibrous pulp which is mixed with the pulp coming from the original grinder, so that it again passes through the grading system.—J. F. B.

*Paper pulp; Method and apparatus for bleaching —.* J. E. Heiskanen, Canton, N.C. U.S. Pat. 1,277,926, 3.9.18. Appl., 7.2.18.

THE pulp is treated in a series of circulating units, each consisting of a pair of vertical tanks, the bottoms of which taper to a connecting passage in which a propeller is situated. Adjustable means are provided whereby a portion of the pulp circulating in each unit is allowed continuously to flow over into the next unit of the series, whilst the remainder is returned to the tank from which it was propelled, the amount of pulp continuously passing over from each unit to the next being equal to the amount continuously fed into the apparatus. —J. F. B.

*Paper-pulp; Beating or grinding of —.* E. Partington, Westwood Park. U.S. Pat. 1,277,637, 3.9.18. Appl., 23.3.18.

SEE Eng. Pat. 110,482 of 1917; this J., 1917, 1232.

*Fireproofing composition.* U.S. Pat. 1,276,742. See I.

*Artificial leather.* U.S. Pat. 1,269,339, 1,270,055, and 1,277,528. See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Silk weighting with zirconium sulphate.* E. Ristenpart. Färber-Zeit., 1918, 29, 26.

EXPERIMENTS show that the weighting of silk by zirconium sulphate cannot replace the weighting by tin salts.—L. L. L.



*Vat dyestuffs; Explanation of the printing of coloured reserves on some —.* R. Haller. *Färber-Zeit.*, 1918, 29, 1–3.

In the author's method of printing coloured reserves with vat dyestuffs (this J., 1918, 462 A), Pomeranz (*loc. cit.*) considers that the Indanthrene vat does not penetrate the printed vat colour, because the caustic soda necessary to fix the Indanthrene dyestuff from the vat is not available, having reacted with the iron and manganese reserve salts, with the formation of hydroxides of these metals. The author holds that the hydroxides of zinc and manganese are in a colloidal condition in the printing thickener, and form a membrane which prevents the penetration of the Indanthrene, which is in a colloidal form in the dye-vat. The caustic soda of the Indanthrene bath will, however, penetrate the colloidal matter of the printed colour, and, along with the reducing agent in the printed reserve, will be able to reduce and fix the printed colour on the cloth. The author also considers that the process used at the Felmayer works, in which reserve effects are produced by printing with manganese chloride, bichromate, gelatin, etc., for blue and red effects, also supports his view. Manganese dioxide produced on the fibre, in presence and in absence of glue, shows entirely different forms, especially when the fibre is treated with cuprammonium solution. In the first case there is a continuous membrane in which the manganese dioxide particles are perfectly uniform throughout the fibre. In the second case the fibre swells, passes into solution, and leaves the manganese dioxide in comparatively coarse form. The author considers that all varieties of thickening agents must act as a filter to prevent the colloidal vat dyestuff from passing through to the fibre.

—L. L. L.

*Vat dyestuffs; Printed reserves with —.* H. Pomeranz. *Färber-Zeit.*, 1918, 5, 51–54. (See also this J., 1918, 462 A.)

THE removal or stripping of Indigo from a fabric by reduction and solution in caustic alkali is not successful because of the re-oxidation of Indigo-white. This is prevented by converting the Indigo-white, with the aid of zinc oxide and a quaternary ammonium base, Leukotrop W (B.A.S.F.), into a compound stable in air, which is soluble in alkali and can thus be removed from the fibre. This process has made the reduction method of stripping a commercial success. It also renders possible the printing of bright Indanthrene effects upon an Indigo ground. Oxidation reserves either convert the Indigo-white into insoluble lakes, and therefore prevent its fixation on the fibre, or prevent the vatted dyestuff from fixation on fibre without the intermediate oxidation. Copper salts, both soluble and insoluble, have been found to be good oxidising reserves. The production of red reserves with Paranitriline Red requires absence of copper compounds, and such reserves have been obtained by the use of sparingly soluble salts of *m*-nitrobenzenesulphonic acid; these salts, however, cannot be used with alkaline prints on account of the formation of soluble alkali salts. For Indanthrene vats, aluminium, ammonium, and manganese chlorides are used along with many filling agents, such as clay, lead sulphate, etc. The remarkable observation that the addition of reducing agents to a vat dyestuff, along with the above-mentioned salts, gives a vat reserve, renders possible the production of vat grounds and vat coloured effects. The author discusses Haller's explanation of the production of coloured reserves by printed vat dyes (see preceding abstract), and points out the possibility of the unreduced easily vatted dyestuff acting as an oxidising agent on the more difficultly vatted dyestuff, and so itself acting as a reserve.—L. L. L.

*Waterproofing [fabrics].* E. Jentzsch. *Färber-Zeit.*, 1918, 29, 3.

For the waterproofing of lightly woven fabrics, so that water will not drop through, but may very slowly penetrate, the author finds that basic acetate or formate of aluminium is the most suitable, prior to wax-proofing.—L. L. L.

*Pyrrole Blacks.* Angeli. See IV.

#### PATENTS.

*Yarns or fabrics; Machines for boiling, bleaching, dyeing, sizing, or similarly treating —.* J. Bright and Bros., Ltd., and J. Evans, Rochdale. Eng. Pat. 119,173, 22.1.18. (Appl. 1225/18.)

In a continuous line of machines for treating yarns or fabrics in the rope state, comprising a series of boxes each with a pair of gripping or drawing-through rollers, guide rollers are provided immediately before and after the squeeze rollers, the guide rollers being mounted on yielding bearings controlled by springs, which take up any irregularities in the tensions of the yarns and prevent undue stretching and breakage.—J. F. B.

*Textile fabrics; Printing of —.* The Calleo Printers' Association, Ltd., and F. Ashton, Manchester. Eng. Pat. 119,298, 5.10.17. (Appl. 14,391/17.)

THE fabric is first treated with a chrome mordant, then padded with one of the customary "naphthol prepares," and dried. It is next printed with a suitable diazo compound, e.g. a diazo derivative of nitroaniline,  $\alpha$ -naphthylamine, etc., and a discharge mixture capable of discharging the chrome mordant is overprinted wholly or partially on the portions of the fabric which had previously received the azo colour. A suitable discharge may contain citric or tartaric acid or a salt of these acids. The fabric is subsequently dyed with any suitable mordant dyestuff.—J. F. B.

*Stains; Removal of — from cloth.* Th. Goldschmidt A.-G., Essen. Ger. Pat. 306,707, 19.4.17.

GLYCOL or a mixture of glycol and water is used to remove stains (coffee, chocolate, wine, etc.) from cloth, not removable by petroleum spirit.—L. A. C.

*Fabric pieces; Processes and plant for treatment [weighing] of —.* Wegmann & Co., Baden. Switzerland. Eng. Pat. 111,466, 3.10.17. (Appl. 14,290/17.) Int. Conv., 18.11.16.

SEE U.S. Pat. 1,259,526 of 1918; this J., 1918, 297 A.

*Dyeing machines.* H. M. Dudley, Philadelphia, U.S.A. Eng. Pat. 112,630, 11.1.18. (Appl. 681/18.) Int. Conv., 11.1.17.

SEE U.S. Pat. 1,258,545 of 1918; this J., 1918, 265 A.

*Laundry starch.* U.S. Pat. 1,276,722. See XVII.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Sulphuric acid; Concentration of — by the Krell-Strzoda tube system.* W. Strzoda. Z. angew. Chem., 1918, 31, 185–187.

THE preliminary concentration of chamber acid from 50° B. to 60° B. usually takes place in the Glover towers or in open lead pans. Closed vessels are

used for further concentration to avoid loss of acid in the distillate, which reaches a strength of 60° B. as the concentration approaches 97–98%. Few substances are sufficiently resistant to acid to be used for the construction of the necessary plant. At one time platinum and glass were used. The Kessler-Düron concentrator is constructed of vulcanite, and the concentration is effected by means of hot gases from coke or coal furnaces working on the counter current principle. In this apparatus it is not economical to concentrate the acid above 93–94%. Later iron boilers have been brought into use in the Hartmann-Benker film concentrator and other apparatus. In the Krell-Strzoda process of concentration (Eng. Pat. 26,732 of 1913; this J., 1914, 643), the concentration tubes and certain other parts are made of an acid-resisting iron enclosed in a shell of cast iron, the intermediate space being filled with an acid-proof cement, which seals any cracks which may appear in the brittle acid-resisting iron during use. The concentrating tubes are arranged parallel on the slightly inclined bed of a furnace. They are connected with each other and with the distillate receiver in such a manner that the acid passes from the first tube to the second, and so on through the series to the acid cooler and receiver. The distillate is taken from the top of a crescent-shaped bend in the connection between each two tubes to a cooler and receiver. Advantages claimed for this process are that the working conditions are easy to control, a comparatively short time is required for the construction of the plant, for subsequent repairs or renewals and cleaning, and that a colourless, clear, pure quality of acid is produced. In a small Krell-Strzoda plant of up to 6 tubes, the production is somewhat less than 0.9 ton of 97–98% acid per tube per 24 hours, and 0.9–1.0 ton with more than 6 tubes. The coal consumption is about 4 cwt. per ton of 97–98% acid. Instead of condensing the distillate from the Krell-Strzoda plant in water-cooled lead pipes, H. Petersen passes the hot vapour up a small tower, in which it is dissolved by a descending stream of 50% chamber acid, yielding acid of 85–90%, which is passed again through the concentrating apparatus. The process is now in operation in several works. It is essential that the tubes be kept clean and free from slime and incrustation, as otherwise they are burned and soon require renewal.—S. S. A.

*Potassium salts; Deposits of — at Dallol (Erythraea).* M. Glua. Atti R. Accad. Lincei, 1918, 27, I., 331–335.

The analytical results are given of a number of samples from the salt deposits of Dallol in S. Danania (Erythraea). At many points these deposits consist largely of potassium chloride (up to 98.6%), whilst at others sodium chloride predominates. In all cases the proportions of magnesium chloride are low, and only traces of bromides are present in a few instances. The potassium minerals occur at the surface, and are localised at certain points of the salt area. The thermal springs of Dallol have a temperature of about 80°–90° C., and consist of saturated solutions of magnesium chloride containing traces of sodium chloride and appreciable proportions of magnesium bromide.—T. H. P.

*Nitrites and nitrates in mixtures; Methods for the determination of —.* A. Oelsner. Z. angew. Chem., 1918, 31, 170–172, 178–179.

Various methods are discussed for the determination of nitrites and nitrates in presence of each other, for instance in culture media for nitrifying and denitrifying bacteria. The diphenylamine reaction may be employed for the estimation of nitrates colorimetrically or, at a certain dilution, for that of nitrites. The nitrate reaction is stated to be

more intense with diphenylbenzidine, and the nitrites may be separately determined by permanganate. Various gasometric methods have been based on the decomposition of the nitrite with formation of nitrogen or nitric oxide. Ferrous chloride in acetic acid solution decomposes the nitrite only; afterwards if hydrochloric acid be added the nitrate is also decomposed. The decomposition of the nitrite may alternatively be effected by boiling with ferrous sulphate alone, without acid, or nitric oxide may be liberated by the action of potassium iodide and dilute hydrochloric acid. According to Winogradsky's method the nitrite is converted into nitrate by titration with permanganate, and the total nitrate then determined as nitric oxide by Schloesing's method. The author has worked out another method based on the oxidation of the nitrite by permanganate and the determination of the total nitrate as ammonia after reduction by zinc and iron in alkaline solution. The titration with permanganate is carried out in acid solution. On account of the volatility of nitrous acid, the nitrite solution is added from a burette to the permanganate, previously acidified with sulphuric acid and heated to 40° C. Towards the end, the nitrite solution must be added quite slowly because the discharge of the pink colour requires some time; after the titration it is advisable to add permanganate until a faint permanent pink is restored. For the nitrate determination it is necessary to take a quantity equivalent to between 10 and 30 mgrms. of nitrogen; with less than 10 mgrms. the results are too high, and with more than 30 mgrms. they are too low. If it is necessary to employ a larger quantity of the solution than for the nitrite titration, a corresponding quantity of acidified permanganate must be added to oxidise the nitrite. With pure mixtures of nitrite and nitrate the method is accurate to within 1–2 mgrms. of total nitrogen per 100 c.c., and affords a good measure of the course of the nitrifying process, provided ferrous salts are excluded from the culture medium. The media generally contain citric acid, but the action of the latter on permanganate is so slow that the nitrite titration is not appreciably affected on this account, although a small constant error may be introduced by the action of certain other reducing substances. The influence of ammonium compounds on the total nitrogen determination is avoided by boiling the liquid with alkali before reduction of the nitrate. A feasible method for the determination of nitrites and nitrates in the presence of much organic matter, by precipitation with nitron, has been investigated by Franzen and Löhmann (this J., 1909, 546). Nitrites may be eliminated without conversion into nitrates by precipitation with 2,4-diamino-6-hydroxypyrimidine sulphate, or may be converted into methyl nitrite with standard hydrochloric acid and methyl alcohol and the loss of acidity determined by titration. The residual nitrate may then be reduced to ammonia.—J. F. B.

*Sulphates; Volumetric determination of —.* Vansteenberghie and Bauzil. Ann. Chim. Analyt., 1918, 23, 210–214.

The solution is heated at 60°–70° C. with an excess of sodium carbonate, filtered, the filtrate acidified with hydrochloric acid, and boiled for 1 min. with a measured excess of 1.22% barium chloride solution. The barium sulphate is collected on a filter, washed, and the excess of barium in the filtrate is precipitated as carbonate by the addition of sodium carbonate. The barium carbonate is collected, washed, and titrated with N/10 hydrochloric acid, using methyl orange as indicator. The method may be used for the determination of sulphates in urine, serum, blood, etc., after these have been suitably clarified.—W. P. S.



*Gas-purification masses; Examination of spent* —. A. Müller. Chem.-Zeit., 1918, 42, 457.

The suggested conventional method of determining sulphur and cyanogen (Prussian blue) in the spent purification sludge from gas-works (this J., 1918, 195 a) is open to criticism. In the determination of the sulphur errors may easily result during the measuring of the carbon bisulphide solution, whilst there is no proof that extraction of the sulphur is complete. In the author's experience Drehschmidt's method of extraction (J. Gasbel., 1892, 269) is much more trustworthy and rapid. The extraction crucible is a cylindrical porcelain vessel with a perforated bottom, upon which is placed a layer of glass-wool and a compact layer of asbestos. From 15 to 20 grms. of the sample is dried in this crucible at about 80° C. until constant in weight, the crucible then introduced into the extraction apparatus, and the sulphur extracted with about 70 c.c. of carbon bisulphide. For the determination of Prussian blue Feld's method (J. Gasbel., 1904, 545) is more rapid and accurate than Knublauch's method (this J., 1889, 732) based on the interaction of copper sulphate and potassium ferrocyanide:—Two grms. of the spent material is triturated in a mortar with 1 c.c. of ferrous sulphate solution (278 grms. per litre) and 5 c.c. of sodium hydroxide solution (320 grms. per litre), and, after 5 mins., 30 c.c. of magnesium chloride solution (610 grms. per litre) is slowly stirred in, and the whole washed into a distillation flask with sufficient hot water to make up about 200 c.c. After about 5 mins. 100 c.c. of boiling mercuric chloride solution (27.1 grms. per litre) is added to the boiling liquid, and the boiling continued for about 10 mins. The flask is then connected with the distillation apparatus, the receiver of which is charged with 20 c.c. of sodium hydroxide solution (80 grms. per litre), and after the introduction of 30 c.c. of dilute sulphuric acid (392 grms. per litre), the liquid is distilled for about 20 to 30 mins. The distillate is made up to 250 c.c., a trace of lead carbonate being added to remove any turbidity due to sulphur, and 100 c.c. is treated with 5 c.c. of potassium iodide solution (41.5 grms. per litre) and titrated with N/10 silver nitrate solution, 1 c.c. of which is equivalent to 0.009556 gm. of Prussian blue,  $\text{Fe}_7(\text{CN})_{12}$ .—C. A. M.

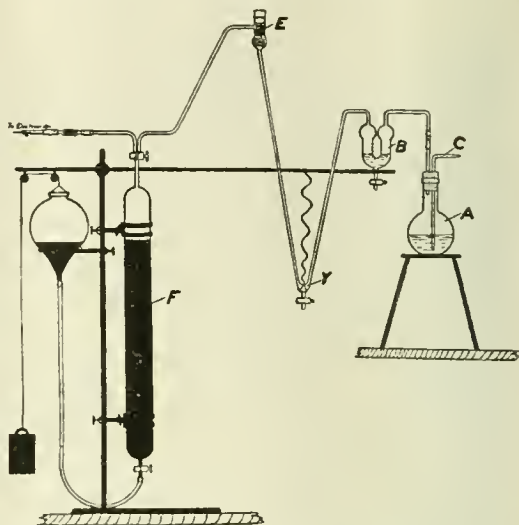
*Cyanogen products [; Reactions of —].* V. Macri. Boll. Chim. Farm., 1918, 57, 261–265.

CRUDE carbonylferrocyanide from the wash waters of Laming's gas-purifying mixture was purified, and the pure product, and also alkali cyanide, thiocyanate, and ferrocyanide, were subjected to the action of a number of reagents; the results are tabulated. The carbonylferrocyanide is a highly stable compound.—T. H. P.

*Radium emanation; Apparatus for the separation of — and its determination electroscopically.* J. E. Underwood and H. Schlundt. Amer. Electrochem. Soc., Sep.—Oct., 1918. [Advance copy.] 7 pages.

The apparatus (see fig.) consists of a flask, A (150 c.c.), provided with a rubber stopper, a sealed inlet tube, C, and an outlet tube which is connected with a U-tube, B, containing concentrated sulphuric acid. B is connected, through a bent tube, Y, and a trap, E (containing glass wool impregnated with sulphuric acid, and an upper layer of cotton), with a gas burette, F (200–300 c.c.), containing mercury. In conducting a determination of radium in monazite sand or concentrate, the mercury is run to the top of the burette and the stopcock closed. To the flask, containing a weighed quantity (10 grms.) of the sample, about 75 c.c. of concen-

trated sulphuric acid is added and the flask attached quickly to the apparatus. The burette is then connected with the flask, the levelling vessel lowered, and the sample boiled with the acid under somewhat reduced pressure. When decomposition is complete the flame is removed and the tip of the inlet tube, C, broken, thus admitting air by which the emanation remaining in the flask and train is carried into the burette. After standing for about 10 mins. to allow the thorium emanation to decay, the gas is transferred from the burette to an



ionisation chamber previously exhausted, the gas left in the connecting tubes of the electroscopie being swept into the chamber by fresh portions of air drawn into the burette through the inlet tube, C. The ionisation current in the electroscopie is measured after a lapse of three hours when, the calibration constant of the instrument being known, the quantity of emanation is readily deduced. To the value so obtained is added that fraction of the emanation which is continuously emitted at ordinary temperatures, and for which a determination on a separate sample is necessary. —W. E. F. P.

*The compound  $\text{H}_2\text{B}_4\text{O}_7$  and its salts [borites].* R. C. Ray. Chem. Soc. Trans., 1918, 113, 803–808.

THE product obtained by heating a mixture of magnesium powder and anhydrous boron trioxide (passing a 120-mesh sieve) to a bright red heat in a current of hydrogen contains magnesium borite,  $\text{MgB}_4\text{O}_7$ . This has not been isolated, but its aqueous solution, when treated with the equivalent amount of potassium hydroxide and concentrated after filtration, yields the potassium salt,  $\text{K}_2\text{B}_4\text{O}_7$ , crystals stable in the absence of moisture. Determinations of the molecular weight in water by the cryoscopic method indicate that the formula is  $\text{K}_2\text{B}_4\text{O}_7$ , not  $\text{KB}_2\text{O}_3$ .—C. S.

*Wood charcoal in jacketed vessels for the storage of liquid oxygen; Spontaneous explosion of —.* L. Wöhler. Ver. deut. Chem., Sep., 1918. Z. angew. Chem., 1918, 31, 192.

LIQUID oxygen is stored in double-walled metal containers, the vacuum jacket being packed with specially prepared wood charcoal. One c.c. of good "vacuum charcoal" will absorb, at the temperature of liquid air, 1 litre of oxygen and 1 litre of nitrogen, which is about double the absorption capacity of ordinary wood charcoal. The charcoal

packing thus protects the evacuated space against gradual small leakages and prolongs the efficiency of the container. Cases have occurred where in consequence of an injury to the inner wall, liquid oxygen has penetrated to the charcoal, causing a violent explosion. Not every kind of "vacuum charcoal" is explosive, and ordinary wood charcoal is quite indifferent. The activity of the explosive charcoal has been traced to the presence of iron in the form of ferric oxide, which acts as an oxidation catalyst. The heat generated by the absorption of oxygen by the "vacuum charcoal" of high absorption capacity serves as an initial exciter for the catalytic oxidation and the combination of high absorption capacity with the presence of ferric oxide is a necessary condition, since ordinary charcoal, even with the addition of ferric oxide, is non-explosive. "Vacuum charcoal" becomes explosive in presence of 2–3% of iron in the form of ferric oxide deposited *in situ*, not merely mixed as a powder. The percentage of iron in the sample is, however, no guide to its dangerous or safe character, since if the iron be concentrated in certain portions of the sample, the explosion may start in these portions and extend to the remainder of the mass. Explosions have been recorded with charcoal having an average content of 0.6% of iron.

—J. F. B.

*Zirconia.* Meyer. See VIII.

*Refractory materials.* Mott. See VIII.

*Hypochlorite solutions.* Cullen and Austin. See XIXb.

#### PATENTS.

*Phosphoric acid solutions; Process of treating* —. F. K. Cameron, Washington, D.C. U.S. Pat. 1,276,870, 27.8.18. Appl., 11.11.16.

AN aqueous solution containing not less than 12% of phosphoric acid is treated with sufficient excess of gaseous ammonia under pressure to precipitate the whole of the phosphoric acid as tri-ammonium phosphate. A portion of the ammonia is afterwards recovered by converting the tri-ammonium phosphates into an ammonium phosphate or mixture of phosphate containing a smaller proportion of ammonia.—I. A. C.

*Potash or potassium salts; Extraction of* — from potash-bearing materials. E. A. Ashcroft, London. Eng. Pat. 119,492, 28.8.17. (Appl. 12,363/17.)

To extract potash from potash-bearing materials, such as felspar, muscovite, micas, and the like, the material, either alone or mixed with a reducing material (e.g., carbon or a sulphide) is suspended in a fused medium of sodium chloride or potassium chloride, or both, and treated with chlorine gas at a suitable temperature. A chlorine carrier or catalyst (e.g., an iron salt) may be added with advantage, and the operation is preferably conducted in a converter. The potassium is converted into chloride, which is extracted with water.

—J. H. P.

*Hydrogen peroxide; Stable* — and method of making the same. A. Schaidhauf, Frankfurt, Germany, Assignor to Roessler and Hasslacher Chemical Co., New York. U.S. Pat. 1,275,765, 13.8.18. Appl., 2.8.12.

A NON-ACID solution of hydrogen peroxide is treated with an insoluble aluminium compound, giving as a stable end product a solution of hydrogen peroxide with "sensible proportions" of the insoluble aluminium compound.—C. A. K.

*Manganese compounds [dioxide]; Making* —. C. Ellis, Montclair, N.J., Assignor to National Carbon Co., Inc., New York. U.S. Pat. 1,276,739, 27.8.18. Appl., 27.10.17.

RAW material containing basic manganese compounds is heated with acid sodium sulphate to form manganese sulphate which is then converted into manganese dioxide.—W. E. F. P.

*Tungsten trioxide; Method of producing finely divided* —. C. A. Pfautstiehl, Waukegan, Assignor to Pfautstiehl Co., Inc., Chicago, Ill. U.S. Pat. 1,277,475, 3.9.18. Appl., 19.11.14.

AN aqueous solution of ammonium paratungstate is treated with nitric acid in a very finely divided state to precipitate tungsten trioxide as a fine powder.—J. H. P.

*Aluminium oxide; Process of manufacturing* — from aluminium silicate or a substance containing aluminium silicate and obtaining sodium carbonate as a by-product. Y. Takeshima, Tokyo, Japan. U.S. Pat. 1,277,503, 3.9.18. Appl., 23.10.17.

A MIXTURE of aluminium silicate, calcium carbonate, sodium sulphate, and carbon in the molecular proportions corresponding to:  $x\text{Al}_2\text{O}_3$ ,  $y\text{SiO}_2 + 2x\text{Na}_2\text{SO}_4 + (2x + 2y)\text{CaCO}_3 + 4x\text{C}$ , is extracted with water after roasting. Carbon dioxide is passed into the solution obtained to precipitate aluminium hydroxide and obtain sodium carbonate as a by-product.—J. H. P.

*Lime; Process of slaking* —. W. E. Carson, Riverton, Va. U.S. Pat. 1,277,855, 3.9.18. Appl., 11.10.17.

To obtain a dry hydrated lime, quicklime is mixed with a quantity of water "sufficient for hydration and evaporation," the chemical action allowed to proceed for a time until a predetermined temperature has been reached, and the mixture then suddenly chilled to a lower temperature at which the slaking is completed. A catalyst may be added.

—J. H. P.

*Hydrogen; Manufacture of* —. C. T. Thorssell and H. L. R. Lundén, Gothenburg, Sweden. Eng. Pat. 119,591, 9.1.18. (Appl. 549/18.)

IRON sponge in the form of thin leaves or balls is impregnated with a strong solution of sodium or potassium carbonate or hydroxide, and dried at a moderate temperature. Alternatively the sponge may be pulverised, mixed with 10% of its weight of sodium carbonate, and water, and the mixture moulded and dried. When used in the manufacture of hydrogen by alternate oxidation by steam and reduction by reducing gas, the activity of this material does not deteriorate.—W. F. F.

*Wool-scouring liquors.* U.S. Pat. 1,277,784. See V.

*Electrolytic cells.* Eng. Pat. 118,355. See XI.

### VIII.—GLASS; CERAMICS.

*Glass furnaces; Formation of certain rock-forming minerals in and about* —. G. V. Wilson. J. Soc. Glass Tech., 1918, 2, 177–216.

AN examination of material from a glass furnace at Kinghorn, Fife, which had burst owing to the solvent action of the molten glass on the floor of the tank, and of other specimens showed that under certain conditions wollastonite, augite, tridymite,



and quartz can all crystallise out from the same melt, the wollastonite crystallising out before the tridymite, and the latter occurring only where the mass cools slowly. Felspar (oligoclase), magnetite, and biotite (black mica) are formed by the corrosive action of hot molten glass on aluminous materials, such as bricks and pots. Wollastonite, melilite,  $3\text{CaO} \cdot 2\text{SiO}_2$ , and augite are also produced in fragments of limestone enclosed in molten glass, and generally as a result of the contact alteration of limestones by igneous materials. Augite is formed first and removes the bulk of the magnesia, some alumina, and some iron from the melt: the remaining alumina then combines with lime, silica, and a little soda, magnesia, and iron to form melilite, and the excess of lime and silica separate partly as wollastonite and partly as  $3\text{CaO} \cdot 2\text{SiO}_2$ . Sillimanite and corundum are formed by changes which occur in the refractory materials (clays) used, these changes being influenced by the presence of a little glass or other fluxes. The corundum appears to be formed from a part of the sillimanite which has been dissolved or re-dissolved in the mass. The effect of the gradual absorption of soda by bricks or glass pots is shown in a diagram. As the brick is gradually destroyed, the sillimanite first formed disappears, its silica and part of its alumina going to form felspar and feldspathoid molecules, whilst the remainder of its alumina separates as corundum. The absence of pseudowollastonite ( $\alpha\text{CaSiO}_3$ ) shows that the temperature of the molten glass at the commencement of crystallisation was not above  $1190^\circ\text{C}$ . (inversion temperature of pseudowollastonite to wollastonite). The absence of cristobalite indicates that the temperature was at no time as high as  $1450^\circ\text{C}$ . (inversion temperature of cristobalite to tridymite). The presence of quartz crystals in the amethyst glass in the lower part of the material suggests that the latter remained fluid below  $870^\circ\text{C}$ . (inversion temperature of tridymite to quartz). The absence of quartz in the main body of the glass shows that the latter had solidified at  $870^\circ\text{C}$ . or had become too viscous to allow of crystallisation. Molten glass has a rapid solvent action on the common bricks with which it comes in contact, forming oligoclase felspar with a little biotite and magnetite. A fragment of firebrick which had been for a long time enclosed in molten glass was full of sillimanite needles, except in the areas in contact with the glass. The action of molten glass on the refractory materials in the furnace results in the abundant formation of corundum on the inside of the crown of the furnace, and sillimanite in the sides and bottom. The sillimanite is normally only developed in the vitrified portions of the pot or brickwork. The junction between the glass and an aluminous pot or brickwork is almost a straight line, but the surface of a silicious refractory material is much corroded by the glass. Hence, the clays used for glass pots should be as aluminous as possible. The various minerals were separated as follows: Sillimanite was left after treating the material with dilute hydrofluoric acid. Corundum was left after boiling fragments of the material with fused sodium carbonate for several hours and treating the residue with strong hydrochloric acid. Wollastonite and tridymite were separated by gravitation in a mixture of bromoform and benzol of such density that the wollastonite sank and the tridymite floated. The properties by which the different minerals were identified are fully described, and their mode of formation as represented by Rankin and Wright's concentration-temperature diagram for the system lime-silica (this J., 1915, 139) and Shepherd and Rankin's corresponding diagram for the system silica-alumina (this J., 1909, 1124; 1911, 543) is discussed.—A. B. S.

*Glass pots; Firing of* —. M. W. Travers. J. Soc. Glass Tech., 1918, 2, 170–174.

IN gas-fired glass furnaces, the empty pot should be completely enclosed in the furnace (this J., 1918, 235 T) and heated to  $1350^\circ\text{C}$ . for three days or until the pot material is completely vitrified and has a thick and even layer of sillimanite. A vitrified pot is not corroded by glass, but a biscuit one is rapidly attacked when at a temperature of  $1200^\circ\text{C}$ . or above. Glazing is unnecessary with vitrified pots, but when filling them with glass batch, the temperature should not be allowed to fall unduly.

—A. B. S.

*[Glass] pot failure*. S. N. Jenkinson and P. Marson. J. Soc. Glass Tech., 1918, 2, 175–176.

Pots fail by the bottom cracking when they have not been heated sufficiently before being filled. Distortion occurs when the pots are supported on bricks. In a down-draught furnace the bottom of the pots will be heated more than in an up-draught one and this should lessen the cracking.—A. B. S.

*Glassware; Resistant properties of some types of foreign chemical* —. J. D. Cauwood and W. E. S. Turner. J. Soc. Glass Tech., 1918, 2, 219–235.

WHEN examined by the method described in a previous paper (this J., 1917, S73), glasses containing a moderate proportion of boric and zinc oxides are the most resistant, those in which the zinc is replaced by aluminium being less advantageous. A proportion of boric oxide in excess of 12 mols. per 100 mols. of silica does not increase the stability of the glass. Boric oxide lessens the resistance of glass to alkali and hydrochloric acid. Resistance to acids is increased by raising the silica, but not the boric oxide content of the glass, the resistance to alkali being simultaneously reduced. Resistance to sodium carbonate is greatest in glasses containing alumina. Alkali-lime glasses are not very resistant to water; they behave well with strong caustic alkali, moderately so with dilute caustic alkali, and are attacked strongly by sodium carbonate. They are the best glasses available for resisting acid and are recommended for analytical operations involving the use of acid solutions. Lime glasses are not resistant to water and sodium carbonate. Alumina is a valuable all-round constituent, but may increase the difficulty of melting and working the glass. For chemical purposes, different glasses should be selected according to the reagents employed. Six glasses remained in the "Good" class in each of the six tests applied (boiling water, autoclave at  $183^\circ\text{C}$ ., 2N NaOH, N/10 NaOH, 2N  $\text{Na}_2\text{CO}_3$ , boiling HCl); of these three were British, one American, and two German.—A. B. S.

*Chemical glassware; Effect of the continued action on* — of water, acid, and alkali. J. D. Cauwood and W. E. S. Turner. J. Soc. Glass Tech., 1918, 2, 235–239.

FLASKS selected as representative of the three types of glass made for chemical ware were repeatedly subjected to the action of various fluids. The surface of soda-lime chemical glasses increases in resistance on continued treatment with water or hydrochloric acid. The resistance of borosilicate glasses is slightly increased by repeated treatment with water, but not with hydrochloric acid. Not any of the glasses used for chemical ware are improved by the continued action of either caustic soda or alkali carbonate. The irregularities in the results obtained appear to be due to lack of homogeneity of the glass itself. It is useless to compare the resistance of

different glasses by making only one or two tests when the actual extent of attack is small or the results are fairly close together.—A. B. S.

*Cataphoresis [of kaolin, clay, etc.]: Study of —.*  
S. Kondo. *Kōgyō-Kwagaku-Zasshi* (J. Chem. Ind. Tokyo), 1918, 21, 385—401.

EXPERIMENTS on the cataphoresis of kaolins, ball clay, earthenware-body, and silicic acid gave the following results:—Particles of kaolin suspended in water show a velocity of migration practically proportional to the voltage of the current, the velocity in the case of Georgia kaolin at the potential difference of one volt per cm. and at 20° C. being  $19.2 \times 10$  cm. per second. The velocity of migration of kaolin particles is inversely proportional to the viscosity of the medium, and in time shows a decrease. The velocity is increased by the addition of small quantities of sodium hydroxide to kaolin, but is retarded by increasing the amount beyond a certain limit, e.g. 0.05% in the case of Georgia kaolin. In presence of small amounts of sodium hydroxide, the deposits of kaolin and of earthenware-body particles on the anode were sufficiently compact for weighing. The amount deposited increases with the voltage and strength of the current. The friction between plastic clay briquettes and steel plate is reduced practically to zero in a few seconds by the action of a weak electric current. Cataphoresis of sodium silicate solution yields pure silicic acid. The passage of a direct current causes an accumulation of soluble salts in ball clay at one end of a briquette.—C. A. M.

*Firebricks: Corrosive action of flue dust on —.*  
J. W. Mellor and W. Emery. *Ceram. Soc., Refractories Sect.*, Oct. 17, 1918. [Advance proof.]

THE corrosive action of flue dust on firebricks was investigated by injecting various dusts through a Brayshaw burner into a gas-heated furnace, the time of heating, the velocity of the flue dust, and the nature of the atmosphere being under control. The dust-charged flame impinged on the surface of the brick being tested. The "dusts" used were a boiler flue dust of slate brown colour, a boiler flue dust of a brown colour, deep red-coloured dust from the top of the retort bench of a gas works, dust from the slag chamber between a steel smelting furnace and a regenerator, dust from a blast-furnace Cowper stove, "bull dog," red hematite ore, tap cinder, lime from calcined marble, lime and common salt (1:1), salt, sodium sulphate, salt and potash spar (1:1), and sodium silicate and calcium silicate (1:1). The effects obtained under oxidising conditions were as follows:

Dust	Firebrick	Silica brick	Fine-grained silica brick
Boiler dust high in lime and ferric oxide	d.P.; m.C.	—	—
Ferruginous boiler dust	s.P.; s.C.	—	—
Ochreous dust from retort brick	k.P.; s.C.	d.P.; g.C.	—
Basic slag	d.P.; s.C.	d.P.; m.C.	—
Ferruginous dust high in lime	k.P.; m.C.	d.P.; m.C.	—
"Bull dog"	—	d.P.; s.C.	—
Hematite (reducing flame)	—	d.P.; s.C.	s.P.; s.C.
Tap cinder	d.P.; m.C.	d.P.; m.C.	d.P.; s.C.
Lime	s.P.; m.C.	s.P.; m.C.	—
Lime and salt	s.P.; m.C.	s.P.; m.C.	—
Salt	d.P.; s.C.	s.P.; m.C.	—
Sodium sulphate	s.P.; s.C.	s.P.; m.C.	—
Salt and felspar	s.P.; s.C.	s.P.; m.C.	—
Soda-lime glass	s.P.; s.C.	s.P.; m.C.	—

NOTE.—P=penetration: C=corrosion: s=slight: d=deep: k=complete: m=medium: g.C.=great corrosion or slagging.

The dust penetrates more readily into fireclay bricks than into silica bricks. The bond is usually attacked first and the coarser grains last. In fine-grained silica bricks, the depth of penetration is less than with coarser grained ones. Under oxidising conditions, iron oxide does not corrode silica bricks to any noteworthy extent, but under reducing conditions, ferrous silicate is formed and acts as a corrosive flux. "Bull dog" dust and iron oxide generally corrode fireclay bricks more than silica bricks. The alkaline vapour in some dusts influences the conversion of quartz into a form of silica with a lower sp. gr. When the surface of a brick has been covered with a glaze formed by the action of the dust, the subsequent rate of corrosion is greatly diminished unless the glaze is sufficiently fluid to drip away; the corrosion is then rapid. Attempts to increase the resistance of the brick by applying a blowpipe flame to the surface were unsuccessful. The depth of penetration of the dust cannot be used as the basis of a quantitative measure of the corrosion.—A. B. S.

*Refractory materials; Standardisation of tests for —. Parts I. and II. Report of Committee of Ceramic Soc., Refractories Sect.*, May 15 and Oct. 18, 1918. [Advance proof.]

I. *Analysis of fireclays, ganisters, quartzose rocks, and manufactured products.*—*Hygroscopic moisture* is determined by drying about 5 grms. of the finely ground sample at 110° C. to constant weight. *Loss on ignition* is determined by heating about 1 gm. of the dry sample in a platinum crucible, first over a small flame, then over a Meker or Teclu burner, and finally over a blowpipe or in a muffle for about 20 mins., cooling, and weighing. After re-heating over the blowpipe for about 5 mins. the cooled crucible is re-weighed. *Silica* is determined by fusing 1 gm. of the sample with 10–15 grms. of sodium carbonate and treating the product with water and hydrochloric acid. The liquid is evaporated to dryness and the residue heated in an air oven at 110° C., moistened with hydrochloric acid, 60–70 c.c. of hot water added, and, after filtering, the residue is washed with hot water until free from chlorides, and is set aside. The filtrate is evaporated to dryness, heated as before, again treated with acid and water, filtered, and the residue washed. The two filter papers and contents are ignited slowly, then over a blowpipe for 15 mins. with the lid off, then finally 5 mins. with the lid on. The residue is weighed as "uncorrected silica"; it is treated with water, sulphuric acid, and hydrofluoric acid, evaporated to dryness, ignited over a burner for 5 mins., and weighed. The loss in weight represents silica. *Alumina, iron, etc.*, are determined by adding solid ammonium chloride and ammonia to the filtrate from the silica, the latter being at 80°–90° C., and filtering off the precipitate immediately. The residue is washed, dissolved in hydrochloric acid, and the alumina, etc., is re-precipitated by adding an excess of ammonia. It is filtered off, and washed with a solution containing 5 c.c. of concentrated nitric acid and excess of ammonia in 1 litre. The combined filtrates are evaporated to about 100 c.c., ammonia is added, and the precipitate filtered off and washed with the ammonium nitrate solution. The three filter papers, with contents, are dried in the platinum crucible containing the "silica residue," ignited, cooled, and weighed. The precipitate is fused slowly with 5–6 grms. of fused potassium bisulphate or pyrosulphate, dissolved in dilute sulphuric acid, made up to 250 c.c., and retained for determining the iron and titanium. *Ferric oxide* is determined by the ordinary permanganate process, or preferably colorimetrically with potassium thiocyanate, and



standard iron solution containing 0.6302 gm. of ferric potassium alum and 5 c.c. of concentrated sulphuric acid per litre. When the permanganate process is used, the iron is reduced with sulphur dioxide, excess of which is removed by boiling, and finally by a current of carbon dioxide so as to avoid the reduction of titanium oxide. *Titanium oxide* is determined colorimetrically in the solution of the alumina, etc., precipitate by adding hydrogen peroxide and comparing the colour with that of 10 c.c. of a standard solution (containing 1 gm. of pure titanium oxide, 10 grms. of potassium pyrosulphate, and 10 c.c. of concentrated sulphuric acid per litre), diluted until the two tints are alike. The weights of iron and titanium oxides are deducted from the total weight of the alumina, etc., precipitate, and the difference is reported as alumina. *Lime* is precipitated as oxalate in the boiling filtrate from the alumina, etc., the solution is allowed to stand on a water-bath for two hours, filtered, the residue washed once, dissolved in nitric acid, and re-precipitated as before, the precipitate being washed well with hot water, dried, ignited, and weighed as lime. *Magnesia* is precipitated in the filtrate from the lime precipitate by sodium ammonium phosphate. The precipitate is filtered off, dissolved in nitric acid, and re-precipitated, collected in a Gooch crucible, washed with cold ammonia solution (1:8), ignited, and weighed as magnesium pyrophosphate. *Alkalis* are determined by the Lawrence-Smith method on 0.5 gm. of the finely powdered sample and are weighed as mixed chlorides. The potash is then precipitated by evaporating the chlorides with 30% perchloric acid and then with 10 c.c. of water. A mixture of 97 vols. of absolute alcohol, 3 vols. of water, and 0.25 vol. of perchloric acid is added, and the mixture filtered through a weighed Gooch crucible. The residue is washed with the alcohol mixture, dried at 120° C., and weighed as potassium perchlorate.

II. *Analysis of dolomite and magnesite.*—*Hygroscopic moisture* is determined as in I. *Loss on ignition* is determined as in I., but the ignition over a blast burner should be prolonged another 10 mins. *Alkalis* are determined as in I. *Silica* is determined by fusing 1 gm. of the sample as in I., care being taken to use an excess of acid in washing the silica. Two evaporations are seldom necessary. *Alumina, etc.* is determined after diluting the filtrate from the silica to about 300 c.c., as described in I., except that the re-precipitation of the alumina, etc., is seldom necessary. *Lime* is determined as in I. after diluting or evaporating the filtrate from the alumina, etc., to about 300 c.c. *Magnesia* is determined as in I. after concentrating the combined filtrates from the lime to about 300 c.c. Washing must be very thorough, and the material must be ignited throughout to at least 800° C. before weighing. *Phosphorus and sulphur* are determined by fusing 1 gm. of the sample with sodium carbonate, as for silica, but using a sulphur-free flame. The silica is removed as usual, the filtrate diluted to 500 c.c., and 250 c.c. taken for the phosphorus and sulphur respectively. Sulphur is precipitated as barium sulphate in the faintly acid solution, heated to 90° C., by barium chloride added slowly, with constant stirring, filtered after 3–4 hours, and the precipitate washed, ignited, and weighed. *Phosphorus* is determined by neutralising with ammonia, adding ammonium nitrate and nitric acid, heating to about 80° C., and precipitating with ammonium molybdate with constant stirring. After standing 3 or 4 hrs. the solution is filtered, the residue dissolved in ammonia, and re-precipitated with ammonium nitrate and ammonium molybdate, heated to 80° C. until gas bubbles begin to form, acidulated with hot 5% nitric acid with

constant stirring, and after standing 3 or 4 hrs. filtered through a Gooch crucible, the precipitate washed with the mixture previously employed, dried, ignited at 800° C., and weighed as  $24\text{MoO}_3 \cdot \text{P}_2\text{O}_5$ .

III. *Identification of various forms of silica in silica bricks.*—A suitable method of identification is by means of the refractive indices, which are respectively 1.549 for quartz, 1.477 for tridymite, and 1.484 for cristobalite. Becke's bright line method may be used, but preferably the material is immersed in a solution of mercury potassium iodide in water diluted until its refractive index for sodium light is 1.477; any tridymite present will then show ultramarine fringes, cristobalite, orange fringes, and quartz will be fringed with white light.

IV. *Porosity, water absorption, and specific gravity tests.*—A portion of the sample is cut into cubes of 2 in. side, dried at 110° C., weighed, soaked overnight in water under reduced pressure, and the sp. gr. determined by weighing in water. The surface is wiped and the piece is again weighed; the increase shows the amount of water absorbed.

V. *Shrinkage of clays on drying and firing.*—The clay is moulded to form a block measuring  $4\frac{1}{2}$  in.  $\times$   $1\frac{1}{2}$  in.  $\times$   $\frac{1}{2}$  in. Two thin vertical lines are drawn on it about 9 cm. apart, their distance being accurately measured. The distance is again measured after drying the test-pieces at the ordinary temperature, then for 4 or 5 hrs. at 70°–80° C., and finally at 110° C. Each test-piece is also before and after drying weighed to ascertain the amount of water it contains. The test-pieces are fired in a muffle or sagger at the rate of 60° C. per hour to 900° C., and afterwards at 20° C. per hour, along with Seger cones to indicate the temperature. Test-pieces are drawn at the following cones and corresponding temperatures:—010a (900° C.), 08a (940°), 05a (1000°), 02a (1060°), 1a (1100°), 4a (1160°), and 6a (1200°) for lower temperatures, or at 010a (900° C.), 05a (1000°), 1a (1100°), 4a (1160°), 6a (1200°), 8 (1250°), 10 (1300°), 12 (1350°), 14 (1410°), and 16 (1460° C.) for higher temperatures, and the shrinkage, porosity, water absorption, and specific gravity determined, and the colour changes and hardness noted.

VI. *Tensile strength of dried clays* is measured in a machine similar to that used for cement. The test-pieces must be dried slowly and regularly and must break accurately in the middle. The average of five or six tests should be taken.

VII. *After-contraction or after-expansion* is determined as directed in the Gas Engineers' Specification (this J., 1911, 28, 804; 1912, 642; 1917, 701, 1273).

VIII. *Normal refractoriness* is determined on a test-piece of the material cut to the shape of a Seger cone (about  $1\frac{1}{2}$  in. high), as described in the Gas Engineers' Specification (*loc. cit.*).

IX. *Refractoriness under load* is determined in a similar manner to normal refractoriness, but the test-piece is a rectangular block  $3\frac{1}{2}$  in. long and 2 in. by 2 in. cross-section. The load is applied through a lever. The rate of heating should be such that Seger cone 16 (1460° C.) squats in 4 hrs.

X. *The thermal expansion and the hot and cold sizes of firebricks* are determined by Coppée's method (see this J., 1917, 1273).

XI. *The crushing strength of cold materials* is determined in a machine in which the load is applied with the utmost regularity. Hand-operated machines are not satisfactory. The opposite faces of the sample must be ground accurately parallel. At least five bricks should be tested.

XII. *Grading.*—Refractory materials should be tested for grading by means of standard sieves, preferably those established by the Institution of

Mining and Metallurgy (this J., 1907, 1240) in which the wire and aperture or mesh are of equal diameter. The material is made into a slip and washed through each sieve in turn, the residue on each sieve being afterwards dried and weighed. For the materials used in the manufacture of silica bricks, a set of three sieves (Nos. 5, 50, and 120) usually suffice. For finer materials, the use of a No. 120 and a No. 200 sieve, followed by elutriation to separate particles of less than 0.01 mm. diam. is desirable. Schoene's elutriator (omitting the manometer) is recommended, the water having a velocity of 0.18 mm. per sec. to remove particles of less than 0.01 mm. diameter. To obtain comparable results, distilled water should be used and the addition of acids, etc., to facilitate the disintegration of the material should be avoided, but a little ammonia may be used. To break up adventitious clots, the sample should be mechanically shaken with water for about 4 hours; some clays require a longer treatment. The material is then passed successively through a No. 120 and a No. 200 sieve and into the elutriator. In order to compare the fineness of two or more clays, the surface factor should be calculated (Trans. Ceram. Soc., 1910, 9, 94).

XIII. *Corrosive action of flux dusts.*—See preceding abstract.—A. B. S.

*Silica bricks; Constitution of* —. A. Scott. Trans. Ceram. Soc., 1917-18, 17, 459-474.

The chief properties required in rocks for the manufacture of silica bricks are a high percentage of silica, a small percentage of impurities—insufficient to reduce the refractoriness unduly, but large enough to accelerate the conversion of the quartz—and a structure which will give angular fragments when crushed. At least seven crystalline modifications of silica exist and one or more of them are present in a silica brick at some stage in its history. In the kiln, the bond (usually lime or clay) reacts with some silica dust to form silicates, and the remaining fine-grained quartz is converted first to cristobalite and afterwards to tridymite. The larger grains of quartz and ganister are attacked and dissolved by the fused silicates and are reprecipitated as cristobalite. When a fine-grained cement is present in the ganister used, conversion continues wherever the cement occurs. Some American bricks examined by the author contained only about 25% of unaltered quartz; the average in five British bricks made by different firms was 45%. The difference is due to the American bricks being burned at a higher temperature (cone 15 and above) and for a much longer time than those made in this country. Bricks which have been used in a furnace show a steady reduction in quartz and a corresponding increase in cristobalite and tridymite from the end which has been coldest to that which has been hottest. At a temperature of 1550° C., the matrix of a silica brick appears to consist of a very viscous, molten, saturated solution of silica in silicates of calcium, etc., and as the brick cools, the soluble silica crystallises out as well-developed skeletons of cristobalite, which may later be converted into tridymite. A piece of brick consisting wholly of tridymite was kept at 1550° C. for 2 weeks and was then quenched; the tridymite was unchanged, showing (in opposition to Fenner, this J., 1913, 22) that this is the most stable form of silica at high temperatures. When bricks in a furnace are attacked by dust, volatilised alkalis, etc., there is a greater formation of glass and a high proportion of cristobalite. Bricks in the roofs of steel furnaces, exposed mainly to iron oxides, exhibit several distinct layers; starting from the hot end of the bricks, these are: (i) A grey material show-

ing little trace of the original material, having apparently been molten, and consisting largely of tridymite with some cristobalite, and, in some cases, numerous minute particles of magnetite which fill the interstices between the tridymite crystals. In other instances, cristobalite predominates in the grey part of the bricks, together with some tridymite and numerous small skeletal crystals of fayalite. (ii) A black material partly structureless, but showing traces of the original structure and an appreciable amount of tridymite with more black magnetic iron oxide than in the grey portion. This magnetite occasionally reacts with the silica forming fayalite. (iii) A pale-coloured material resembling the unaltered brick, but containing a considerable proportion of cristobalite. (iv) A red material in which the matrix is partly converted into a very fine-grained aggregate, mainly cristobalite, and thin plates of hematite which produce the colour. (v) A narrow layer of unaltered material at the extreme end. The proportion of unaltered quartz falls steadily from the hot end to a point slightly beyond the centre of the brick, probably due to some local concentration of the fluxes. Bricks in the port of a steel furnace are penetrated for about 10 cm. by iron oxide. Sections of the lighter-coloured portions of the bricks resemble unglazed bricks, but show a greater conversion of the quartz in the matrix, the rock fragments being unaltered. Towards the hot end, the tridymite crystals are larger and the size of the rock fragments diminishes rapidly. Even at the hot end of such bricks some unaltered quartz exists with a matrix containing tridymite, iron oxide, and red crystals of fayalite. The amount of unaltered quartz probably depends on the size of grains of the original rock and on the port bricks being heated for a shorter time than those in the roof. The "slagging" or "dripping" of silica bricks in furnaces depends largely on the reduction of any iron oxide to the ferrous state, the ferrous oxide then reacting with the silica to form fayalite.

—A. B. S.

*Silica products.* A. Bigot. Ceram. Soc., Refractories Sect., Oct. 18, 1918. [Advance proof.]

SILICA products (especially the bricks used in steel furnaces) should not be softened, deformed, or show signs of fusion when heated to 1710° C. nor should they disintegrate on cooling. If a silica rock which expands considerably on heating to this temperature is to be used, it must be finely ground. Edge-runner mills should not be used for grinding the silica rock as they tend to produce rounded grains. Cylinder mills and other appliances used in the abrasive industry and yielding sharply angular grains are preferable. The bricks, etc., should be burned at about 1600° C., preferably in a tunnel kiln. Silica bricks taken from the arches of a Martin furnace at Creusot's works showed a zone of unaltered material and a second brown zone with white grains of unaltered material disseminated throughout its mass, but diminishing in number and size as the third zone was approached; this third zone was grey and devoid of grains of silica. On prolonged use the grey zone gradually increased and the brown zone diminished or invaded the zone of unaltered material. The brown material contained 78.45% SiO<sub>2</sub> and 13.10% Al<sub>2</sub>O<sub>3</sub>, as compared with 84.6% and 9.25% respectively in the grey material. The brown material also contained slightly larger proportions of lime, magnesia, manganese oxide, and alumina. The iron in the grey material was in the ferrous state (due to the penetration of the reducing gases in the furnace) and ferro-magnetic; in the brown material about half of it was attracted by a magnet. The brown



material melted abruptly and completely at 1710° C. The grey and unaltered materials passed through a pasty condition before melting at 1730° C. Under the microscope, the grey and brown materials showed a complete transformation of the silica into cristobalite and tridymite. The brown material contained much tridymite and free silica; the grey material presented a microgranular mass of cristobalite without action on polarised light. The crushing strength at ordinary temperatures of the brown and grey materials was greater than that of any commercial refractory material. The crushing strength at 1500° C. of the grey material was superior to that of all commercial refractory materials except carbon. The results obtained show that, contrary to general opinion, some silica bricks containing only 80% SiO<sub>2</sub> are as refractory as others containing 95% SiO<sub>2</sub> and have a much greater crushing strength than the latter. The proportion of lime and iron oxide in the grey material was 10–14%, whereas the permissible maximum in good silica bricks is usually considered to be 4%. These results are confirmed by previous investigations on enamelled silica bricks from the Temple of Darius (circa 500 B.C.) which contain about 11% of non-silicious matter and do not melt below 1730° C. On determining the crushing strength of refractory materials at various temperatures and plotting the results on a temperature-strength diagram, the author and V. Bodin found that clays, bauxites, silica products, and carborundum behave at first like a plastic substance and show a gradual reduction of strength with increasing temperature, followed by a rapid increase and a well-defined maximum strength at 1000° C., whilst chromite, magnesite, etc. show no such maximum. The only silicious material which behaved abnormally was the grey material previously mentioned; this did not soften on heating, but was disintegrated at 1500° C. The remarkably sharp maximum strength at 1900° C. shown by clays and similar materials may be associated with the gradual decrease in the expansion of materials which was observed by Edwards and Rigby (Trans. Ceram. Soc., 1917, 16, 271; Mellor, this J., 1917, 1273). If this is the case, magnesite and chromite should not show a decreasing expansion when heated. The temperature-strength graph furnishes new data which may be introduced into specifications for refractory materials.—A. B. S.

*Silica and other refractory bricks made from non-plastic materials.* G. W. Mottram. Ceram. Soc., Refractories Sect., Oct. 17, 1918. [Advance proof.]

REFRACTORY bricks should be made of particles of selected sizes, in such proportions as to give a product with the required characteristics. In the customary method of grinding ganister, calcined fireclay (grog), and other non-plastic materials there is usually a deficiency in the proportion of very fine material. Fireclay was finely ground with two or three times its weight of calcined Stourbridge fireclay and the product was mixed with ground calcined Stourbridge fireclay so as to produce a mixture containing 90% of calcined and 10% of raw clay. Bricks made from this mixture showed no contraction after repeated burning, and retained their sharp edges after a week's exposure in the port of an open-hearth steel furnace. The same principle may be applied to the grading of silica or other brickmaking material, grinding 15–30% of it with lime or other binder and then adding it to the remainder of the graded material. The durability of the product is two or three times as great as that made when the usual method of grinding is used.—A. B. S.

*Magnesite as raw material [for refractories].* T. Crook. Ceram. Soc., Refractories Sect., Oct. 17, 1918. [Advance proof.]

The magnesite used for making refractory bricks should be dead-burned and crystalline, with not more than 5 or 6% of silica, alumina, and lime, and not more than 1% of iron oxide. Calcining must be effected at or above 1400° C., depending on the nature of the magnesite used. The sintered magnesite should be pulverised and graded before being made into bricks. The bricks should not shrink more than 15% when burned. The success of the Austrian magnesite bricks appears to be due to careful selection of material, efficient calcining, appropriate grading, and the use of sufficient pressure in making the bricks. The magnesites used for refractory purposes are spathic breunnerite (Styria), spathic magnesite (Quebec, Washington, and Norway), compact magnesite (Greece, California, Italy, India, and Australia), and hydromagnesite (Atlin, B.C.). The spathic varieties are coarsely crystalline and show definite cleavage; the compact or crypto-crystalline varieties show no cleavage. Spathic magnesite and breunnerite usually contain lime as an impurity; compact magnesite frequently includes an admixture of quartz, serpentine, and other forms of silica, but little lime and iron. Breunnerite—the variety of magnesite from which the Austrian magnesite bricks are made—contains a variable percentage of ferrous carbonate (chalybite) in isomorphous admixture. It has an average sp. gr. of 3.15, whilst that of pure magnesite is only 3.02. On ignition the ferrous carbonate is converted into magnetite which gives the calcined material its characteristic black colour. The blackness and magnetic properties of the calcined breunnerite facilitate the separation of dolomite, calcite, and other impurities, which are not easily distinguished in the other varieties of calcined magnesite. Breunnerite also loses its carbon dioxide at a rather lower temperature than other forms of magnesite. The Austrian deposits are extensive, and the material is so easily quarried, readily purified, and cheaply burned by means of brown coal found in the locality that the sintered product can be placed on the market much more cheaply than other varieties of sintered magnesite. Breunnerite also occurs in Quebec, Mysore, and in uncommercial quantities in the United Kingdom. Spathic magnesite resembles marble and dolomite in texture, but has a slightly higher specific gravity. The adventitious dolomite, serpentine, and other impurities in it should be separated as far as possible at the quarry, as they are less easily recognised in the burned material. Spathic magnesite is characterised by a very low iron content (seldom above 2%). Compact or crypto-crystalline magnesite usually contains silica in the form of colloidal silica, quartz, or magnesium or other silicate. Before use, the adherent serpentine, calcite, etc. should be removed by hand dressing. Hydromagnesite is unsuitable for refractory purposes. In comparing the four varieties of magnesite, importance should be attached to the coarseness of the particles, as facilitating the separation of adventitious minerals, and to the effect of iron sulphide and other impurities. Iron oxide, when present, may increase the thermal conductivity of the material and so induce sintering at a slightly lower temperature. The end-product of calcination (periclase) is the same with each variety of magnesite, but it can be produced from breunnerite at a slightly less cost than from the other varieties. The deposits of breunnerite and spathic magnesite appear to be much larger than those of compact magnesite and are likely, therefore, to be preferred by those requiring magnesite as a refractory material. Analytical

and statistical figures and details of the distribution and geological characters of the various forms of magnesite are given.—A. B. S.

*Magnesites and magnesite bricks.* W. Donald.  
Trans. Ceram. Soc., 1917-18, 17, 486-561.

Austrian magnesite (from the Veltch and neighbouring valleys), a massive crystalline spar of a yellowish-white to light yellow colour with lamellar grain and intrusions of chalybite ( $\text{FeCO}_3$ ), chlorite schists, serpentine, and various other silicates, is much less pure than Greek magnesite, but is washed and carefully hand-picked before and after calcination. The composition of the dead-burned product is very uniform averaging 85%  $\text{MgO}$ , 8%  $\text{Fe}_2\text{O}_3$ , and 3%  $\text{SiO}_2$ . The sp. gr. of the raw material is 2.63 to 3.1, that of the caustic magnesite 3.2, and of the dead-burned product 3.545. Grecian magnesite (chiefly from Euboea) when calcined averages over 90%  $\text{MgO}$ , 3%  $\text{CaO}$ , 0.7  $\text{Fe}_2\text{O}_3$ , and 4%  $\text{SiO}_2$ . The chief impurities are dolomite, quartz, and material from the overburden. The irregular distribution of the impurities makes calcination more difficult than in the case of the Austrian material. In the Canadian magnesite deposits, the distribution of the impurities is even more irregular. Calcium and silicon compounds should be removed as completely as possible as they reduce the melting point of the magnesite. Differences in the dead-burned magnesite from the different sources of magnesite are probably due chiefly to the conditions of burning and to the nature of the impurities present. A series of mixtures of iron oxide, alumina, and silica in the same proportions as those in which they occur as impurities in 13 different makes of magnesites had temperatures of incipient fusion remarkably close to 1100° C. The bulk of the magma in magnesite bricks corresponds to the average composition of the impurities, but some of the impurities form distinct compounds with the lime present. A notable difference between the impurities in bricks made of Grecian and Austrian magnesites is that in the former the magnesium silicates are much more bulky and highly viscous and do not penetrate the particles of magnesite so uniformly as in the latter. If bricks made of Grecian magnesite are fired 30 times at cone 30 the brown colour disappears, leaving a mass of pure white mineral with a micro-structure like that of Austrian bricks. On re-firing under normal commercial conditions, the brown colour reappears. Photo-micrographs show a much greater alteration in crystalline structure in Grecian bricks re-fired at 1750° C. than in those made of Austrian magnesite; hence, the latter are more durable in use. When re-fired at 1850° C., Grecian bricks show evidence of the segregation of impurity and local weakness, which would easily lead to rupture. In Austrian magnesite bricks the texture is remarkably uniform, the bricks consisting of even-sized particles not more than  $\frac{1}{2}$  in. in diameter and a magma which interpenetrates the particles much more uniformly than is the case with bricks made of Grecian magnesite. It is suggested that ferrous oxide or manganese added to the magnesite would act as a catalyst and secure the better conversion of the magnesite into large crystals as well as form definite mineral compounds of greater stability and density. Two types of magnesite brick are now made in Great Britain: one is of a finely ground material and weighs 72-76 cwt. per thousand, the other is a coarser material which weighs 82-86 cwt. per thousand and shows a greater difference in light and shade between the particles and magma. In both types a large proportion of the magnesite is in the calcined as distinct from the dead-burned state and the impurities surround rather than penetrate the particles as they do in the fully-dead-

burned Austrian bricks. The average sp. gr. of the British made bricks is only 3.435 as compared with 3.572 for the Austrian bricks, corresponding to a difference in weight of 10-15%. The chief faults of British magnesite bricks (which are mostly made of Grecian material) are spalling, especially in the roofs of electric furnaces where the bricks also tend to be deformed; inability to resist corrosion by basic slag, especially when porous; and crumbling to dust after 5 months' use in the bottoms of open-hearth and other furnaces, even when covered with a thick layer of rammed, sintered dolomite mixed with 10-20% of basic slag and tar as a binder. Austrian bricks are much better in these respects. Part of the weakness of the British bricks may be due to the ease with which they disintegrate when heated to 1200° C. under strong reducing conditions; at 1500° C. the disintegration is much less marked. When minute crystals of magnesite grow into larger ones, there is intense concentration with increased density, and therefore porosity between the various growths. If the growth could be secured before or during the burning of the bricks, the present objectionable shrinkage and spalling when the bricks are used in steel furnaces would be avoided. All magnesite bricks fall to a light powder when subjected to the action of steam. The crushing strength of magnesite bricks depends on the temperature and pressure at which they are burned and the nature of the impurities. The lower the porosity the greater the strength of the finished brick. The Austrian bricks are made under a pressure of 300 tons and British bricks are greatly improved by the use of a high pressure in moulding and a pressure of 15 lb. per sq. in. during burning. Improvements may be sought in prolonged heating of the magnesite under highly reducing conditions, and in the presence of a catalyst rather than by heating at excessively high temperatures. Selected Grecian material finely ground with a suitable proportion of ferrous carbonate could probably be dead-burned satisfactorily at 1400°-1500° C. in rotary kilns (as used in Austria), if they could withstand the necessary reducing atmosphere. In the Austrian material, the ferrous carbonate is present in a form isomorphous with the magnesite. Recent consignments from the Greek mines show that in the material which has been burned at 1750° C. in modern gas-fired shaft kilns, the requisite microstructural growth has taken place, though not quite regularly; it could probably be improved by greater care in grinding and burning. The British-made bricks of Grecian magnesite which behave most like the Austrian ones in furnace linings are those most closely corresponding in microstructure. Bricks can be made of Grecian magnesite under suitable conditions of heat and pressure, in which the formation of crystalline magnesite is complete, and they give better resistance to all temperatures up to 1750° C. and to corrosion by slag or attack by furnace atmosphere than Austrian bricks. If a pressure of 150 tons per brick is used in moulding them, the finished bricks weigh more than Austrian ones of the same dimensions, but have the same porosity (16%). This is more than sufficient to allow for thermal expansion in use without disrupting the bricks. It is considered that by the use of a suitable catalyst, bricks superior to the Austrian ones could be made commercially from Grecian or other relatively pure magnesite.—A. B. S.

*Magnesite bricks; Micro-structure of —.* A. Scott. Trans. Ceram. Soc., 1917, 17, 475-485.

COMMERCIAL calcined magnesite appears to be a very fine-grained amorphous mass of magnesite with local double refracting areas containing the im-



purities. Dead-burned magnesite contains crystals of periclase, and magnesite bricks show two well-defined types of structure. In the first type there are sharp polygonal crystals of periclase containing some isomorphous iron compound, not yet identified, which gives the crystals a well-defined cubic cleavage not possessed by pure periclase. The crystals are coloured red with disseminated iron oxide and contain inclusions of dark crystals of magnesioferrite ( $\text{MgFe}_2\text{O}_4$ ). In the second type small rounded crystals of periclase are embedded in a crystalline matrix of low melting point and variable composition, containing a calcium silicate—monticellite (m.pt.  $1460^\circ\text{C}$ .), and possibly forsterite, together with another unidentified mineral. Crystals of the first type are found in bricks having iron oxide as the main impurity; those of the second type are more common in magnesite containing appreciable quantities of lime and silica, but some bricks contain both types of crystals. In the manufacture of magnesite bricks the first type is the more valuable, as the silicate matrix in the second type is too fusible an ingredient and should, if possible, be eliminated. The conversion of the amorphous magnesia into large-grain crystals of periclase is facilitated by silicious impurities, in presence of which the magnesia is dissolved, and re-precipitated as periclase. This increase in grain size results in an increase in the sp. gr. of the material, the amorphous magnesia corresponding to Mellor's  $\alpha$ -form and the periclase to his  $\beta$ -form (this J., 1917, 551). Owing to the matrix facilitating crystallisation, it is difficult to discriminate the fragments of dead-burned magnesia in bricks which have been in use for a long time, as sections of them show a uniform structure.

—A. B. S.

*Zirconia; its occurrence and application.* H. C. Meyer. *Ceram. Soc., Refractories Sect.*, Oct. 18, 1918. [Advance proof.]

ZIRCONIA occurs chiefly in the Caldas region, Brazil, as a mechanical mixture of brazilite, zircon, and an unnamed zirconium silicate readily soluble in hydrofluoric acid and containing about 75%  $\text{ZrO}_2$ . Small briquettes, measuring  $36 \times 20 \times 5$  mm., were made and burned at  $1427^\circ\text{C}$ . A briquette made of pure zirconia (99%  $\text{ZrO}_2$ ) was quite soft after drying and when burned could be scratched with the finger nail. A briquette of water-ground ore containing about 84%  $\text{ZrO}_2$  was extremely dense and hard. A briquette of partially purified material, containing about 95%  $\text{ZrO}_2$ , was also very dense and hard. Another briquette made from 60 grms. of ore (80-mesh) and 40 grms. of water-ground ore had a linear shrinkage of only 3%, a rather coarse texture, and considerable tensile strength. Other briquettes made by ramming the material in the form of a "stiff mud" into a steel mould, drying, and then burning at  $1600^\circ\text{C}$ . gave the following results: Zirconia ore bonded with magnesium chloride solution of sp. gr. 1.23: showed no fusion, slight shrinkage, hardness greater than glass, very dense texture, great toughness; it was corroded by ferrous oxide. Zirconia ore bonded with water vapour gave similar results. A mixture of equal parts of zirconia ore and dead-burned magnesia bonded with magnesium chloride solution of sp. gr. 1.23 fused to a spongy mass of dark colour. A mixture of zirconia ore 10%, dead-burned magnesia 88%, magnesium chloride (sp. gr. 1.23) 2%, gave a light brown, hard and dense product, with sharp corners, no signs of fusion; it was not corroded by ferrous oxide. Zirconia ore bonded with water-glass of sp. gr. 1.26 gave an extremely hard, dense product, not fused, and slightly cracked. Briquettes made of zirconia ore (80-mesh), calcined magnesia, and water-ground zirconia ore had a

rather loose texture, but were resistant to abrasion and of fair tensile strength; when white hot could be dropped into water without damage. Full-sized bricks made of zirconia ore with 5% of fire-clay, burned in a silica brick kiln at  $1640^\circ\text{C}$ ., softened at a temperature well below the fusion point of the ore. After being in contact with carbon at  $1800^\circ\text{C}$ . for  $1\frac{1}{2}$  hours, the surface was less affected than magnesia bricks, but more so than carborundum. A satisfactory commercial process for the manufacture of zirconia bricks consists in grinding the crushed ore to about 60-mesh in a dry pan. All particles finer than 100-mesh are removed and the coarser material is mixed with an equal weight of the fine water-ground ore, made into a stiff paste, and moulded in the same manner as silica bricks. The bricks are dried very slowly and are then burned. The percentage of loss due to cracked and warped bricks is rather high, but the damaged pieces may be reground.

Zirconia may also be used in combination with yttrium in Nernst glowers, as a substitute for lime in "magic" lanterns and in pencils for Blériot headlights, in the production of an acid-proof enamel, as an addition to fused silica ware to prevent devitrification, and as a substitute for bismuth salts in X-ray photography.—A. B. S.

*Refractories; Electric furnace treatment of* —. R. S. Hutton. *Trans. Ceram. Soc.*, 1917-18, 17, 233-243.

ELECTRIC furnaces have only been used to a relatively small extent in the manufacture of refractory materials. In the preparation of raw material, the chief advantage which electric furnace treatment can offer is in the facility with which fully shrunk material may be obtained and the ease with which some of it may be purified and so rendered more refractory. Where actual fusion of the refractory material is required a simple arc furnace will generally prove the most useful, but a resistance furnace is more efficient. In one series of trials about half a ton of highly shrunk magnesia was produced by completely embedding a vertical carbon rod 50 cm. long and 2 cm. diameter in the material to be heated, and passing a current of 400 amps. at 30 volts through the rod for 2 hours at a time. The energy consumption was only 1.17 kilowatt-hours per kilo. of shrunk magnesia. Only small articles can, at present, be burned in an electric furnace, but carbon plates coated with carborundum and held in graphite water-cooled terminals may form the basis of a larger technical furnace for use at  $2000^\circ\text{C}$ .—A. B. S.

*Refractory materials; Relative volatilities of* —. W. R. Mott. *Amer. Electrochem. Soc.*, Sep. 30—Oct. 2, 1918. [Advance copy.] 34 pages.

THE apparatus used was a Helios photo-engraving lamp with a metal case having a pin-hole 12 in. from the arc, allowing the image of the arc to be projected on a suitable screen in a dark room, thus magnifying it 20 diameters. The arc required 25 amps. at 50 volts. The carbons were ordinary 12 in.  $\times \frac{1}{2}$  in. high-grade solid enclosed arc carbons with an ash of 0.05-0.10%, the lower positive one having a cup-shaped hole ( $\frac{3}{8} \times \frac{3}{8}$  in.) at its end. About 10 mgrms. of the refractory material was placed in the cup and heated for 3 minutes by the electric arc so that the vapour from the volatilised material travelled from the positive to the negative electrode. The deposits on the latter were arranged accurately in a position and in an order depending on their relative volatility. The results were compared with those from tungsten (b.pt.  $6000^\circ\text{C}$ .), and iron saturated with carbon (b.pt.  $3500^\circ\text{C}$ .). The

temperature of the positive crater of a carbon arc was taken as 3700° C.

Of ten methods tried, the most accurate was one of fractional distillation, though mixtures of lead and bismuth, tin and copper, and nickel and iron volatilised at practically the same temperature. This method is less satisfactory with metals in which carbon is soluble. With oxides, the experiments usually resolve themselves into a study of the order of reduction by carbon. On heating mixtures of various refractory materials with iron, the change from the iron arc to that of the refractory residue can be readily observed. Tungsten is left as a final residue when a mixture of it with any other material is distilled. Tungsten carbide decomposes into its constituents and the carbon volatilises. Next to tungsten tantalum is least volatile. Zirconium carbide is the least volatile carbide. The following oxides volatilise in the order given:  $K_2O$ ,  $Na_2O$ ,  $Li_2O$ ,  $V_2O_5$ ,  $B_2O_3$ ,  $BaO$ ,  $SrO$ ,  $MnO$ ,  $FeO$ ,  $CoO$ ,  $NiO$ ,  $Cr_2O_3$ ,  $TiO_2$ ,  $V_2O_5$ ,  $SiO_2$ ,  $CaO$ ,  $MgO$ ,  $Al_2O_3$ ,  $Ti_2O_3$ ,  $V_2O_3$ ,  $Er_2O_3$ ,  $CeO_2$ ,  $Nd_2O_3$ ,  $La_2O_3$ ,  $BeO$ ,  $Yt_2O_3$ ,  $ZrO_2$ , and  $ThO_2$  last. The boiling points of the following refractory oxides were estimated from the distance in the arc image from the crater to the refractory material, this being roughly proportional to the volatility:  $BaO$  2000° C.,  $TiO_2$  below 3000° C.,  $Cr_2O_3$  3000° C.,  $CaO$  3400° C.,  $SiO_2$  3500° C.,  $MgO$  3600° C.,  $Al_2O_3$  3800° C., and  $BeO$  3900° C. Other boiling points, determined by fractional distillation of mixtures of the material with one of known boiling point, are: iron saturated with carbon 3500° C., palladium 3600° C., carbon 3700° C., chromium carbide 3800° C., vanadium carbide 3900° C., rhodium 4000° C., platinum 4950° C., uranium carbide 4100° C., ruthenium 4150° C., lanthanum oxide 4200° C., titanium carbide 4300° C., yttrium oxide 4300° C., columbium (niobium) carbide 4300° C., zirconium oxide 4300° C., thorium oxide 4400° C., iridium 4400° C., osmium 4450° C., molybdenum carbide 4500° C., yttrium carbide 4600° C., thorium carbide 5000° C., zirconium carbide 5100° C., tantalum (carbide?) 5500° C., and tungsten (carbide) 6000° C. Carbon sublimes at 3700° C. and boron nitride at near 3000° C. The boiling point in absolute degrees divided by the melting point gives a ratio (average 1.8) which is similar for similar elements in the periodic table and characteristic of all elements except the alkali metals, the heaviest easily reduced elements, and the non-metals. A bibliography on the subject is included.—A. B. S.

*Firebrick from the crown of an electric steel-melting furnace.* W. J. Rees. Trans. Ceram. Soc., 1917-18, 17, 248-249.

A FIREBRICK from the crown of a  $\frac{1}{2}$ -ton Greaves-Etchells electric steel-furnace which had lasted for 145 charges and had proved much more durable than silica brick, was made of a refractory bauxite clay from the Millstone Grit lavas of Ayrshire. The notable features of the brick are its high refractoriness (cone 37) and the proportions of silica (51.86%), alumina (38.7%), titanic oxide (6.68%), and iron oxide (1.56%) which it contains.—A. B. S.

*[Plaster pottery] moulds; Deterioration of — during storage.* J. W. Mellor. Trans. Ceram. Soc., 1917-18, 17, 331-335.

PLASTER moulds—especially those used in casting clay containing soluble salts—deteriorate and become "rotten" if stored in a damp state owing to the formation of crystals of sodium sulphate. To prevent this deterioration the moulds should be dried as rapidly and thoroughly as possible after

being used and should be stored in a dry place where no moisture can condense on them.—A. B. S.

*Quaternary [pottery] mixtures; A method for the graphic determination of —.* H. S. Newman. Trans. Ceram. Soc., 1917-18, 17, 336-339.

THE composition of a quaternary mixture may be graphically represented by the perpendicular distance of a point from the four sides of a square, and the author suggests that the composition of mixtures of ball clay, stone, china clay, and flint, as used by potters, may be represented by such a square diagram. No mixtures having more than 50% of any one constituent can be represented by such a diagram.—A. B. S.

*Fritted glazes; Investigation of the solubility of lead in —.* P. Bartel. Sprechsal, 1918, 51, 25, 31, 35, 39, 43. Z. angew. Chem., 1918, 31, Ref., 290.

THE author subjected frits with a composition ranging from  $PbO, 1.5SiO_2$  to  $PbO, 4SiO_2$  which had been ground to pass completely through a No. 175 sieve, to the action of 4% acetic acid under the conditions of the German official test for determining the solubility of lead in glazed ware (see this J., 1910, 354). It was found that the resistance of the glaze to the acid was not proportional to the silica content of the glaze, but that it increased with the silica content up to 2.5 mols.  $SiO_2$  per mol.  $PbO$  and then diminished with more silicious glazes. On replacing a part of the lead oxide by soda, potash, lime, baryta, magnesia, or zinc oxide, less lead was dissolved by acetic acid. On the addition of alumina to the material used for the frits, the solubility of the lead was still further diminished and was reduced to *nil* in several frits containing either  $Na_2O$ ,  $K_2O$ ,  $CaO$ ,  $BaO$ ,  $MgO$ , or  $ZnO$ . Of the 817 frits examined, 430 yielded less than 2% of lead to the acetic acid solution; of these 11 were simple lead silicates containing alumina, 79 contained soda, 64 potash, 74 lime, 50 baryta, 85 magnesia, and 67 zinc oxide. Of these frits 14 contained 1.5 mols.  $SiO_2$ ; 24, 1.75  $SiO_2$ ; 37, 2.0  $SiO_2$ ; 47, 2.25  $SiO_2$ ; 48, 2.5  $SiO_2$ ; 49, 2.75  $SiO_2$ ; 52, 3  $SiO_2$ ; 39, 3.25  $SiO_2$ ; 37, 3.5  $SiO_2$ ; 42, 3.75  $SiO_2$ ; 41, 4  $SiO_2$ . Most of the frits which contained 1.5  $SiO_2$  were clear, but those containing 2.75 or more  $SiO_2$  were seldom so. The addition of boric acid to reduce the viscosity and the melting point of the frit was unsatisfactory.—A. B. S.

*Hydraulic separation.* Draper. See I.

*Refractories in steel production.* Reynolds. See X.

*Refractories in zinc industry.* Audley. See X.

#### PATENTS.

*Glass chemical ware [measuring cylinders, etc.].* A. Mond, London. Eng. Pat. 119,297, 3.10.17. (Appl. 14,293/17.)

GRADUATED measuring cylinders and the like are made of a glass tube of uniform bore fitted with a detachable plug or stopper which serves as a foot and may be removed to facilitate cleaning the vessels. The cost of grinding in the foot is compensated by the reduced cost of graduation.—A. B. S.

*Glass cylinders; Device for use in connection with the drawing of —.* F. M. Francart, Springdale, Pa. U.S. Pat. 1,272,652, 16.7.18. Appl., 17.4.16.

THE tube through which the glass is drawn is provided with a skirt at its lower end and an inner annular recess or trap above the skirt, so that



when the tube is depressed into the glass around the drawing area, the surface impurities flow towards and are trapped in the recess.—A. B. S.

*Glass; Manufacture of* —. L. T. Sherwood, Connellsville, Pa. U.S. Pat. 1,277,493, 3.9.18. Appl., 18.11.16.

In the manufacture of glass from a batch containing nitre-cake, carbon is added in proportion amounting to at least 11% of the nitre-cake.

—A. B. S.

*Silica bricks; Manufacture of* —. Soc. Anon. des Prod. Refractaires de l'Ouest, Paris. Eng. Pat. 118,116, 6.8.18. (Appl. 12,788/18.) Int. Conv., 6.8.17.

DISINTEGRATED or calcined quartz or quartzite containing 95% of silica is crushed, and part of it is then ground to flour, re-mixed with the crushed material in suitable proportions, and the mixture made into silica bricks in the customary manner.

—A. B. S.

*Refractory bricks, blocks, tuyères and the like employed in connection with furnaces for smelting metals, etc.; Manufacture of* —. H. E. Mason and A. Couper, Horwich. Eng. Pat. 119,101, 27.9.17. (Appl. 13,906/17.)

REFRACTORY blocks, etc. are made by mixing finely ground calcined magnesite with water and 6% of a fluxing agent such as basic slag, iron oxide, iron ore, metallic iron, or iron slag (either singly or mixed in any desired proportions) and 1% of a salt or salts of boric acid and/or perboric acid. Alternatively, the goods so made may be reduced to powder and re-made after the addition of water or other liquid, or raw magnesite may be used instead of the calcined material and the burned blocks may be ground and re-made. The fluxing agents and salts may be mixed, made into blocks and calcined; the product may then be ground to powder, mixed with calcined magnesite, and made into blocks, etc.—A. B. S.

*Tunnel furnaces.* E. E. Brand, Halsingborg, Sweden. Eng. Pat. 119,366, 2.1.18. (Appl. 14/18.)

In a tunnel kiln having three compartments for preheating, heating, and cooling respectively, apertures are formed throughout the entire length of the roof and/or walls of the cooling chamber, through which air is forced at right angles to the customary current of air passing through the tunnel, thereby cooling the goods more effectively.—A. B. S.

*Brick-kiln.* E. H. A. Bolibuck, Los Angeles, Cal. U.S. Pat. 1,277,539, 3.9.18. Appl., 28.9.16.

A KILN is provided with stationary partitions fitted with doors so as to form a number of compartments which communicate in series and also, when the doors are opened, in parallel. Independent furnaces or burners on the top of the kiln communicate with the different compartments, and damper-controlled flues in the lower part of each compartment communicate with the main flues in the walls of the kiln.—A. B. S.

*Refractory article and method of making same.* S. C. Linbarger, Assignor to The Carborundum Co., Niagara Falls, N.Y. U.S. Pat. 1,277,227, 27.8.18. Appl., 22.3.18.

CRYSTALLINE silicon carbide is added to refractory mixtures of graphitic and clay in order to lessen or prevent the formation of parting planes in the moulded mass.—A. B. S.

*Enamelling metal articles and furnace therefor.* P. Dupont, Paris. Eng. Pat. 114,618, 14.2.18. (Appl. 2667/18.) Int. Conv., 30.3.17.

A BATH or other article to be enamelled is placed in a furnace of similar shape, composed of very porous refractory material, covered externally with a metal casing, which is separated from it by a very narrow space. The furnace is heated by a mixture of gas and air preheated by the waste gases and injected into the space between the refractory material and the casing, and ignited on the inner surface of the refractory material by means of a series of independent burners. The products of combustion circulate between the refractory material and the article to be heated, and pass through two discharge passages, one in the base and the other in the cover of the furnace, to a supplementary furnace which is used for preheating the articles to be enamelled. The cover of the furnace is also of porous refractory material, but the angles are provided with pieces of non-porous refractory material. The refractory material is rapidly raised to incandescence and when the article to be enamelled has reached the requisite temperature, the cover is removed and the surface of the article is covered with powdered enamel. In an alternative arrangement, the refractory material may be much thinner and non-porous; it is then preferably strengthened by a metal casing and is heated by a series of burners, the flames from which burn in the space between this casing and an outer casing.—A. B. S.

## IX.—BUILDING MATERIALS.

*Cement; Elasticity of neat* —. L. Jouane. Comptes rend., 1918, 167, 591—593.

To investigate the elasticity of mortars and concretes, prisms of the material are placed horizontally with one end fixed solidly in a wall or vice, the other being held in a clip suspended from a hook attached to the beam of a balance and carrying beneath it a funnel. The movements of the free end of the test-piece are shown by a mirror attached to the knife-edge of the balance. Sand flows into the funnel at a definite rate until a definite load is applied to the free end of the test-piece, after which the supply is cut off by an electrically controlled device which also opens a valve in the bottom of the funnel so that the sand flows out at the same rate as it had entered. In one experiment, a test-piece made of a mixture of 1000 grms. of cement and 350 grms. of water, measuring when air-dried, 38.6 cm. × 2.96 cm. × 1.43 cm. and weighing 286.07 grms., was subjected to loads gradually increasing to 500 grms. The deflections showed that with loads too small to produce permanent deformation the material has

a clearly marked coefficient of elasticity  $E = \frac{Pl^3}{a^3bf}$

where  $P$  = load in kilos.,  $f$  = corresponding deformation in cm.,  $l$  = distance between the points of support at each end of the test-piece,  $a$  = breadth, and  $b$  = thickness of the test-piece. The elasticity was also determined by vibrating the test-piece and recording the oscillations (45.5 per sec.) on a smoked sheet. In this case

$$E = \frac{48d\pi^2 N^2 l^4}{a^2 \times 9.81 \times 10^5 \times \sqrt{1.87}}$$

in which  $d$  is the density in grms. per c.c.,  $N$  = number of oscillations per sec., and  $l$  = distance between the fixed and free ends of the test-piece. The test-piece examined had a coefficient of elasticity of  $1.080 \times 10^5$  kilos. per sq. cm. by the first

method and  $1.071 \times 10^3$  kilos. per sq. cm. by the second one. Equally concordant results were obtained with other test-pieces.—A. B. S.

#### PATENTS.

*Timber and other materials; Process and apparatus for preserving and drying* —. G. B. and G. W. McMullen, Picton, Ont., Canada. U.S. Pat. 1,277,619, 3.9.18. Appl., 1.9.16.

The material to be dried is subjected in a closed chamber to a saturated atmosphere which is raised rapidly to the maximum safe temperature. It is then dried by causing a rapid current of air at or below  $160^\circ \text{F}$ . ( $71^\circ \text{C}$ .) to flow over it, the humidity of the air being regulated so that the moisture evaporates from the surface of the material at the same rate as it passes by capillary action from the interior to the surface. A portion of the moisture-laden air is allowed to escape and is replaced by dry air, the humidity of the air being gradually decreased.—A. B. S.

*Cement; X-ray-protective* —. P. Poetschke, Milford, Del. U.S. Pat. 1,278,010, 3.9.18. Appl., 8.10.18.

A CEMENT for obstructing X-rays is composed of lead carbonate 2 parts, cement-forming powder 1 part, and a liquid added to produce a plastic mass. —A. B. S.

*Fireproofing composition*. U.S. Pat. 1,276,742. See I.

*Slaking lime*. U.S. Pat. 1,277,855. See VII.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Refractories used in steel production; Essential properties of* —. A. Reynolds. Trans. Ceram. Soc., 1917–18, 17, 385–458.

THE author maintains that molten steel can be partially oxidised by silica and silicious slags, the resulting ferrous oxide causing segregation. In the production of steel the removal of each acid and base from the ore charge, including fluxes, should be a distinct metallurgical process, and the different stages should be conducted in separate furnaces, suited for one and unsuitable for another step. The use of silicious materials and clays in the production of casting nozzles and ladles should be avoided. The production and retention of perfect steel is impossible if it comes into contact with any material which is neither neutral nor basic, suitable neutral or basic materials must be found to take the place of the acid refractories now used. A further objection to acid linings for ladles, etc., in addition to their oxidising action, is that they become covered with manganese monosilicate which is rapidly "roasted" as the ladle is emptied and the manganese and iron oxides produced will spoil any steel which is next poured into the ladle. The reduction of the alkaline oxides in the refractory lining of a steel furnace at  $1500^\circ \text{C}$ . yields metals which are powerful catalytic agents and may bring about many unsuspected reactions, including the conversion of magnesia into an agent which oxidises steel and the possible nitrogenation of the steel. The presence of these oxides in refractories should, therefore, be avoided. Acid hearths have the advantage of providing for the rapid formation of silico-manganese ( $\text{MnSi}$ ), which is one of the most powerful deoxidants, and thus produce better steels than basic furnaces. Basic refractories must be free from alkalis and from occluded oxygen. Alkalis in the form of silicates are undesirable, but less harmful than the alkaline

oxides. Acid silicates or silica should be absolutely excluded. Lime or dolomite is only admissible under such conditions of high temperature that it remains caustic. New refractories such as zirconia are useless unless their heats of formation are extremely high and their protoxides "unroastable." Improved magnesite bricks and neutral or basic aluminium and magnesium silicates are the most promising. They must be made from mixtures and treated so that at least the working face is free from acid portions. Thus, a mixture of fireclay and alumina would be very slightly less harmful than fireclay alone. The maximum temperature which such materials need to resist is somewhat below  $1600^\circ \text{C}$ . In the manufacture of crucible steel a three-jar equilibrium of slag-metal, slag-crucible, and metal-crucible may be obtained, but it is impossible to obtain the triple equilibrium slag-metal-crucible. The "cutting" of the crucible at the junction of metal and slag is therefore inevitable and its disastrous effects can only be avoided by using successively smaller charges of material each time the crucible is used. In newly built or repaired acid open hearths, the steel made from the first charge is of inferior quality, on account of the silicic acid in the hearth oxidising the iron in the charge. A series of new crucibles in which pieces of Swedish Bessemer ingot were melted showed clearly that the crucibles acted like an acid open hearth in catalytically oxidising some of the iron and absorbed a considerable proportion of manganese from the steel. The slag produced absorbed silica from the crucibles. In the proposed final process of deoxidation or "finishing" of the steel, by the addition of silico-manganese to the previously refined metal, the lining of the furnace should be nearly neutral or basic. Both the slag and lining must be quite inactive to iron and must therefore be as free as possible from iron or manganese silicates. A lining of aluminium monosilicate with a trace of alumina or of magnesium monosilicate would be refractory if rendered sufficiently basic, and it should be possible to prepare the latter from pure quartz and a magnesia which had been freed from iron, manganese, and alkalis by a wet process. Bricks, stoppers, and nozzles for such a furnace may be made of a mixture of fireclay and bauxite in the proportions required to form sillimanite and their surface fused in a special furnace (see Eng. Pat. 111,355; this J., 1918, 31A). Basic nozzles and stoppers are not satisfactory, as they do not remain caustic, readily decrepitate, and so spoil the steel, as the decrepitated matter is worse than slag in an ingot. Some refractories are unexpectedly volatile under reducing conditions at temperatures several hundred degrees below their normal softening point. Thus, the gas flues of two large producers were choked with a light feathery deposit containing 63% of silica. Several small crucibles made of kaolin and alumina in proportions to form aluminium monosilicate and others made of magnesite were buried in a loose carbon resistance furnace and charged with steel. They were heated slowly and all with the exception of one of the magnesite crucibles fumed away before the steel was molten (i.e., below  $1450^\circ \text{C}$ ). The old crucible withstood three treatments before it failed and appeared to be free from some unknown catalyst which was present in the others. The iron and manganese volatilised in electric arc furnaces form protoxides which attack the silicious roof of the furnace. The corresponding vapours in gas-fired furnaces form higher oxides which "season" and actually improve the roofs.—A. B. S.

*Steel; Heterogeneity of* —. H. Le Chatelier and B. Bogitch. Comptes rend., 1918, 167, 472–477.

THE polished surface of commercial steels, when attacked by suitable etching reagents, shows a



heterogeneous structure visible to the naked eye. This macrographic heterogeneity is unaltered by heat treatment, and Stead (this J., 1915, 616) attributed it chiefly to the unequal distribution of phosphorus. The authors' experiments lead to the view that the heterogeneity is entirely due to oxygen in solid solution in the steel, and that neither phosphorus nor sulphur has any direct influence. Electrolytic iron, with or without the addition of iron phosphide or sulphur, when melted in an atmosphere of hydrogen was homogeneous on etching, but the same metal, if melted with access of air, or with addition of iron oxide, showed a heterogeneous structure. If heated below its melting point the heterogeneity only occurred in the surface layers. In cast steels as the metal solidifying last will be the richest in oxygen, heterogeneity is particularly marked in segregated areas. The difference between the electrical resistance of steel as calculated from the chemical composition (which excludes oxygen), and as actually determined, is considered to be due to the influence of oxygen in the steel.—C. A. K.

*Hardened steel; Spontaneous generation of heat in recently* — C. F. Brush, R. A. Hadfield, and S. A. Main. *Proc. Roy. Soc.*, 1918, A, 95, 120—138.

In a previous paper (this J., 1917, 505) it was shown that a nickel-chrome steel hardened from 777°C. or above generated heat at a gradually decreasing rate for many hours. When, however, the metal was quenched from a temperature below the Ar1 point (from 532° to 667°C.), an absorption of heat resulted. Similar experiments carried out on a plain carbon steel showed a generation of heat after quenching from above the critical range but no generation or absorption of heat when the temperature of quenching was insufficient to result in the steel becoming hardened. A white iron showed a generation of heat after quenching from 757°C., but no trace of heat change was observed in the case of a pure Swedish charcoal iron. By replotting the results it is shown that the evolution or absorption of heat is greatest immediately after quenching and thereafter continuously decreases, so that: rate of evolution  $\times$  time after quenching = a constant. It is shown that the spontaneous contraction which occurs in hardened steel, given by the equation:  $C_t = 159 \log_{10} T + 134$ , where  $C_t$  is the linear contraction in millionths of the original length after  $T$  hours, is insufficient to produce all the heat evolved and it is suggested that a slow change goes on in the steel for a considerable time after quenching, and that both the evolution of heat and contraction are results of this change.

—F. C. Th.

*Ferro-alloys [ferro-uranium]; Manufacture of* — in the electric furnace. R. M. Keeney. *Amer. Inst. Min. Eng.*, Sep., 1918. *Chem. and Met. Eng.*, 1918, 19, 281—282.

ATTEMPTS to prepare ferro-uranium (20% U) direct from sodium uranate (the by-product of radium extraction), by adding to molten steel a mixture of the uranate with coke and fluorspar, resulted in the production of the double carbide,  $\text{Fe}_2\text{C}_2\text{U}_2\text{C}_2$ ; and the latter could not be decarburised by means of silica or iron oxide, since the uranium was oxidised by these materials and passed into the slag. By heating sodium uranate to a high temperature with carbon in a stationary Siemens electric furnace having magnesite walls and a carbon bottom, a spongy metal containing over 90% U and 35–4% C was obtained. The recovery of uranium by this means was 50–60% in a single operation, and over 85% when the unreduced residue (uranium oxide and carbon) was re-treated with another charge; the power consumption averaged

about 8 kilowatt-hours per pound of metal. Attempts to prepare ferro-uranium by adding the spongy metal to molten steel were unsuccessful, owing to the oxidation of the uranium during the long time required to effect complete melting. By heating a mixture of uranium oxide, coke, and fluorspar with steel turnings in the Siemens furnace, ferro-uranium containing 50% U and less than 5% C was produced; and this alloy was successfully employed in the production of low-uranium steels (2–4% U).—W. E. F. P.

*Copper; Relation of sulphur to overpoling of* — S. Skowronski. *Amer. Inst. Min. Eng.*, Sep., 1918. *Chem. and Met. Eng.*, 1918, 19, 279.

FROM experiments made with pure cathode and high-sulphur copper, it is concluded that the "pitch" of copper is affected by both oxygen and sulphur. Cuprous oxide counteracts the effect of sulphur by increasing the solubility of sulphur in copper, or by inducing a physical condition of equilibrium between the sulphur, tending to "overpole," and the cuprous oxide tending to "underpole" the metal. A comparatively large amount of cuprous oxide is required to counteract the effect of a trace of sulphur.—W. E. F. P.

*Refractories in the zinc industry.* J. A. Audley. *Ceram. Soc., Refractories Sect.*, Oct. 18, 1918. [Advance proof.]

THE chief requirements in retorts used in the zinc industry are refractoriness, resistance to dust and slag, mechanical strength and tenacity, resistance to sudden changes in temperature, low permeability to zinc vapours, and durability. Zinc is absorbed by the retort, especially by a new retort, with formation of blue zinc aluminate (spinel), the silica from the clay and also quartz grains present in the clay being partially converted into tridymite. Fraenkel (this J., 1909, 1254) considers that some zinc is lost in the form of a volatile  $\text{ZnSSi}$  which is not condensed in the receiver of the retort. Mühlhäuser found that the total loss of zinc in the case of new retorts diminished day by day in the following amounts: 32.35, 29.89, 20.97, 16.07, 16.97, 13.41, and 8.30% respectively. Iron compounds may be reduced to ferrous oxide or to metallic iron which may then form a slag having a highly corrosive action on the retorts. Manganese and lead silicates also corrode the retorts. Calcium and magnesium silicates alone are not very harmful, but in combination with ferrous and manganese silicates are highly corrosive. For this reason, the custom of adding lime to the charge has been discontinued. The retorts are usually made of a mixture of fire-clay and grog; they have a low thermal conductivity, will not withstand sudden changes in temperature, are readily attacked by materials in the charge, and are often sufficiently porous to allow zinc vapour to escape. A highly aluminous material, low in silica, would be preferable. In many Belgian works and in Spain the retorts are highly silicious, thinner, and of greater thermal conductivity, but they are less resistant to the corrosive action of the charge. In Belgium and Westphalia the retorts are sometimes glazed externally to render them impervious to zinc vapour. The prevailing temperature in the furnace when new retorts replace old ones is 1200°–1250°C., and as new retorts usually leave the furnace in which they are burned at 750°–780°C., they undergo a sudden rise of about 600°C. when placed in position; this tends to cause cracks, which later become healed by the action of dust from the furnace, though the material on either side of a crack is usually highly corroded. After being in the furnace for 24 hours, the exterior of the retorts is usually covered with a vitreous layer formed by

the action of dust on the retort, and small cavities (originating from the action of heat on iron oxide and other impurities) are produced. The temperature inside the retort, during the 24 hours after it has been charged, rises from 781° to 1188° C., being below 1050° C. for only about 7 hours. There is a difference of 286° C., gradually diminishing to 88° C., between the inside and the outside of the retort during distillation. After being in use for several days, the retort becomes very tough and elastic, but as the temperature falls, the strength decreases and the retort usually cracks during cooling. The lower part of a retort tends to have a higher specific gravity than the upper part (2.93:2.48), possibly due to the infiltration of materials capable of forming fusible silicates. The mouth of a retort is less heated than the remainder, the cracks thus formed being usually filled with sublimed zinc oxide. Micrographic examinations of used retorts show that the alumina in the clay is gradually converted into a zinc aluminate (spinel) containing about 1% of ferrous oxide, with a sp. gr. of 4.45–4.52, not attacked by any acid except boiling sulphuric acid. Quartz granules and some fragments of grog are found in the interior of the mass, together with about 50% tridymite and some willemite (zinc silicate). These changes do not reduce the refractoriness of the retort material. Incrustations on the retort usually consist of zinc oxide, with or without ferrous silicate, zinc plagioclase, and other silicates. Cavities in the retort material are usually filled with glassy silicates due to the action of portions of the charge on the retort and on each other. Modern improvements in the production of the retorts include the use of hydraulic shaping presses to increase the density of the material and so reduce the loss of zinc by absorption and permeability. Attempts to reduce the vapour pressure in the retorts have not been successful. The average life of hydraulically pressed retorts is about 40 days, and of hand-made retorts 25 days. Horizontal retorts are generally used, but vertical ones might shorten the time required for distillation, particularly if the charge were introduced in the form of briquettes. The use of magnesia for retorts and muffles has been suggested by Steger, on account of its greater thermal conductivity, greater resistance to corrosion, higher refractoriness, and impermeability to zinc vapour. The application of protective coatings of zirconia or fused alumina and the admixture of carborundum with the retort material are worth further attention, and the manufacture of retorts by casting may reduce their cost. For the median wall of the furnace, Mühlhäuser found red-brown bricks of St. Louis clay, which in the burned state contained 56.03% silica, 39.26% alumina, about 3% ferric oxide, and only small amounts of other metallic oxides, were suitable. He concluded that bricks burned at 100° C. higher than the maximum temperature of the portion of the furnace in which they are used will meet all demands.—A. B. S.

*Monel metal.* J. Arnott. *Engineering*, 1918, 106, 451.

The constituent metals of Monel metal are present in the proportions occurring in the original ore mined at Sudbury, Canada. Analyses of the rolled bar give approximately 66% Ni, 30% Cu, 3.5% Mn + Fe, 0.13% Si, with small amounts of phosphorus and sulphur; and metal for casting contains 1 to 1.5% Si. The structure of Monel metal is that of a solid solution, the rolled bar showing sharply defined crystal grains, usually twined. Mechanical tests on the rolled rod show a tensile strength of 39.2 tons; elongation on 2 in., 38.5%; reduction in area, 66.9%; Brinell hardness number, 174. When annealed at 800° C. the tensile test does not fall below 34 tons per sq. in., with a Brinell number of

143, and the strength is not dependent on the work applied. Monel metal possesses considerable strength at high temperatures; at 500° C., a temperature at which naval and manganese brass have no mechanical strength, the tensile strength is nearly 29 tons per sq. in. Casting of the metal is rendered difficult by the high melting point (about 1360° C.) and the excessive shrinkage.

—C. A. K.

*Hardness [of metals]: Value of the indentation method in the determination of —.* R. G. C. Bateson. *Inst. Mech. Eng.*, Oct. 18, 1918. [Advance copy.] 21 pages.

BENEDICKS' suggestion that the influence of the diameter of the ball in the Brinell test is eliminated by multiplying the value obtained by the fifth root of the radius of the ball is discredited. Brinell hardness numbers calculated from the depth of the impression are higher than those calculated from the diameter. Load-depth diagrams consist of an initial curve followed by a straight line, and the hardness number calculated from the slope of this line is independent of the load. If  $P$  and  $P_1$  are the two pressures selected and  $d$  and  $d_1$  the corresponding diameters of

impression, hardness no. =  $\frac{16P.D^{n-2}}{\pi(2d)^n}$  where  $n = \frac{\log P_1 - \log P}{\log d_1 - \log d}$ . Further for a given angle of

impression, preferably chosen such that the diameter of the impression is equal to half the diameter of the ball, the hardness No. =  $\frac{4P}{\pi d^2}$ , which is independent of the diameter of the ball. A measure of hardness with respect to dynamic stresses which is independent of the form of the indenting tool is given by

Energy of blow in kilogram-mètres

Vol. of indentation in c.c.

—F. C. Th.

*Ludvik hardness test.* W. C. Unwin. *Inst. Mech. Eng.*, Oct. 18, 1918. [Advance copy.] 8 pages.

Errors in indentation tests, such as the Brinell test, in which the indentations produced under different loads are not geometrically similar are obviated in the Ludvik test by the use of a right-angled cone instead of a ball. The hardness figures are then to a great extent independent of the load and also of the sharpness of the point of the cone, a truncated tool answering equally well. It is suggested that in the Brinell test the hardness would be better defined as  $\frac{P}{\pi a^2}$ , where  $a$  is the radius of the impression instead of using the spherical area, and similarly in the Ludvik test. Under these conditions the Brinell and Ludvik tests would yield identical results.—F. C. Th.

*Resistance of metals to penetration when tested by impact, and a new hardness scale in energy units.* C. A. Edwards and F. W. Willis. *Proc. Inst. Mech. Eng.*, May, 1918, 325–367. (See also this J., 1918, 528 A.)

The following method is suggested for the determination of the hardness of metals at elevated temperatures up to 900° C. A 10 mm. Brinell ball rigidly attached to a free moving cross-head of known and variable weight drops a known distance on to the sample rigidly fixed to a heavy base. If  $d$  is the diameter in mm. of the indent produced,  $E$  the striking energy in inch-lb., and  $C$  a constant for each metal,  $d = CE^{0.25}$ . For a striking energy of 63 inch-lb. the ordinary Brinell hardness no. =  $\frac{7455}{d^2}$ , where  $d$  is the diameter of the indentation produced by the impact. The impact method of



measuring hardness eliminates the time factor, which is a source of uncertainty in the Brinell test as ordinarily carried out. It is considered that the estimation of hardness by the rebound of a hammer falling from a fixed height is unreliable, since different amounts of penetration are obtained in all tests.—F. C. Th.

*Metals and alloys; Internal strains developed in — by rapid cooling.* Portevin. *Comptes rend.*, 1918, 167, 531—533.

In the examination of the state of elastic equilibrium existing in metals after quenching, the method used by Heyn and Bauer (*Int. Zeits. Metall.*, 1911, 16—48; cf. this J., 1914, 596) was employed, successive layers of the metal being removed and the variation in length of the remaining piece being accurately measured. Solid and hollow cylinders of copper, nickel, brass, and steels were examined. The value of the stresses produced by quenching depends on the dimensions of the cylinder and the physical characteristics of the metal. The longitudinal stresses on a 20 mm. length gave a value of under 2 kilos. per sq. mm. for copper; for nickel, between - 10 and + 6 kilos. per sq. mm.; and for brass containing 60% Cu  $\pm$  15 kilos. per sq. mm. A solid cylinder (70 mm. diam.) of semi-hard steel when quenched from 850° C., reheated to 550° C., and re-quenched in water gave a longitudinal compression of 14 kilos. per sq. mm. as against 27 kilos. when re-quenched from 700° C. For special work a certain rate of cooling may be adopted, sufficiently slow to avoid after-deformation, but on the other hand by too slow cooling certain steels (e.g. phosphoric and nickel-chromium steels) may fail on account of brittleness.—C. A. K.

*Silica products.* Bigot. See VIII.

*Zirconia.* Meyer. See VIII.

*Firchbrick from electric steel furnace.* Rees. See VIII.

*Refractory materials.* Mott. See VIII.

#### PATENTS.

*Iron; Electro-deposition of —.* F. A. Shepherd, and Brazil, Straker, and Co., Bristol. Eng. Pat. 119,200, 26.8.18. (Appl. 7074/18.)

An electrolyte for the electro-deposition of iron is prepared by boiling an aqueous solution containing 24 lb. of ferrous sulphate, 2½ lb. of ammonium chloride, and 18 lb. of iron filings in 11 gallons of water, and, whilst boiling, adding a solution of potassium bromide, 3 oz., boric acid, 2 oz., and common salt, 2 oz., in a gallon of water.—B. N.

*Metals [iron]; Production of finely-divided —.* T. A. Edison, Assignor to Edison Storage Battery Co., West Orange, N.J. U.S. Pat. 1,275,232, 13.8.18. Appl., 22.12.11.

Non-pyrophoric, electrolytically active iron, in a finely divided and highly porous condition, is prepared by reducing a suitable iron compound with hydrogen, and displacing the latter from the reduced metal by nitrogen before exposure to the atmosphere.—W. E. F. P.

*Steel shapes; Process of treating —.* J. Brunner, Evanston, Ill. U.S. Pat. 1,277,372, 3.9.18. Appl., 1.2.17.

STEEL shapes are rolled to a size rather greater than the required finished size, cooled below, and then reheated slightly above, the thermal critical range, and finally rolled to the finished size.—C. A. K.

*Steel [; Cobalt-chromium —].* P. R. Kuehnrich, Sheffield. U.S. Pat. 1,277,431, 3.9.18. Appl., 17.10.16.

SEE Eng. Pat. 106,187 of 1916; this J., 1917, 886. The proportions of carbon and chromium specified are 1.2—3.5 and 8—20% respectively.

*Magnetic iron product, and method of making same.* T. D. Yensen, Swissvale, Pa. U.S. Pat. (A) 1,277,523 and (B) 1,277,524, 3.9.18. Appl., 6.3.16 and 11.6.17.

(A) MOLTEN refined iron in which the total content of phosphorus, sulphur, and manganese is less than 0.05%, and of carbon below 0.02%, is treated with a deoxidising agent *in vacuo* to reduce the oxygen content to 0.05%. The magnetic material thus obtained contains intercrystalline impurities aggregating less than 0.15% and has a magnetic permeability of 13,000—70,000 at a flux density between 5000 and 10,000 gauss. (B) An iron product containing at least 99.7% Fe, not more than 0.05% C, 0.05% O, and 0.05% Mn + P + S, with a saturation intensity of magnetisation of 1800—1850.—C. A. K.

*Alloys containing nickel and zinc; Manufacture of —.* Stabilimenti "Biak," A. Pouchain, Assignees of P. Peynetti, Turin, Italy. Eng. Pat. 113,272, 5.11.17. (Appl. 1710/18.) Int. Conv., 23.10.16.

In the preparation of a nickel-zinc alloy, the zinc is first heated in a closed vessel until vaporised; the temperature is then raised and the nickel is added, and "melted in the mass of zinc vapour."—C. A. K.

*Electroplating apparatus.* J. E. Evans-Jackson, London. From G. T. Potthoff, New York. Eng. Pat. 119,091, 20.9.17. (Appl. 13,547/17.)

A BARREL to hold the articles to be plated is mounted to rotate in a tank containing the plating solution. Means are provided for rotating the barrel for several revolutions in one direction, and then automatically several revolutions in the other direction. The barrel is brought to rest after each period of rotation, and when at rest the source of current is automatically disconnected from the conducting means leading to the anode and the cathode in the barrel. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pat. 26,849 of 1904; this J., 1905, 1242.)—B. N.

*Tungsten metal powder; Manufacture of —.* O. Bertoya, London. Eng. Pat. 119,117, 15.10.17. (Appl. 14,915/17.)

The reduction of tungsten trioxide to metal is effected by hydrogen or a gas containing hydrogen, which is injected under moderate pressure, not exceeding a few pounds per sq. in., into the lower ends of a number of inclined externally heated retorts, through which the tungsten trioxide passes in succession. Disturbance of the surface of the oxide constantly exposes fresh material to the action of the gas, and the descent of the charge is facilitated by the change in its physical character as reduction progresses.—T. H. B.

*Gas [heating] furnace; Regenerative —.* W. Topham, Patricroft, Lancs. Eng. Pat. 119,273, 22.9.17. (Appl. 13,652/17.)

In a regenerative gas furnace for reheating ingots, billets, blooms, or the like for rolling or hammering, separate gas and air passages are provided for the two ends of the furnace, and are used alternately when the regenerators are reversed. The gas and

air conduits terminate in vertical portions on opposite sides of a wall, the outlets at the top of the wall being inclined towards one another so as to mix the hot gas and air in a chamber just behind a bridge member, over which the burning mixture passes into the furnace. The gas and air outlets are at a lower level than the top of the bridge. An auxiliary chamber is provided at the lower end of the vertical portion of the air conduit, in which solid matter may accumulate without obstructing the conduit, and holes are provided for its removal. To prevent overheating of the top of the wall where the mixture ignites, an air channel is provided within the wall.—W. F. F.

*Electrodes; Metal — used in depositing and soldering by the electric arc.* E. H. Jones, London. Eng. Pat. 119,279, 26.9.17. (Appl. 13,885/17.) Addn. to 14,468/15 (this J., 1916, 1222).

OTHER metals than mild steel may be used as base for the electrode, and are electroplated with another metal or metals.—B. N.

*Metals [nickel and copper]; Separation of —.* G. W. Pawel, Hudson Falls, and E. A. ReKate, Lancaster, N.Y., Assignors to United States Nickel Co., New Brunswick, N.J. U.S. Pat. 1,276,809, 27.8.18. Appl., 12.9.17.

RICH nickel-copper matte is roasted to remove the excess of sulphur, without affecting the sulphide sulphur, and is then mixed with about one-third of its weight of desulphurised copper obtained in a later stage of the process, finely ground, and agitated with hot strong sulphuric acid. The solution is evaporated and the nickel recovered by crystallisation. Copper and precious metals are recovered from part of the residue and the remainder is used to dilute the matte in an earlier stage of the process. Alternatively an easily soluble matte may be obtained by extensive bessemerising instead of by dilution.—T. H. B.

*Alloy.* F. S. Hodson, Philadelphia, Pa. U.S. Pat. 1,276,916, 27.8.18. Appl., 28.8.17.

AN alloy consisting of zinc, 80–85 parts, aluminium 18–14 parts, and copper 2–1 parts.—T. H. B.

*Alloy.* H. S. Cooper, Cleveland, Ohio, Assignor to The General Alloys Co. U.S. Pat. 1,277,046, 27.8.18. Appl., 17.4.18.

AN alloy, resistant to corrosion, consists of Ni over 50, Zr 1 to 25, Al 1 to 12, Si 1 to 10, and W under 25%, the amounts of zirconium, aluminium, and silicon together being greater than that of tungsten, and the silicon and aluminium together greater than the zirconium.—T. St.

*Ore concentration; Process of —.* W. A. Scott, Chicago, Ill., Assignor to Minerals Separation North American Corporation. U.S. Pat. 1,276,974, 27.8.18. Appl., 15.12.16.

AN unacrated emulsion of an insoluble frothing agent in a comparatively small quantity of water is forced through fine orifices and added to an ore pulp, from which the metalliferous matter is then recovered by flotation with the aid of a gas.  
—T. H. B.

*Flotation process and apparatus.* J. A. Pearce, Idaho Springs, Colo. U.S. Pat. 1,277,750, 3.9.18. Appl., 29.5.17.

A FLOTATION separating unit consists of an agitating compartment and a relatively deep flotation compartment communicating at the bottom. The float-

ation compartment is provided with a froth overflow, and a separate tailings discharge at a lower level, which feeds the next agitation compartment in series. Pulp is fed into the top of the agitating compartment and is aerated and expelled into the bottom of the flotation section, so that the agitation section is normally practically empty and the flotation compartment full.—C. A. K.

*Coating metals; Method of — [with lead].*  
*Method of plating metal castings.* R. J. Shoemaker, Milwaukee, Wis. U.S. Pats. (A) 1,276,977 and (B) 1,276,978, 27.8.18. Appls., 5.7 and 6.12.17.

(A) THE article to be plated is introduced into a bath of molten lead through a layer of flux composed of 80% of zinc chloride and 20% of common salt. (B) THE article is immersed in a bath of caustic soda and borax heated to a red heat and is afterwards introduced into a body of molten metal through a supernatant flux.—T. H. B.

*Metallic coatings; Production of — by spraying finely-divided molten metal on moving objects.* Metallatom Ges.m.b.H., Berlin-Tempelhof. Ger. Pats. (A) 307,378, 27.7 and (B) 307,406, 14.12.17.

(A) A NUMBER of articles to be coated with metal are placed in a rotary container so that they fall freely whilst exposed to the metallic vapour and particles from a spraying device. Openings are provided in the container for the removal of the excess of atomised metal. The spraying device is fixed at an angle at one end of the container and delivers the atomised metal the whole length of the container. A sand-blasting device may also be fitted to the apparatus. (B) THE article to be sprayed with atomised metal is moved mechanically in such a manner that it is coated uniformly, or the sprayed material may be caused to rotate around the article. If the material to be coated is in the form of strips or wires, it is moved by suitable means through a closed rotating vessel, in which it is sprayed, the thickness and density of the coating being regulated by the time the material remains in contact with the metal spray and by the temperature inside the container; the wire or strip may also be passed between pressing devices. Several articles arranged parallel to each other may be moved and coated simultaneously; if desired the wires may be rotated at the same time as they are being coated and are passing longitudinally through the container.—A. B. S.

*Crucible [for metal founding].* J. J. Boericke, Merion, Pa. U.S. Pat. 1,277,032, 27.8.18. Appl., 20.7.14. Renewed 29.6.18.

A CRUCIBLE is constructed in separate sections with a refractory friable continuous lining, permitting the ready exposure of the contents by removal of the separable portions.—C. A. K.

*Metallurgical operations; Apparatus for carrying on —.* J. J. Boericke, Merion, Pa. U.S. Pat. 1,277,033, 27.8.18. Appl., 20.7.14. Renewed 29.6.18.

AN enclosed chamber is divided into two connecting compartments by means of a battle-wall. One compartment is used as a reducing chamber, and is fitted with a door for the admission of a crucible (see preceding abstract). The other section serves as a fume-collecting compartment, and has a condensing flue opening from it. Material is admitted into the reducing chamber from a hopper by means of an externally controlled feed pipe.—C. A. K.



*Metals; Method of producing* — J. J. Boericke, Merion, Pa. U.S. Pats. (A) 1,277,034 and (B) 1,277,035, 27.8.18. Appl., 20.7.14. Renewed 29.6.18.

(A) METALLIC compounds are reduced by igniting a mixture of the metallic compound and a reducing agent (aluminium) and feeding a further quantity of the mixture on to the ignited material, so that the reaction is propagated in an upward direction and is under control. The reduction may be effected in a crucible of relatively large area to give access of air to the bulk of the mixture. (B) The process is modified by supplying the reducing agent in ingot or lump form.—C. A. K.

[*Copper;*] *Metallurgical process [for extraction of —]*. O. B. Dawson, El Paso, Tex., Assignor to Dawson Metallurgical Furnace Co. U.S. Pat. 1,277,047, 27.8.18. Appl., 11.7.17.

OXIDISED copper ore is heated in the presence of air to just below the melting point of copper, and sufficient carbon is then added to reduce the charge and to liberate enough carbon monoxide to provide the heat necessary to melt the metal formed. The liberated carbon monoxide is maintained at a pressure above atmospheric, and the charge is agitated to assist reduction and cause the metal particles to agglomerate. The mass is finally discharged into an atmosphere of water vapour to prevent re-oxidation, and the metal separated from the gangue.—T. St.

*Converter; Small — with side tuyères and provision for the removal of slag*. C. Raapke, Güstrow, Ger. Pat. 307,332, 18.1.18.

A SICKLE-SHAPED niche is made in the lining of a converter opposite the tuyères, in such a position as to compel the slag to follow a circular path and to remain in the converter. The slag retained in the niche, being protected from the action of the molten metal and the gases, does not absorb iron or silica, and so does not become excessively fluid and prevent the regular escape of the gases which—with thin slags—are liable to spurt badly. The retention of the slag within the cupola also retains more heat in the iron and facilitates the removal of gases from the metal.—A. B. S.

*Blast furnaces; Charging apparatus for —*. Blast Furnaces and Equipment, Ltd., and F. Ritson, London. Eng. Pat. 119,504, 28.9.17. (Appl. 14,023/17.)

*Alloys*. F. S. Hodson, Golder's Green. Eng. Pat. 119,486, 16.7.17. (Appl. 10,276/17.)

SEE U.S. Pat. 1,276,916 of 1918; preceding.

*Alloy*. F. Milliken, Lawrence, N.Y. U.S. Pat. 1,277,989, 3.9.18. Appl., 9.2.16.

SEE Eng. Pat. 118,825 of 1918; this J., 1918, 660 A.

*Electrode for arc soldering*. A. B. Pescatore, London. U.S. Pat. 1,277,639, 3.9.18. Appl. 10.6.18.

SEE Eng. Pat. 118,296 of 1917; this J., 1918, 628 A.

*Bronzing or colouring of metallic surfaces*. Q. Sestini and T. Rondelli, Bergamo, Italy. U.S. Pat. 1,277,761, 3.9.18. Appl., 15.5.17.

SEE Eng. Pat. 106,774 of 1916; this J., 1917, 888.

*Refractory bricks, etc.* Eng. Pat. 119,101. See VIII.

## XI.—ELECTRO-CHEMISTRY.

*Electrostatic precipitation*. O. H. Eschholz. Amer. Inst. Min. Eng., Sep., 1918. Chem. and Met. Eng., 1918, 19, 285—286.

IN practically all commercial plants for electrostatic precipitation, the mechanical rectifier is still employed for converting high-tension alternating into high-tension unidirectional current. In this apparatus a positive separation is made between the alternating current supplied and the unidirectional impulses sent to the electrostatic treater. By reason of the comparatively slow rate of discharge, the voltage across the treater is practically constant. Systems depending on the use of mechanical rectifiers are classified according to the source of power, that considered most satisfactory being one in which the power for a single transformer-rectifier-treater unit is obtained from a low-tension, single-phase, alternating-current generator for treater load only. The apparent advantage of low first cost is illusory, since the whole cost of the electrical equipment is only 10—15% of the cost of the treater and 2—7% of the value of the yearly recovery.—W. E. F. P.

*Refractories*. Hutton. See VIII.

### PATENTS.

*Electrolytic cells, adapted for the manufacture of caustic alkali from sodium chloride*. N. Statham, Hastings-on-Hudson, N.Y., U.S.A. Eng. Pat. 118,355, 26.9.17. (Appl. 13,884/17.)

IN an electrolytic cell of the diaphragm or Hargreaves type the diaphragm is provided on its outer side with relatively impervious material of varying thickness, so as to allow substantially uniform percolation of the electrolyte through the diaphragm at different heights, and at such a rate that back diffusion from the cathode chamber is prevented. Good results are obtained when the cathode electrolyte contains substantially equal proportions of electrolytically-produced caustic soda and undecomposed salt, or a slightly greater amount of the former substance. (Reference is directed, in pursuance of Sec. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 5197 of 1893, 20,889 of 1903, and 27,830 of 1907; this J., 1894, 528; 1904, 24; 1909, 96.)—B. N.

*Accumulators; Electric — or secondary batteries*. F. La Roche, Hampstead. Eng. Pat. 119,100, 26.9.17. (Appl. 12,853/17.)

AN electrode for secondary batteries is composed of about 75% of lead, 15% of nickel, and 10% of silver or cadmium, cast in one piece, with a ribbed surface and raised edge, with or without lugs or terminals.—B. N.

*Carbon electrodes; Method of transforming by heating in an electric furnace, anthracite, coke, and other materials rich in carbon into a product suitable for the manufacture of —*. S. E. Sierrin, Hôganås, Sweden. Eng. Pat. 119,164, 15.12.17. (Appl. 18,689/17.)

ANTHRACITE, coke, or other material rich in carbon is transformed into a product suitable for the manufacture of carbon electrodes by heating the material in an electric resistance furnace to 1800° to 2000° C., i.e. to a temperature approaching but not reaching that at which transformation of the carbon into graphite takes place. A suitable furnace consists of a casing of fireproof material with a vertical cylindrical cavity for the charge. The upper

electrode passes through the top of the furnace, and around it are channels for the supply of material. The bottom of the furnace consists of the lower electrode, through which is a cavity for removal of the charge. The furnace works as a resistance furnace, in which the charge itself forms the resistance.—L. A. C.

*Electric furnaces; Construction of — of the heated coil type.* J. E. P. Kievits, and Kynoch, Ltd., Birmingham. Eng. Pat. 119,302, 13.10.17. (Appl. 14,838/17.)

The heating coil is made from a wire formed into a close spiral and then wound round a tube of refractory material, the coils being embedded in cement. The spiral of wire is covered with paper or like substance, to prevent the cement obtaining ingress to the interior of the spiral, and the paper is subsequently burnt off whilst a current of air is being blown through the spiral passage.—B. N.

*Electric furnaces; Electrode holders for —.* A./S. Arendal Smelteverk, Eydehavn, Norway. Eng. Pat. 114,841, 5.4.18. (Appl. 5866/18.) Int. Conv., 13.4.17.

*Storage battery plates; Separators for —.* W. Morrison, Chicago, U.S.A. Eng. Pats. 111,469, 16.10, and 114,816, 25.9.17. (Appls. 14,979 and 13,786/17.) Int. Conv., 11.11.16 and 2.4.17.

SEE U.S. Pats. 1,228,368 and 1,228,369 of 1917; (this J., 1917, 891.)

*Separating liquids.* U.S. Pats. 1,276,386—7. See I.

*Calcining coal.* U.S. Pat. 1,277,707. See IIa.

*Detecting moisture.* Eng. Pat. 119,533. See XXIII.

## XII.—FATS; OILS; WAXES.

*Oil of red salmon, oil of "abura-garei," and liver oil of "chô-no-fuye."* S. Nakatogawa. Kôgyô-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 411—414.

Oil obtained from the body of the red salmon (*Oncorhynchus nerka*) had a reddish-orange colour, and remained fluid at  $-10^{\circ}\text{C}$ . After refining with kambara earth it had the following characters:—Sp. gr. ( $15^{\circ}/4^{\circ}\text{C}$ .), 0.9248; acid value, 7.4; saponif. value, 186.4; iodine value (Wijs), 148.1;  $n_D^{20}=1.4775$ ; m.pt. of fatty acids,  $26^{\circ}2'-28^{\circ}5'\text{C}$ .; and insoluble bromides, 38.9%. Two samples of oil of abura-garei (a species of flat fish, *Gymnosetta dubia*, found off the Japanese coast) had the following characters:—Sp. gr. ( $15^{\circ}/4^{\circ}\text{C}$ .), 0.9181, 0.9172; acid value, 6.1, 3.8; saponif. value, 182.9, 187.1; iodine value (Wijs), 95.8, 94.9;  $n_D^{20}=1.4725$ , 1.4730; m.pt. of fatty acids (one sample),  $19^{\circ}-21^{\circ}\text{C}$ .; and insol. polybromides (one sample), 10.91%. This oil is suitable for the manufacture of hardened oils. The liver of "Chô-no-fuye," *Calorhynchus japonicus*, a kind of cod, yields about 12% of oil of a brownish-yellow colour. It deposits "stearine" on standing. A sample from the coast of Izu, Japan, had the following characters:—Sp. gr. ( $15^{\circ}/4^{\circ}\text{C}$ .), 0.9254; acid value, 2.1; saponif. value, 187.7; iodine value, 120.57; and  $n_D^{20}=1.4761$ . It did not contain the hydrocarbon squalene,  $\text{C}_{30}\text{H}_{50}$ .—C. A. M.

*Apricot kernel oils; Mongolian and Chinese —.* S. Ueno. Kôgyô-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 525—527.

The kernels of the Mongolian apricot seeds (*Prunus armeniaca*, L.) contained 54% of oil, which, when extracted with ether, had the following characters: Sp. gr. at  $15^{\circ}/4^{\circ}\text{C}$ ., 0.9187; acid value, 0.7; saponif. value, 188.6; iodine value (Wijs), 105.4; and  $n_D^{20}=1.4723$ . The kernels of the Chinese apricot (*P. armeniaca*, L.) contained 53% of oil with the following characters:—Sp. gr. at  $15^{\circ}/4^{\circ}\text{C}$ ., 0.9101; acid value, 3.75; saponif. value, 182.3; iodine value (Wijs), 90.4; Behner value, 95.7; Reichert-Meissl value, 0.7; and  $n_D^{20}=1.4659$ .—C. A. M.

*Calcium soaps; Direct determination of — by means of solvents.* E. Salm and S. Prager. Chem.-Zeit., 1918, 42, 463—464.

CALCIUM soaps prepared from bone-fat, glue-leather fat, neat's-foot oil, linseed oil, and castor oil were extracted for 16 hours with various solvents, and the amount of extract determined. In all cases the soap swelled up into a gelatinous mass when heated with petroleum spirit, carbon bisulphide, benzene, or carbon tetrachloride, but not with acetone or ether, so that concordant results were obtained with the latter solvents. With the exception of castor oil calcium soap (solubility 92.2%) all the soaps dissolved with difficulty in acetone. Ether had a greater solvent action than acetone, especially in the case of the neat's-foot oil and linseed oil soaps. Hence acetone should be used rather than ether for separating neutral fats from calcium soaps. The calcium salts of bone-fat and glue-leather fat are readily soluble in benzene and carbon tetrachloride, and one of these solvents should be used instead of ether or acetone for extracting the total fat, including calcium soaps, from fatty material containing such soaps, provided that the material does not swell up in the treatment. The solubility of calcium soaps in benzene increases with the temperature, and is complete at the boiling point of the solvent after about 6 hours' extraction in a Besson flask. Moisture reduces the solubility, so that the material should first be dried at  $95^{\circ}$  to  $100^{\circ}\text{C}$ . The results thus obtained agree closely with those given by Fahrion's method (this J., 1899, 712).—C. A. M.

*Fatty acids; New method for the accurate estimation of the content of — in soaps.* E. Rosshard and F. Comte. Helv. Chim. Acta, 1918, 1, 251—270.

Errors are introduced in the estimation of fatty acids in soaps when these are weighed as such, owing partly to oxidation of the unsaturated acids and partly to the volatility of the lower fatty acids. The authors weigh them as the lead salts. A well-stoppered 100 c.c. Erlenmeyer flask is charged with about 5 grms. of lead oxide and a few pieces of pumice (dried at  $100^{\circ}\text{C}$ .), about 1 gm. of a fatty acid, and 30 c.c. of ether or petroleum spirit (or a mixture of the two) are added, the flask is shaken vigorously, and the solvent is removed by evaporation at  $60^{\circ}\text{C}$ . (chloroform bath) or  $107^{\circ}\text{C}$ . (toluene bath), at first under slightly reduced pressure to prevent foaming, and finally, when the solvent has disappeared, at 12 mm. pressure until the weight is constant (usually after about 20 minutes). From the equation  $2\text{R}\cdot\text{COOH} + \text{PbO} = (\text{R}\cdot\text{COO})_2\text{Pb} + \text{H}_2\text{O}$ , it is evident that the increase in the weight of the flask and its contents represents the weight of the acid anhydride; the weight of the acid is obtained by multiplying by the requisite factor. Examples are given in the case of individual acids showing that the method is trustworthy. When ascertaining the percentage of a mixture of fatty acids, the



mean molecular weight of the mixture must be determined. The method gives satisfactory results with the mixtures of acids obtained by hydrolysing natural oils, soaps, and colophony. The hydrolysis is effected in a Huggenberg and Stadlinger saponometer. The soap (4–6 grms., or 10 grms. in the case of a soft soap) is dissolved in hot water and introduced into the saponometer which contains 25–30 c.c. of N/1 sulphuric acid (a slight excess) and a few drops of methyl orange. Water is added to the mark 100, and, after sufficiently cooling, about 50 c.c. of a mixture of equal parts of ether and petroleum spirit. The mixture is shaken vigorously, 50 c.c. of the ether-petroleum spirit mixture is added, the mixture is again shaken, the acid layer is run off, and the ethereal solution, after being washed with 100 c.c. of water, is removed, and an aliquot part, about 25 c.c., is treated with lead oxide as described above.—C. S.

#### PATENTS.

*Oil press.* P. Zumberg and J. Sardin, Detroit, Mich. U.S. Pat. 1,277,677, 3.9.18. Appl., 18.4.18.

An outer casing with horizontal openings through its walls is secured to an inner casing with vertical slots through its walls. The casings are made in two halves and the outer halves can be rigidly secured round the inner casing.—J. H. P.

*Fatty acid esters; Forming and hydrogenating* —. C. Ellis, Montclair, N.J. U.S. Pat. 1,277,708, 3.9.18. Appl., 28.9.6.

UNSATURATED free fatty acid (oleic acid) present in fatty oils is esterified by heating the oil with an alcohol (ethyl alcohol), and the product is then treated with hydrogen in the presence of a hydrogenating catalyst.—L. A. C.

*Soap and process of making the same.* O. Ludekens, Assignor to the Wite-Kat Soap Co., San Francisco, Cal. U.S. Pat. 1,276,383, 20.8.18. Appl., 23.7.17.

A hot aqueous solution of soap is mixed with a substance, such as beeswax or "para wax," capable of forming an envelope enclosing minute particles of the liquid, and the mixture agitated, with or without aeration, to reduce it to the form of a paste —C. A. M.

*Wool-scouring liquors.* U.S. Pat. 1,277,784. See V.

*Edible oils.* Eng. Pat. 119,052. See XIXA.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### PATENTS.

*Gas-black, hydrogen, etc.; Process for manufacturing* —. C. D. McCourt, London, and C. Ellis, Montclair, N.J., Assignors to Surface Combustion, Inc., Wilmington, Del. U.S. Pat. 1,276,385, 20.8.18. Appl., 23.1.12.

METHANE is forced through a bed of strongly-heated, porous, refractory material, the resulting gas-black separated from the hydrogen, and the latter burned to maintain the heat of the bed.—C. A. M.

*Phenotic condensation products; Manufacture of* —. H. Wade, London. From Redmanol Chemical Products Co., Chicago, U.S.A. Eng. Pats. 119,252 and 119,253, 25.7.17. (Appls. 10,705 and 10,706/17.)

SEE U.S. Pats. 1,242,593 and 1,242,592 of 1917; this J., 1917, 1242.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*[Rubber] latex coagulation; Theory of* —. G. S. Whitby. Agric. Bull. Fed. Malay States, 1918, 6, 374–384.

THE phenomena of the coagulation of latex are compared with those of the clotting of milk, and the conclusion is drawn that with Hevea latex the coagulation in the presence of small amounts of acetic acid is more closely analogous with the clotting of milk by rennin than by the effect of acids or natural souring. With milk there is a critical concentration of acid below which both lactic acid and acetic acid not only fail to effect coagulation but indeed exert an anti-coagulating effect as compared with untreated milk; this peculiarity is probably due to the acids exerting an inhibitive influence on the growth of the lactic acid bacterium, and is in marked contrast with their coagulating effect on Hevea latex at all concentrations. The clotting of milk by acids is almost instantaneous, whereas the coagulation of Hevea latex under similar conditions may require from several minutes to several hours, the rate being comparable with that of the reaction between milk and rennin. The coherent clot in the last case also more closely resembles ordinary rubber coagulum than does the flocculent precipitate from acidified milk. The "natural" coagulation of Hevea latex is not a simple function of the acidity which is produced by bacterial action, and coagulation by heating or by tannic acid yields a flocculent or non-coherent coagulum analogous with that produced on acidifying milk. Rennin is activated by the presence of calcium salts and the influence of calcium salts on the "natural" coagulation of latex is probably of a similar nature. Bactericides, such as toluene or thymol, which are harmless to most enzymes, do not inhibit natural coagulation of latex, whereas hydrocyanic acid and potassium cyanide, which "paralyse" enzymes, prevent coagulation. Although small quantities of hydrochloric or nitric acid favour the coagulation of latex, with larger quantities a "neutral point" is reached at which no effect is produced, and still larger quantities again cause coagulation; this neutral point is attributable to the paralysis or destruction of the enzyme by acid at that particular concentration. The evidence of Eaton and Grantham (this J., 1916, 263) in support of their view that natural coagulation is due to lactic acid produced by bacterial action is not inconsistent with the enzyme theory. The initial formation of a coagulum in fresh latex allowed to undergo natural change is therefore regarded as due to enzyme action and the same agency, activated by acids, is probably responsible for the usual coagulation in the presence of small quantities of acetic acid (see also Barrowcliff, this J., 1918, 481). Bacterial action even in natural coagulation is only a secondary factor. Under unusual conditions, other types of coagulation may occur, e.g. delayed coagulation due to the putrefactive decomposition of latex which has previously been sterilised, and the formation of non-coherent coagula under the action of heat or of such substances as tannic acid, which are precipitants for the protein of the latex.—D. F. T.

*Destructive distillation of the beads of automobile tyres.* A. Dubosc. Caoutchouc et Gutta Percha, 1918, 15, 9617—9619.

BEADS of automobile covers, after removal of all but the last layer of fabric, were cut into fragments and heated in an iron retort, provided with a thermometer. The issuing gases and vapours were passed through an ice-cooled condenser into a measured volume of water. The products included a residue of 52%, oil 28.99%, water 8.33%, and gases (by difference) 10.68%; the residue consisted of carbon (10.92%) and mineral substances (11.08%), including zinc oxide, litharge, calcium carbonate, magnesia, and insoluble matter; the condensed liquids contained acetic acid, benzene, toluene, xylenes, ethylbenzene, and dipentene, but no isoprene or heveene; the gases were hydrogen sulphide and acetylene. Decomposition commenced to be vigorous between 75° and 145° C. and was complete at 245° C.—D. F. T.

## XV.—LEATHER; BONE; HORN; GLUE.

*Gelatin solutions; Influence of electrolytes upon the osmotic pressure of* —. J. Loeb. J. Biol. Chem., 1918, 35, 497—508.

THE influence of electrolytes upon the osmotic pressure of gelatin runs parallel to the influence exerted on the swelling, viscosity, and precipitation by alcohol (see this J., 1918, 383A, 384A, 520A). —H. W. B.

### PATENTS.

*Skins; Method and apparatus for drying* —. J. U. Flanagan, Assignor to F. F. Slocomb and Co., Wilmington, Del. U.S. Pat. 1,273,598, 23.7.18. Appl., 13.1.14.

SKINS are suspended, doubled, in a closed chamber, into which heated air enters, under pressure, through inlets exactly under the open spaces between the doubled parts of the skins. The air passes out into an exhaust chamber.—F. C. T.

*Tanning.* J. K. Tullis, Paris. Eng. Pat. 117,922, 6.7.17. (Appl. 8912/17.)

HIDES are first partly chrome tanned by any ordinary process, or simply immersed in a solution of sodium bichromate, after which the tanning is completed by treating the hides with a solution of wood pulp extract, either alone or in conjunction with any of the usual vegetable tanning materials or extracts.—F. C. T.

*Tanning of hides and skins.* B. Levin, Hale, Cheshire. Eng. Pat. 117,941, 7.8.17. (Appl. 11,347/17.)

HIDES and skins after cleaning, etc., are first treated with a solution of alum, or other soluble aluminium compound, and then with a solution of sodium pyrophosphate in sufficient quantity to convert the aluminium compound in the pores of the hides into insoluble aluminium pyrophosphate.—F. C. T.

*Tanning; Process of* —. W. Petrie, Endicott, N.Y., Assignor to Petrie Process Co., New York. U.S. Pat. 1,273,662, 23.7.18. Appl., 2.4.17.

HIDES are agitated, e.g. in a rotary drum, with a small quantity of concentrated tanning liquor. Air has free access to the hides, and the liquor is renewed as it is absorbed.—F. C. T.

*Leather; Manufacture of* —. A. Obser, Newark, N.J. U.S. Pat. 1,268,876, 11.6.18. Appl., 18.4.17.

HIDES are given a preliminary tannage in a bark liquor and are then split. The splits are pickled and subsequently subjected to mineral tannage. The leather is finally neutralised, washed, and finished. For stuffing the leather a fat liquor is used consisting of salt, fat, cottonseed oil, and mcollon.

—F. C. T.

*[Leather] product; Indurated* —. J. McIntosh, Morristown, Pa., Assignor to Diamond State Fibre Co., Bridgeport, Pa. U.S. Pat. 1,269,292, 11.6.18. Appl., 26.1.17.

THE claim is for leather or animal membrane impregnated with solid synthetic resin or condensate. (See U.S. Pat. 1,236,460 of 1917; this J., 1917, 1092.) —F. C. T.

*Leather; Substitute for — and process of and apparatus for manufacturing the same.* J. Ward, Bedford. Eng. Pat. 119,304, 16.10.17. (Appl. 14,982/17.)

LEATHER scrap or waste is roasted in a hot air kiln until crisp, ground into powder, and mixed with vulcanised rubber previously worked up into a fine powder. Sulphur is added to the mixture, also resin and sodium silicate if required, the mixture being heated to convert it into a semi-liquid state, in which form it is applied to coarse felt to coat the surface and impregnate the material.—B. N.

*Leather; Process of producing artificial* —. T. J. Turner, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,269,339, 11.6.18. Appl., 3.9.14. Renewed 19.12.17.

A FABRIC is coated with a plastic nitrocellulose composition by means of calender rolls.—F. C. T.

*Leather; Artificial — and means of producing the same.* W. W. Richards, Assignor to E. I. du Pont de Nemours Powder Co., Wilmington, Del. U.S. Pat. 1,270,055, 18.6.18. Appl., 16.11.12.

A FABRIC is coated with pyroxylin to which has been added an essential oil, musk, or animal oil, or all of these.—F. C. T.

*Artificial leather; Process of producing* —. C. E. Arnold, Wilmington, Del., Assignor to E. I. du Pont de Nemours and Co. U.S. Pat. 1,277,523, 3.9.18. Appl., 3.9.14.

A COATING of a viscous (pasty) solution of nitrocellulose is applied to a fabric, and after partial evaporation of the solvent the coated fabric is passed between rolls to give the desired thickness. —C. A. K.

*Leather; Method of drying varnished patent* —. E. Hintz, Wiesbaden, Assignor to C. Heyl, Worms, Germany. U.S. Pat. 1,269,931, 18.6.18. Appl., 12.12.16.

VARNISHED (patent) leather is exposed to the action of light of short wave length only and rich in ultra-violet rays, and at the same time all air or other oxidising agent is excluded.—F. C. T.

*Tannery waste disposal process.* H. W. Taylor, New York. U.S. Pat. 1,277,776, 3.9.18. Appl., 28.11.16.

TANNERY waste liquors are separated into three classes, i.e., tanning wastes, liming wastes, and miscellaneous waters. The tanning wastes and liming waste are mixed in such proportion that



the resulting liquor is just neutral, and the precipitate formed is filtered off. The liquor is then added to the miscellaneous waste liquor and the mixture treated with a suitable precipitant, *i.e.*, iron sulphate.—L. A. C.

*Gelatin; Process for preparing* —. T. Tada, Tokushima Ken, Japan. U.S. Pat. 1,271,960, 9.7.18. Appl., 1.6.18.

A SOLUTION of gelatin, bleached and clarified, is concentrated *in vacuo* to 30–60% strength, allowed to set in thin sheets in moulds, and subsequently dried at a temperature not lower than 40° C.

—F. C. T.

*Gelatin; Process for preparing* —. T. Tada, Tokushima, Japan. Eng. Pat. 119,210, 3.6.18. (Appl. 9144/18.)

SEE U.S. Pat. 1,271,960 of 1918; preceding.

## XVI.—SOILS; FERTILISERS.

*Azotobacter; Soil reaction and the growth of* —. P. L. Gainey. J. Agric. Res., 1918, 14, 265–271.

NEARLY a hundred samples of soils from widely different sources, both cultivated and uncultivated, were examined for the presence of azotobacter. 28% of the samples gave the typical azotobacter growth, with a fixation of nitrogen of 9.47 mgrms. per culture. In 41% no azotobacter were found, and these soils fixed 3.88 mgrms. of nitrogen on the average. The other soils gave a mixed growth. The presence or absence of azotobacter had no regular relation with any of the conditions of the soils, except possibly that of the hydrogen-ion concentration. Soils in which this factor was less than  $P_{H6}$  contained no azotobacter; those above  $P_{H6}$  contained azotobacter.—J. H. J.

*Calcium cyanamide; Alteration of — on storage.* J. P. van Zyl. Z. angew. Chem., 1918, 31, 203–204.

THE author has examined a sample of calcium cyanamide which had lain exposed on the floor of a barn for 2½ years. The original material was stated to have contained 19% of total nitrogen; the sample after storage showed 14.64%. The residue insoluble in boiling water contained 1.32% of nitrogen; the aqueous extract was analysed by Caro's method and showed 131.3 mgrms. of total nitrogen per grm. of the sample, comprising 99.5 mgrms. in the form of cyanamide, 2.1 as dicyanodiamide, 10 as ammonia, and the rest presumably in the form of urea. It appears remarkable that after storage for 2½ years without special precautions, the freshness of the cyanamide should have been so well preserved, in view of the fact that Kappen had found a total disappearance of cyanamide from an old sample and the conversion of 75% of the nitrogen into dicyanodiamide.—J. F. B.

## PATENTS.

*Superphosphate; Apparatus for the manufacture of* —. G. A. Mower and F. W. R. Williams, London. Eng. Pat. 119,074, 17.9.17. (Appl. 13,334/17.)

THE mixture of phosphate and acid is placed in a movable truck having a removable front wall, side walls hinged at the top, and closed at the bottom by a stationary bed upon which the block of superphosphate remains when the reaction is completed. The front wall of the truck is then removed, and rotary cutters attached to the truck are maintained in contact with the exposed surface by moving the superstructure relatively to the base.—W. E. F. P.

*Fertiliser materials; Process of producing* —. T. C. Meadows, Assignor to Kaolin Products Corporation, New York. U.S. Pat. 1,276,555, 20.8.18. Appl., 8.12.16.

“GREEN SAND” (a deposit of fine or coarse sands, mixed with varying quantities of glauconite, forming a subdivision of the Cretaceous system) containing a substantial proportion of glauconite is calcined to convert the iron into the ferric state, finely ground, and mixed with water to form a sludge. Commercial mono- and di-calcium phosphates are added to the sludge, which is then digested at a pressure above three atmospheres until mono- and di-potassium phosphates are formed.—J. H. J.

*Calcium cyanamide; Process for granulating* —. G. W. Sinclair, Petersham. U.S. Pat. 1,278,041, 3.9.18. Appl., 19.12.16.

SEE Eng. Pat. 105,578 of 1916; this J., 1917, 607.

## XVII.—SUGARS; STARCHES; GUMS.

*Sugar losses in manufacture.* L. Fauque. Bull. Soc. Chim. Maurice, 1918, 9, 45–47. Int. Sugar J., 1918, 20, 477–478.

THE theoretical extraction of first jet sugar is given by the formula:  $\frac{P-P'}{P(100-P')}$ , in which  $P$  represents the purity of the syrup in course of working, and  $P'$  that of the exhausted syrup. The difference between the calculated extraction and the sugar contained in the syrup represents the loss in the molasses, and that between the sugar in the syrup and the sugar entering the factory indicates the loss in the scums plus the undetermined loss. On applying this method of calculation in factories in Mauritius, it was found that the loss in the scums plus undetermined loss may vary between from 0.60 to 0.10, and even less, on the weight of cane crushed.—J. P. O.

*Melezitose; Occurrence of — in a manna from the Douglas fir.* C. S. Hudson and S. P. Sherwood. J. Amer. Chem. Soc., 1918, 40, 1456–1460.

A SAMPLE of manna from the Douglas fir yielded about 50% of pure, crystallised melezitose, and there is evidence that it contained sucrose and some reducing sugar, probably a mixture of dextrose with a smaller quantity of levulose. The percentage composition of the sample of dried manna was approximately: melezitose, 75–83; sucrose, 2.9; reducing sugars, 11.5%. At present, the only other known natural source of melezitose in any quantity is the Turkestan manna (Tarandjabine), which is, however, considerably inferior to the Douglas fir product in point of yield.—H. W.

*l-Glucosan; Conversion of — into dextrin.* A. Pictet. Helv. Chim. Acta, 1918, 1, 226–230.

WHEN kept at 240° C. for 30–60 minutes or, better, at 180° C. for a few minutes in the presence of platinum black as catalyst, *l*-glucosan (see this J., 1918, 49A) is converted into a white amorphous powder,  $(C_6H_{10}O_5)_n$ ,  $[\alpha]_D$  varying from +111.9° to +106.5° in different preparations, which exhibits the characteristic properties of a dextrin and yields dextrose when warmed with dilute sulphuric acid. —C. S.

## PATENT.

*Starch; Process for producing laundry* —. S. B. Chambers, Chicago, Ill. U.S. Pat. 1,276,722, 27.8.18. Appl., 1.12.16.

DRY maize starch, 100 lb., is mixed with 15 oz. of powdered borax and treated with a solution of 6 oz.

of nitric acid in 6 galls. of water; the mixture is allowed to remain for 24–48 hours, during which time it is stirred at intervals to prevent excessive heating, and is finally dried at 130°–155° F. (54.5°–68.5° C.).—J. F. B.

### XVIII.—FERMENTATION INDUSTRIES.

*Alcoholic fermentation; Role of the phosphate in —.* H. Euler and S. Heintze. *Z. physiol. Chem.*, 1918, 102, 252–261.

The extent of the esterification of phosphoric acid which occurs when dried yeast is incubated with dextrose in the presence of disodium hydrogen phosphate and a protoplasmic poison, such as phenol, is dependent on the amount of water remaining in the yeast after the drying process. The maximum esterification was observed when dried yeasts containing from 10 to 15% of moisture were employed. Increasing the quantity of yeast used in the individual experiments appeared to cause a relatively much greater increase in the amount of hexose-phosphate produced.—H. W. B.

*Fermentation [by zymase]; Kinetics of the cell-free —.* O. Meyerhof. *Z. physiol. Chem.*, 1918, 102, 185–225.

The interval which elapses between the addition of sugar to an extract of dried yeast and the appearance of fermentation (the "induction period") can be shortened by previously warming the sugar solution with disodium hydrogen phosphate or by grinding the dried yeast with glass powder. This shortening of the induction period is probably due to the formation or liberation of hexose-phosphate, the addition of a small quantity of which suffices to cause the immediate onset of fermentation with evolution of carbon dioxide. The rate of fermentation is dependent on the amount of free phosphate present; by increasing the latter, the rate at which the velocity of fermentation increases is reduced, but the maximum velocity eventually attained is higher than in the absence of free phosphate until a certain maximum amount of phosphate is reached; further addition of the phosphate then reduces the maximum velocity of fermentation attainable. The addition of other salts such as sodium chloride produces similar effects on the velocity of fermentation; the free phosphate functions therefore as a salt as well as exerts its specific zymase-activating action. Fermentation is accelerated by the addition of hexose-phosphate or of co-ferment such as is present in boiled yeast extract.

—H. W. B.

*Wood-waste as a source of ethyl alcohol.* G. H. Tomlinson. *Chem. and Met. Eng.*, 1918, 19, 552–568.

The past failures in the attempts to utilise sawdust and other forms of wood-waste as a source of alcohol are due to premature development of the process under unsuitable commercial conditions. It is not necessary for the fermentation part of the process to be carried out at the same time as the conversion; the solution of soluble carbohydrates obtained from the wood may be evaporated to a syrup resembling cane molasses and transported for fermentation elsewhere. The residue from the wood-waste (about 70%) is not depreciated in value as fuel, and the important factor in the development of the industry is the cost of the syrup compared with that of cane molasses. The cost of the plant used by the Standard Alcohol Co. at Fullerton, La., was \$456,920, of which about \$200,000 represented the

cost of the plant and equipment for the conversion of the wood into sugar and the separation of this in the form of a solution, and the balance for the fermentation and distillation plant. Working for 22 days in 1913 at three-quarter capacity, this plant dealt with 6125 tons of green waste wood containing 48% of moisture, and yielded 1,688,600 galls. of sugar solution of about 10.3% strength, at a total cost, including allowance for depreciation of plant, of 31.8 cents per 100 galls. The molasses obtained by concentrating 100 galls. of the sugar solution to 12½ galls. may be taken as costing about 2 cents per gall. including evaporation. On the other hand, whilst a gallon of wood molasses would yield only about 0.39 gall. of proof spirit, cane molasses yields about 1 gall. of spirit. In practice, although about 24% of the dry wood was rendered soluble, only about 8.5% was converted into fermentable sugars, but by proper chemical control of the operations the author considers that it should be possible to obtain a product equal to cane molasses in fermentable value.—C. A. M.

*Ethyl and methyl alcohols; Method of determining the amount of methyl alcohol in mixtures of —.* W. G. Topliss. *Amer. J. Pharm.*, 1918, 90, 636–640.

The method depends on the measurement of the volume of hydrogen liberated on treating the dried alcohol with sodium. The sample is compared with a standard sample of denatured alcohol of known composition. The apparatus consists of a small test-tube weighted so as to sink in petroleum spirit contained in a hydrometer jar. The test-tube carries a gas-leading tube of brass terminating in a trap which allows the hydrogen to pass out but prevents the entrance of the liquid. Six drops of the alcohol is placed in the test-tube and a piece of sodium carried on the point of a needle which is suspended by a thread which passes up through the gas outlet. An inverted burette filled with petroleum spirit is placed over the test-tube submerged in the hydrometer jar and releases the suspended needle. The reaction begins at once and the hydrogen is collected in the burette. If two operations are conducted side by side, the sample may be compared with the standard without correcting the volume of gas evolved. If the reading of the unknown sample is less than that of the standard, it is deficient in wood spirit. The alcohols must be dehydrated before making the test by treatment with fused potassium carbonate.

—J. F. B.

#### PATENTS.

*Alcohol-reduced beverages; Apparatus for making —.* H. Heuser, Chicago, Ill. U.S. Pat. 1,277,931, 3.9.18. Appl., 25.11.16.

A VERTICAL vacuum evaporator surrounded by a steam jacket at the sides and the bottom is supplied with a vapour outlet at the top, a liquid outlet at the bottom, and a liquid sprayer arranged to discharge a film of liquid on to the heated sides, thereby momentarily boiling the beverage in the form of a flowing film. Steam is supplied to the jacket by a bank of separately controlled pressure steam injectors.—L. A. C.

*Alcohol; Process for producing —.* B. Kazmann, Battle Creek, Mich., U.S.A. Eng. Pat. 119,333, 15.11.17. (Appl. 16,829/17.)

SEE U.S. Pat. 1,267,681 of 1918; this J., 1918, 558A.

*Solidified alcohol.* U.S. Pat. 1,277,149. See II.A.



## XIXA.—FOODS.

*Milk; New formula for the calculation of added water in* —. L. J. Harris. Analyst, 1918, 43, 345–347.

A FORMULA is given by means of which the percentage of added water is calculated on the assumption that the original milk, before the addition of water, contained the official minima of fat and solids-not-fat (3.0 and 8.5%, respectively); allowance is made for any excess or deficiency of fat. Let  $N$  and  $F$  be the percentages of solids-not-fat and of fat in the milk-water mixture, and  $x$  the percentage of milk of minimum fat and solids-not-fat content in the mixture, then the solids-not-fat should be  $8.5 \frac{x}{100}$ , the fat  $3 \frac{x}{100}$ , and the water  $100 - 11.5 \frac{x}{100}$ . Now the rising of cream has resulted in the fat content changing from  $3 \frac{x}{100}$  to  $F$ , and the remaining total constituents (water + solids-not-fat) will have changed from  $100 - 3 \frac{x}{100}$  to  $100 - F$ , and there will now be

$$\frac{100 - F}{100 - 3 \frac{x}{100}} \times \left( 100 - 11.5 \frac{x}{100} \right) \% \text{ of water,}$$

$$\text{and } \frac{100 - F}{100 - 3 \frac{x}{100}} \times 8.5 \frac{x}{100} \% \text{ of solids-not-fat.}$$

$$\text{Hence } N = \frac{8.5x(100 - F)}{10,000 - 3x},$$

$$\text{and } x = \frac{10,000 N}{3N + 8.5(100 - F)}.$$

The percentage of added water in the milk will be  $100 - x$ .—W. P. S.

*Pastry; Detection of milk in* —. J. Grossfeld. Z. Unters. Nahr. Genussm., 1918, 35, 457–471.

THE method proposed depends on the conversion of lactose into mucic acid. Fifty grms. of the sample is shaken with 500 c.c. of water, filtered, and 250 c.c. of the filtrate evaporated to a thin syrup with the addition of 5 c.c. of glacial acetic acid; the syrup is treated with 100 c.c. of warm 90% alcohol, the mixture filtered, and the filtrate evaporated to dryness. If a large quantity of sucrose is also present, this dry residue must be dissolved in 100 c.c. of alcohol, 100 c.c. of ether added, the precipitated sugars collected after 24 hours, and again treated with alcohol and ether. In this way the lactose is concentrated in the precipitate and the greater part of the other sugars removed. The precipitated lactose is heated for a short time on a water-bath with 30 c.c. of nitric acid (sp. gr. 1.15), the solution filtered, and the filtrate placed aside; mucic acid usually crystallises out within 24 hours, but may require several days. The mucic acid obtained melts at  $200^{\circ}$ – $210^{\circ}$  C., and each grm. requires 9.5 c.c. of  $N/1$  sodium hydroxide for neutralisation. About 30% of the lactose present is converted into mucic acid. The calcium oxide content of pastry may also afford some indication as to whether milk has been used in its preparation; pastry made without milk may contain up to 0.2% of calcium oxide (calculated on the dry substance); the presence of milk increases this quantity, but, especially if the calcium oxide content exceeds 0.35%, the increase may be due to ingredients other than milk.—W. P. S.

*Fat-soluble vitamine [of butter-fat].* I. H. Steenbock, P. W. Boutwell, and H. E. Kent. J. Biol. Chem., 1918, 35, 517–526.

THE fat-soluble vitamine in butter-fat is destroyed by heating the butter-fat at  $100^{\circ}$  C. for 4 hours.  
—H. W. B.

*Starch; Brake-fern (Pteris aquilina, L.) as a source of* —. A. Zlataroff. Z. Unters. Nahr. Genussm., 1918, 35, 483–484.

ELEVEN kilos. of washed and dried brake-fern roots yielded 7.6 kilos. of meal which contained:—water, 8.04; starch, 46.00; cell tissue, 22.11; ash, 10.48%. The meal had a grey colour and a bitter taste, but the latter might be removed by washing and sedimentation; it appears to be more suitable as a cattle food than for bread-making.—W. P. S.

*Cottonseed products; Comparative toxicity of* —. W. A. Withers and F. E. Carruth. J. Agric. Res., 1918, 14, 425–452.

FEEDING experiments on rats, rabbits, poultry, and pigs were made with cottonseed meal, raw cottonseed kernels, ether-extracted kernels, and gossypol. Toxic properties were found in all the products. If the meal had been prepared by the "cold-pressing" process, most of the toxic properties passed into the oil. Cooking of the products under oil-mill conditions caused them to become non-toxic to rabbits and pigs. This was due to the oxidation of the gossypol to its derivative "D-gossypol," which was far less toxic. (See also this J., 1917, 562, 937, 1061, 1285; 1918, 164 A, 319 A.)—J. H. J.

*Coffee; Determination of caffeine in* —. E. Vautier. Ann. Chim. Analyt., 1918, 23, 207–210.

FIVE grms. of the finely powdered sample is moistened in an extraction thimble with 5 c.c. of ammonia and then extracted in a Soxhlet apparatus for 4 hrs. with ether; the extract is evaporated, the residue treated with boiling water, the solution filtered, and the insoluble portion washed with boiling water until 150 c.c. has been collected. The filtrate is evaporated, the residue dissolved in a small quantity of boiling water, the solution filtered, the insoluble portion washed, and the filtrate again evaporated in a small basin. The residue thus obtained is heated and the sublimed caffeine collected on a watch-glass and weighed.—W. P. S.

*Silage made from various crops; Acidity of* —. R. E. Neidig. J. Agric. Res., 1918, 14, 395–409.

EXPERIMENTS were made to determine whether other crops than maize could be used for the production of silage. It was found that oats, peas, clover, and mixtures of oats with peas, wheat with peas, and clover with wheat straw made excellent silage, and produced acetic, propionic, and lactic acids in similar amounts to maize silage. Alfalfa (lucerne) made poor silage, and produced butyric acid only.  
—J. H. J.

*Nucleic acids.* Feulgen. See XX.

*Determining histidine, etc.* Lautenschläger. See XX.

*Vitamines.* Uhlmann. See XX.

## Erratum.

THIS J., Nov. 15, 1918, p. 667 A, col. 2, line 6 from bottom should read "constituents of milk caused by bacteria before any".

## PATENTS.

*Milk and similar liquids; Process for sterilising* —. F. G. Lecomte, Amsterdam. Eng. Pat. 116,490, 20.7.17. (Appl. 10,455/17.) Int. Conv. 7.6.17.

MILK, condensed or raw, is placed in a closed vessel, and air at a pressure of 1.2 atmospheres is forced into the liquid for about 30 minutes, so that some air is dissolved. The milk is then sterilised at 105°–110° C.—W. F. F.

*Lactical fluids; Method of treating* —. [Manufacture of butter.] E. B. Heller, Assignor to The Vacuum Churn Corporation, New York. U.S. Pat. 1,276,910, 27.8.18. Appl., 19.1.15. Renewed 19.6.16.

MILK is pasteurised, churned, and worked in a rotating partially exhausted vessel, having smooth, non-porous, non-absorbent walls, which are cooled below 62° F. (17° C.), with the object of preventing adherence of the globules of butter formed in the process.—C. A. M.

*Edible oils and fats; Means for imparting a definite flavour to* —. De Bruyn, Ltd., and J. De Bruyn, London. Eng. Pat. 119,052, 21.6.17. (Appl. 8929/17.)

CRUDE proteins are extracted from vegetable matter such as the seeds or fruits of leguminous plants, the extract is inoculated at about 70° F. (21° C.) with an active culture of lactic bacteria, and after attaining the required degree of sourness it is emulsified with edible oils and fats at a suitable temperature (70° to 100° F., 21°–38° C.). The extract of crude proteins may be evaporated and the residue dried to obtain a dry product which may be kept for subsequent dilution with water and ripening with lactic bacteria. By this means a butter-like flavour is imparted to edible oils and fats more economically and with greater uniformity than by the use of cows' milk.—C. A. M.

*Fruit-dryer*. U. G. Stover, Waynesboro', Pa. U.S. Pat. 1,276,988, 27.8.18. Appl., 15.10.17.

FRUIT is dried in a frame, with open upper and lower ends, which contains a series of removable superposed trays. Attached by hinges to opposite sides of the top of the frame are two plates, having openings in their margins near the hinges, and adapted to be brought together over the top of the frame, or fixed at different positions in relation thereto.—C. A. M.

*Food product*. P. R. Welch, Assignor to The Welch Grape Juice Co., Westfield, N.Y. U.S. Pat. 1,277,672, 3.9.18. Appl., 4.10.17.

A MIXTURE of grape pulp and juice is stored until argol crystallises throughout the mass. The argol is separated, and the pulp and juice are mixed with sugar and cooked until the proper consistency is reached.—J. H. P.

*Eggs; Process of recovering substances from fresh, decomposed, or partially decomposed* —. J. M. Hussey, Wichita, Kans. U.S. Pat. 1,277,727, 3.9.18. Appl., 29.12.16.

SUFFICIENT salt is added to fresh, decomposed, or partially decomposed eggs to form a brine, thus dissolving the vitellin, and carbon tetrachloride and methyl alcohol are then added in sufficient quantities to dissolve the oils and fats and to precipitate the albumin. The solution of oils and fats, which also contains lecithin, is drawn off, the fat-solvent (carbon tetrachloride) recovered, and the lecithin

precipitated from the mixture of oils and fats by the addition of sufficient fat-solvent and acetone. The lecithin is then strained out and the fat-solvent and acetone recovered from the solution. After straining off the albumin, methyl alcohol is separated from the solution containing the vitellin by distillation, and the vitellin obtained by the addition of sufficient water to dissolve out the salt, leaving the vitellin as a precipitate.—L. A. C.

*Flour and starch from potatoes and other tubers and roots and other materials; Machinery for use in production of* —. R. G. McLeod, London. Eng. Pat. 119,531, 4.10.17. (Appl. 14,319/17.)

*Milk; Process of making a new food product of* —. P. W. Turney, Richmond, Va., U.S.A. Eng. Pat. 119,430, 24.6.18. (Appl. 10,414/18.)

SEE U.S. Pat. 1,274,218 of 1918; this J., 1918, 669 A.

## XIXB.—WATER PURIFICATION; SANITATION.

*Waters; Hypochlorites in drinking* —. A. R. Ling. Analyst, 1918, 43, 347–348.

TREATMENT of drinking water with hypochlorite converts the ammonia and amino compounds present into chloramine and chloramino derivatives, and does not reduce the ammonia and albuminoid ammonia content as determined by the Nessler method. The fact that the chlorine in chloramines liberates iodine from potassium iodide may be used to ascertain whether a water containing much nitrogenous matter has been treated with hypochlorite. Fifty c.c. of the water and 50 c.c. of distilled water are placed in two cylinders and each treated with 1 c.c. of N/1 sulphuric acid and 0.1 grm. of potassium iodide. If the water has been treated with hypochlorite a brown coloration develops, whilst the distilled water will remain colourless. Nitrites, which also liberate iodine from potassium iodide, would have been oxidised by the hypochlorite.

—W. P. S.

*Water purification; Baryta as substitute for soda in* —. F. Hundeshagen. Z. öfentl. Chem., 1918, 24, 175–186.

SEE this J., 1918, 670 A. The author gives examples of the cost of treatment by the baryta process for various combinations of hardening salts, as well as directions for the chemical control of the process in practice.—J. H. J.

*Disinfection by alcohol; Theory and practice of* —. J. Christiansen. Z. physiol. Chem., 1918, 102, 275–305.

THE disinfecting power of alcohol cannot be associated entirely with its property of precipitating the proteins of bacteria, because those concentrations of various alcohols which cause the same amount of precipitation of a protein differ greatly in their disinfecting efficiency. One of the most important factors in disinfection is the capacity of the disinfectant to penetrate the cell-walls of the bacteria, and this is largely determined by the surface tension of the disinfectant. Of all the alcohols, propyl alcohol appears to possess the most valuable qualities required by a disinfectant for direct application to the epidermis prior to surgical operations. Its solutions have lower surface tensions than solutions of corresponding concentrations of ethyl alcohol, which has hitherto been largely employed in this



connection. Moreover, the toxicity of propyl alcohol is at least four times as great as that of ethyl alcohol, and, owing to its greater solvent power for fats, it is able to enter readily the pores of the skin.—H. W. B.

*Sodium hypochlorite solutions; Hydrogen ion concentration of various indicator end-points in dilute* —. G. E. Cullen and J. H. Austin. *J. Biol. Chem.*, 1918, 34, 553–568.

In the preparation of his hypochlorite solution for wound disinfection (this J., 1915, 919), Dakin added boric acid to a strongly alkaline hypochlorite solution until it no longer coloured powdered phenolphthalein. It is pointed out by the authors that such a solution is still alkaline; in consequence of the bleaching action of the hypochlorite, a much greater alkalinity is required to give a colour with phenolphthalein when hypochlorite is present than in its absence. Experiments showed that in a 0.5% solution of sodium hypochlorite, in which all the hypochlorite is present as NaClO, the end-point to powdered phenolphthalein is at  $p_{\text{H}}$  = about 10.1, and to an alcoholic solution of *o*-cresolphthalein (this J., 1916, 980) about 9.3. With an alcoholic solution of phenolphthalein as indicator, the end-point is uncertain owing to rapid decomposition of the hypochlorous acid which forms a considerable proportion of the total hypochlorite in dilute solutions of hypochlorite. With a 1% solution of sodium hypochlorite, in which 7–17% of the total hypochlorite is present as HClO, the end-point to an alcoholic solution of *o*-cresolphthalein is at  $p_{\text{H}}$  = about 9.6. Such a solution is sufficiently stable to be satisfactory for clinical use, and might perhaps be tolerated upon skin and other tissue surfaces which would not bear the more alkaline solution prepared as described by Dakin. It can be readily prepared by passing chlorine gas through a solution of sodium carbonate, of an initial concentration of 14 grms. per litre, till the concentration of sodium hypochlorite, as shown by titration with thio-sulphate, is 0.5%; the resulting solution has  $p_{\text{H}}$  = about 9.4.

*Phenols as preservatives of anti-pneumococcic serum.* C. Voegtlin. U.S. Pub. Health Service, Hyg. Lab., Bull. No. 112, 1918, 5–13.

It is usual to preserve anti-pneumococcic serum by the addition of 0.3–0.5% of phenol or cresol. As large amounts of the serum are used in injections, an investigation was made by experiments on animals to determine whether the amount of phenols thus injected was injurious. No toxic effects were observed in the animals experimented on.—J. H. J.

*Biological products; Studies in preservatives of* —. *Effects of certain substances on organisms found in biological products.* M. H. Neill. U.S. Pub. Health Service, Hyg. Lab., Bull. No. 112, 1918, 37–46.

It is usual to add 0.5% of phenols to biological products, such as vaccines, serums, etc., to keep them sterile, but occasionally living organisms are found to be present in them. Cultures of some of these organisms, mainly cocci, were exposed to the action of dilutions of phenol, 1 in 110, and of formaldehyde, 1 in 50, and these substances were found to be ineffective as germicides. The use of stronger solutions would be deleterious to the biological product and unduly toxic.—J. H. J.

*Ether; Effect of* — *on tetanus spores and on certain other micro-organisms.* H. B. Corbitt. U.S. Pub. Health Service, Hyg. Lab., Bull. No. 112, 1918, 47–48.

ETHER was found to be ineffective for destroying spores of the tetanus bacillus even after two weeks' treatment, whereas cultures of *Staph. aureus* and *B. typhosus* were easily killed.—J. H. J.

*Nitrites and nitrates.* Oelsner. See VII.

#### PATENTS.

*Evaporating and distilling machinery [for sea-water].* D. Urquhart, London. Eng. Pat. 119,106, 2.10.17. (Appl. 14,217/17.)

To obtain distilled water on board ships which are propelled by internal combustion engines, salt water is evaporated and distilled by means of steam produced by heating distilled water in tubular heaters or boilers by means of the exhaust gases from the engines. The heaters may be of the water-tube or fire-tube type.—W. H. C.

*Water-purification system; Vacuum* — *[for removal of gaseous impurities].* O. M. Campbell, Kansas City, Kans. U.S. Pat. 1,276,871, 27.8.18. Appl., 27.1.16.

WATER is purified by distillation and gaseous impurities are removed by subjecting the vapour during condensation to a "continuous vacuum action" at various points corresponding to the various stages of condensation of the vapour.

—L. A. C.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Nux vomica and its preparations; Variations in* —. H. H. Schaefer. Amer. J. Pharm., 1918, 90, 633–635.

The specification for *Nux vomica* in the U.S. Pharmacopœia VIII required 1.25% of strychnine after the removal of the brucine with nitric acid. This has been revised in the U.S.P. IX, which specifies 2.5% of total alkaloids without regard to the proportions of strychnine and brucine. The author has analysed several samples of powdered *Nux vomica* and its fluid extract and tincture and found considerable variations: the percentage of strychnine in the total alkaloid ranged from 33 to 53. Hence certain samples may pass the new total alkaloid specification which would not pass the old strychnine specification. One sample of *Nux vomica* contained 2.52% of total alkaloids but only 0.73% of strychnine, and another contained 1.26% of strychnine but only 2.38% of total alkaloids. The author considers that there would be no difficulty in meeting a specification which comprised both requirements, viz. at least 2.5% of total alkaloids and 1.25% of strychnine.—J. F. B.

*Biological products; Nature of contaminations of* —. I. A. Bengtson. U.S. Pub. Health Service, Hyg. Lab., Bull. No. 112, 1918, 14–36.

VARIOUS biological products, such as vaccines, anti-toxins, and tuberculins, were examined for their freedom from bacterial contamination. Occasionally organisms were found to be present, mainly staphylococci, streptococci, and bacilli. Spore-bearing bacilli were most frequently found, usually in bacterial vaccines and in tuberculins. The next most frequent were staphylococci, usually found in

small-pox and rabies vaccines. Other organisms found were streptococci, bacilli, and one anaerobic bacillus. The organisms were mainly saprophytic, though in a few cases pathogenic species were isolated from small-pox and rabies vaccines and tetanus antitoxin.—J. H. J.

*Piperazine; Preparation of* —. D. S. Pratt and C. O. Young. J. Amer. Chem. Soc., 1918, 40, 1428–1431.

The following process has proved satisfactory. A mixture of ethylene bromide, aniline, and anhydrous sodium carbonate is heated to gentle boiling for 5–6 hours; the warm melt is extracted with hot water, and the crude diphenylpiperazine is transformed into its nitroso-derivative by suspending it in cold, concentrated hydrochloric acid and gradually adding a saturated solution of sodium nitrite through a tube reaching to the bottom of the acid. The precipitate, probably the hydrochloride of dinitrosodiphenylpiperazine, is added to a 40% solution of sodium bisulphite and the suspension warmed to about 80° C.; the solution is made strongly alkaline with sodium hydroxide, and concentrated by distillation. The piperazine is finally isolated by distillation of the residue with superheated steam and is absorbed by hydrochloric acid. Traces of piperazine are conveniently detected by means of platinum chloride, picric acid, or bismuth potassium iodide (brilliant garnet crystals) or by the formation of dinitrosopiperazine.—H. W.

*Picramic acid; Modified method for the preparation of* —. G. Egerer. J. Biol. Chem., 1918, 35, 565–566.

Picramic acid is dissolved in alcohol, treated with ammonia, and reduced by hydrogen sulphide, first at 50° C. and then at 0° C. The ammonium picramate is deposited in crystalline form and the free acid is subsequently liberated by treatment with acetic acid, and purified by recrystallisation from boiling water. The yield is 75 to 80% of the theoretical quantity. (See further J. Chem. Soc., 1918, i., 496.)—H. W. B.

*Benzaldehyde and benzoic acid; Use of benzaldehyde sulphite compound as a standard in the quantitative separation and estimation of* —. G. A. Geiger. J. Amer. Chem. Soc., 1918, 40, 1453–1456.

BENZALDEHYDE is unsuitable as a standard as it is too readily oxidised, but the bisulphite compound,  $C_6H_5CHO \cdot NaHSO_3 \cdot 2H_2O$ , fulfils the required conditions. The estimation is carried out as follows: Solutions containing as a maximum 0.5 gm. of benzoic acid and benzaldehyde are made distinctly alkaline with sodium hydroxide and extracted with three portions of ether (each 25 c.c.); the combined ethereal extracts are treated with freshly distilled phenylhydrazine (1.5 grms.) and 10 c.c. of a 10% solution of acetic acid in ether. The solution is evaporated in a current of air; the residue is mixed with water (50 c.c.), and the insoluble benzylidene-phenylhydrazine is collected in a tared Gooch crucible, dried for 2 hours at 70° C. in a vacuum, and weighed. The solution from which the benzaldehyde has been extracted is rendered slightly acid with hydrochloric acid (1:3), extracted with chloroform (25 c.c.), saturated with salt, and shaken three times with chloroform (25 c.c. for each operation). The chloroform is evaporated at the ordinary temperature and the residue is dissolved in neutral 50% alcohol and titrated with sodium hydroxide. If the ethereal solution of benzaldehyde is washed with 25 c.c. of N/5 sodium hydroxide, the quantity of benzoic acid separated is larger, and the accuracy of the method is increased.—H. W.

*Benzoylation of some hydroxyl or amino aromatic compounds.* F. Reverdin. Helv. Chim. Acta, 1918, 1, 205–209.

A MIXTURE of the substance and benzoyl chloride is treated with two or three drops of concentrated sulphuric acid and, if necessary, warmed for a short time on the water-bath. The method is successful with substances which are unaffected by the usual methods of benzoylation. (See also J. Chem. Soc., Dec., 1918.)—C. S.

*Nucleic acids; Determination of the purine bases in — after cleavage without the production of humin.* R. Feulgen. Z. physiol. Chem., 1918, 102, 244–251.

THE nucleic acid is hydrolysed by heating with sodium bisulphite at 160° C. under pressure. On cooling, guanine separates quantitatively from the colourless, humin-free solution, whilst the adenine is estimated in the filtrate by conversion first into the phosphotungstate, then into the silver salt, and finally into the picrate, which is dried and weighed.—H. W. B.

*Histidine and other glyoxaline derivatives; Volumetric estimation of* —. C. L. Lautenschläger. Z. physiol. Chem., 1918, 102, 226–233.

THREE methods are described for the estimation of histidine: (a) by direct titration with a standard silver nitrate solution until a red coloration is no longer observed when a drop is mixed with a drop of an alkaline solution of diazobenzenesulphonic acid, (b) by direct titration with diazobenzenesulphonic acid, using, as an indicator, technical "K" salt (which gives a darker-coloured dye with the diazo-acid than histidine), (c) by adding an excess of the diazo-acid, boiling with alcohol to destroy the excess of acid, and then titrating the stable histidine dye with titanium trichloride. The histidine must in all cases be first separated from other substances, such as tyrosine, which react with diazobenzenesulphonic acid, before the titration is carried out. Methods (a) and (c) yield more accurate results than (b).—H. W. B.

*Vitamins; Pharmacology of the* —. H. F. Uhlmann. Z. Biol., 1918, 63, 457–498.

THE close parallelism between the effects on animal tissues produced by pilocarpine and by vitamin preparations from various sources (oryzan, extracts of oats, nettles, blood, etc.) confirms the author's previous conclusion, *viz.*, that the antineuritic vitamin is a substance physiologically closely related to pilocarpine, which acts on the sympathetic nerve endings and thus exerts a controlling influence on the tonus of the muscles in the organism.—H. W. B.

*Formic, acetic, and lactic acids; Separation of* —. I. Onodera. Ber. Ohara Inst. landw. Forsch., 1917, 1, 231–259.

THE solution containing the three acids is extracted with ether for 4 hrs. in a modified Soxhlet apparatus, the extraction flask containing an excess of N/10 sodium hydroxide solution. The excess of alkali is then neutralised with N/10 phosphoric acid solution (this gives a measure of the total acidity), an excess of phosphoric acid is added, and the mixture steam-distilled at 140°–160° C. for 2 hrs. or until the distillate no longer shows an acid reaction; the distillate is collected in an excess of alkali solution, the latter neutralised with sulphuric acid, and diluted to a definite volume. To determine the formic and lactic acids an aliquot portion of this solution is treated with 1.5 grms. of sodium carbonate and an excess of N/10 permanganate



solution, diluted to 100 c.c., and heated at 100° C. for 35 mins., and the excess of permanganate determined iodometrically. Another portion of the solution is then heated for 1 hour at 100° C. with the addition of 1.5 grms. of sodium carbonate and excess of 3.2% permanganate solution, the excess of the latter is destroyed with hydrogen peroxide, the mixture filtered, the filtrate evaporated, and the oxalic acid formed by the oxidation of the lactic acid is precipitated as calcium oxalate and this is titrated in the usual way with permanganate solution. Each c.c. of N/50 permanganate solution is equivalent to 0.0009 grm. of lactic acid; the formic acid is found by difference. Acetic acid is determined by oxidising a third portion of the solution as described, decomposing the excess of permanganate with hydrogen peroxide, evaporating the filtered liquid, acidifying the residual liquid with sulphuric acid, and extracting it with ether, the extraction flask in this case containing a small quantity of water. A portion of the extract is then diluted to 100 c.c. and distilled, 95 c.c. of distillate being collected and titrated. Under these conditions 84.49% of the acetic acid is found in the distillate.—W. P. S.

*Glycols; Manufacture of* — [and explosives therefrom]. H. Hibbert. Chem. and Met. Eng., 1918, 19, 571—573.

APART from its preparation from alcohol, ethylene-glycol may be obtained in a less pure condition by cracking petroleum vapours in an iron tube at about 650° C., mixing the resulting gases with chlorine, purifying the product by distillation, and treating the distillates with alkali carbonates in a closed vessel. The mixture of glycols thus obtained consists of liquids somewhat less viscous than glycerin, but having even more pronounced hygroscopic properties. When nitrated the glycols yield products which freeze at much lower temperatures than nitroglycerin, and, unlike the latter, do not form an unstable crystalline modification at a low temperature. Hence they are much less dangerous to handle, and should prove of especial value for mining and blasting operations at low temperatures. Experiments on a manufacturing scale have shown that when these products are compounded with nitro-cellulose (for which process no solvent is necessary) a new type of explosive is obtained, which causes much less erosion of gun-barrels than cordite. A solution of sugar in glycol yields, when nitrated, another new type of explosive, which has valuable properties for such purposes as blasting, excavating, etc. The use of glycol is suggested as a substitute for glycerin for moistening tobacco, with the object of obviating the formation of acrolein during smoking. Owing to its pronounced hygroscopic properties it should also be a valuable constituent of ointments and other pharmaceutical preparations, and would probably be an efficient substitute for alcohol as a means of preventing the freezing of water in motor radiators. (See also J. Chem. Soc., Dec., 1918.) —C. A. M.

*Methylamine; Preparation of* — H. I. Jones and R. Wheatley. J. Amer. Chem. Soc., 1918, 40, 1411—1415.

THE results recently published by Werner (this J., 1917, 1147) have led the authors to describe experiments made with the object of studying the effect of reduced pressure on organic reactions in which a gas is evolved. Mixtures of ammonium chloride and formaldehyde were (1) slowly distilled at the ordinary pressure, (2) slowly distilled in a vacuum, (3) heated under a reflux condenser for 8 hours and then distilled at the ordinary pressure, and (4) heated under a reflux condenser for 4½ hours in

a vacuum and then distilled in a vacuum. In method (4) there was a decrease in the amount of ammonium chloride left unchanged, a lowering of the temperature and therefore decrease of the yield of dimethylamine and higher methylated substances, and an increase in the acidity of the distillate, the loss of carbon dioxide, and the weight of methylamine produced. Method (2), merely by removing the volatile products as well as the carbon dioxide, caused a marked increase in the weight of the distillate and the amount of ammonium chloride remaining unchanged; it caused the formation of the lowest percentage of dimethylamine but also lowered markedly the amount of monomethylamine formed, brought about a higher percentage of esterification, and gave a distillate of the highest rotatory power. Method (3) gave the smallest percentage of distillate, the highest percentage of non-distillable residue, the highest percentage of esterification, used up a large amount of ammonium chloride, and gave the highest degree of methylation, and consequently the highest percentage of dimethyl and other more highly methylated derivatives, most of which will not crystallise. The theory of the reaction advanced by Werner (*loc. cit.*) is correct and explains all the observed facts. Monomethylamine is best prepared by method (4). —H. W.

*Allyl group in essential oils; Kobert's phloroglucinol-hydrochloric acid reaction for the detection of* — S. Furukawa. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 513—515.

THE red coloration which certain aromatic compounds give with phloroglucinol and hydrochloric acid is not, as claimed by Kobert, invariably a trustworthy test for the presence of the allyl group in essential oils. In the case of eugenol or safrol, for instance, the characteristic coloration is not obtained until the oils have been kept for at least 6 months at the ordinary temperature. On fractional distillation of such oils under reduced pressure, the first fraction does not give the reaction until after it has been kept for a long time. Moreover, certain compounds of the fatty series, such as citronellol and geraniol, give a red-brown coloration with phloroglucinol and hydrochloric acid, whilst others, such as citronellal and citronellie acid, do not give any coloration.—C. A. M.

*Citronella oils* S. Furukawa. Kōgyō-Kwagaku-Zasshi (J. Chem. Ind., Tokyo), 1918, 21, 515—525.

A SAMPLE of citronella oil from Formosa had the following characters:—Sp. gr., 0.9601; total geraniol, 22.15%; and total citronellal, 11.05%. Formosa oil is thus of less value for perfumery than Java or Ceylon citronella oil. The composition of Formosa oil somewhat resembles that of the oil of "ogarugaya," a variety of *Andropogon nardus*, L., growing in Japan. A Java oil was found to contain two phenols (eugenol and possibly chavicol), and three acids, one of which was identified as citronellie acid.—C. A. M.

*Chlorinated toluene.* Lubs and Clark. See III.

*Toluene-o- and -p-sulphonic acids.* McKie. See III.

*Nitrogen determination.* Renaud. See XXIII.

*Determining carbon.* Renaud. See XXIII.

## PATENTS.

*Alcohols; Process of producing* —. E. C. R. Marks, London. From E. I. du Pont de Nemours and Co., Wilmington, Del., U.S.A. Eng. Pat. 119,249, 9.6.17. (Appl. 8259/17.)

A HALOGENATED hydrocarbon, e.g. pentyl or hexyl chloride, is treated with a salt of a non-corrosive fatty acid of high boiling point, e.g. sodium stearate, to form the ester of an alcohol, which is subsequently recovered by hydrolysing the ester. Stearic acid and solid caustic soda are heated in a jacketed vessel at 200°–240° C. and when the water has been expelled, a mixture of pentyl and hexyl chlorides is introduced at the bottom of the vessel. A portion of the halogenated hydrocarbon reacts with the sodium stearate, another portion is converted into olefines, and part distils unchanged. The volatile products are condensed and separated, for which purpose a reflux cooler maintained at a suitable temperature to return the volatile chlorides to the vessel may be provided. The stearic ester in the vessel is cooled to about 150° C., treated with sufficient caustic soda to replace that which has reacted with the chlorides, heated again to 200°–240° C. and the resulting alcohol is distilled off in the presence of a little water or steam.—J. F. B.

*Dimethyl sulphate; Process for the production of* —. E. J. Boake, London, and T. H. Durrans, Oxford. Eng. Pat. 119,250, 20.6.17. (Appl. 8862/17.)

METHYL alcohol (2 mols.) is treated with sulphur dioxide and chlorine (approximately 1 mol. of each), the sulphur dioxide being preferably in slight excess. The gases may be passed either simultaneously or successively; in the former case the necessary excess of sulphur dioxide may be added at the start. Methyl chloride is a gaseous product of the reaction. The dimethyl sulphate is isolated and purified by distillation under reduced pressure; the yield is nearly equal to the theoretical quantity.—J. F. B.

*Secale cornutum [ergot]; Preparation from* —. O. Imray, London. From Soc. Chem. Ind. in Basle, Switzerland. Eng. Pat. 119,287, 27.9.17. (Appl. 13,964/17.)

ERGOT (*Secale cornutum*) after treatment for removal of oil, e.g. with petroleum ether, is extracted with cold water and then with alcohol of about 80% strength containing about 0.1% of a volatile acid, such as formic or acetic acid. After removal of the alcohol and excess acid from the alcoholic extract by distillation *in vacuo*, water being added during the distillation, the extracts are mixed and further concentrated to a liquid of definite strength or to a yellowish-brown hygroscopic powder. Inorganic constituents are removed by treatment with alcohol. The product is soluble in water and suitable for injection.—B. V. S.

*Substances which arrest and cause coagulation of the blood; Separation of* — from animal organs. Luitpold-Werk, Chem.-pharm. Fabr., Munich. Ger. Pat. 306,805, 14.7.16.

A SUBSTANCE which causes coagulation of the blood is prepared by extracting finely divided animal organs with an alkaline aqueous solution and then treating the solution with an acid in the presence of phenol or with salicylic acid; on the other hand, if the organs (or the residue after treatment with alkali) are extracted with dilute acid and the solution neutralised, a substance is obtained which arrests blood coagulation.—L. A. C.

*Ovaries, corpus luteum, or placenta; Manufacture of a physiologically active substance from* —. S. Fränkel and E. Herrmann, Vienna. Ger. Pat. 306,906, 13.1.14. Int. Conv., 9.1.14. Addition to Ger. Pat. 297,392.

THE tissue is extracted with acidulated water, proteins are separated, and the solution concentrated *in vacuo*. The remainder of the proteins, together with some of the phosphatides, are precipitated with alcohol, and the concentrated solution is extracted with ether or a similar solvent. After precipitation of the phosphatides and evaporation of the solvent, the residue is treated with water, cooled and freed from fats, again concentrated, and extracted with ether or a similar solvent. As an alternative method of preparation, the tissue may be dehydrated, extracted with a solvent which does not dissolve water, and the phosphatides precipitated from the solution. After evaporation of the solvent, the residue is treated as before. The product obtained by evaporating the final extract is a brown oil containing cholesterol; if the oil is cooled the cholesterol and its esters crystallise out and can be separated, leaving the pure product.—L. A. C.

*Oxidising aromatic hydrocarbons.* Eng. Pats. 119,517 and 119,518. See III.

*Removing substances from eggs.* U.S. Pat. 1,277,727. See XIXa.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

## PATENT.

*[Photomechanical] printing plates; Process for preparing* —. A. J. Hain, Lakewood, Ohio, U.S.A. Eng. Pat. 119,328, 7.11.17. (Appl. 16,254/17.)

THE plate to be etched is provided with a layer of regularly spaced dots of the minimum size required for the high lights of the finished printing plate, which dots act directly or indirectly as a resist and which in subsequent processes can be caused to expand laterally to correspond with the photographic image impressed on the plate. For example, a zinc plate coated with an acid resist, such as bitumen, is provided with a sensitised film, e.g., of bichromated gelatin. This is then exposed behind a suitable screen plate and the unexposed portions removed in the usual way leaving a layer of regularly spaced isolated dots. These are impregnated with light-sensitive silver bromide by treatment with ammonia, potassium bromide, and silver nitrate, and the intervening spaces are filled in with gelatin. The plate is then exposed through a negative, developed, fixed, and washed, and treated with a mixed potassium bichromate-ferricyanide-bromide solution; by standing about 15 mins. after this treatment the gelatin round each dot becomes hardened to an extent, in area, proportional to the amount of silver, i.e., to the exposure through the negative. The remaining soluble portions of the film are washed away, the acid resist removed from the same areas by a suitable solvent, and the plate etched as usual. Other methods of obtaining the regularly spaced light-sensitive dots may be used and the method of enlarging them laterally may be varied; a print contact method, a physical development method, and a lead intensification method are described.

—B. V. S.



## XXII.—EXPLOSIVES; MATCHES.

*Cellulose and nitrocelluloses.* A. Hervé. *Monit. Scient.*, 1918, 62, 193—196.

NITROCELLULOSE prepared for the manufacture of celluloid contains small proportions of sulphuric acid combined in the form of esters and not removable by simple washing. This sulphuric acid is retained as a relatively stable combination in the presence of bases, when it reacts as an ester salt, but is progressively eliminated by hydrolysis on boiling with dilute mineral acids. For the determination of the combined sulphuric acid, 5 grms. of the fully washed and dry nitrocellulose is boiled under a reflux condenser for 8 hours with 250—300 c.c. of water containing 1 c.c. of strong hydrochloric acid (sp. gr. 1.18). The nitrocellulose is filtered off, the filtrate and washings are concentrated to 150 c.c. and the sulphuric acid precipitated as barium sulphate. The percentage of sulphuric acid fixed varies with the proportion of that acid used in the nitrating mixture. With a constant proportion of about 19% of water, the following results were obtained:—ratio of  $\text{HNO}_3$  to  $\text{H}_2\text{SO}_4$  1:1, 0.0505% sulphuric acid fixed in the nitrocellulose; ratio 1:2, 0.2025%; ratio 1:3, 0.4290%; ratio 1:4, 1.34%; ratio 1:5, 1.81%. This combined sulphuric acid has a strongly depressing influence on the heat test stability of the nitrocellulose and of the celluloid prepared from it, but it is not the only factor. Compounds of low degrees of nitration or of oxycellulose or hydrocellulose also affect stability; these are most easily eliminated by boiling with alkaline water, so that effective stabilisation demands both acid treatment for the sulphuric esters and basic treatment for the inferior nitrated products. This combination of conditions may be produced by boiling the nitrocellulose with a readily dissociable salt, such as 0.1—1.0% of its weight of magnesium chloride, for about 12 hours. Satisfactory products are thus obtained which withstand the Abel heat test for 60 minutes.—J. F. B.

*Mercury fulminate: Detection and determination of free mercury in —.* P. Nicolardot and J. Boudet. *Ann. Chim. Analyt.*, 1918, 23, 206—207.

ONE gm. of the sample is agitated with 100 c.c. of water containing 5 grms. of ammonium thiosulphate; the fulminate dissolves rapidly whilst the free mercury which may be present remains as a grey powder or as small metallic globules. The mercury is collected on a tared filter, washed with water, then with alcohol and ether, dried over potassium hydroxide, and weighed. The quantity of free mercury present is about 0.1% when the fulminate is made in the usual way, but increases if larger proportions of mercury are used in the preparation.—W. P. S.

[Explosives from] glycols. Hibbert. See XX.

## PATENT.

*Explosive.* O. B. Carlson and E. F. Otterdahl. *Monsbo, Sweden.* U.S. Pat. 1,277,043, 27.8.18. Appl., 8.12.15.

The explosive contains ammonium perchlorate, ferrosilicon, wood-meal, a combustible, consisting of fractions from rock-oil rich in naphthenes and having a boiling point above  $250^\circ\text{C}$ ., and a substance obtained by gelatinising liquid trinitrotoluene with nitrocellulose and sodium nitrate.

—T. St.

## XXIII.—ANALYSIS.

*Calibrating pipettes; Simple method of rapidly and accurately —.* S. English. *J. Soc. Glass Tech.*, 1918, 2, 216—219.

ONE neck of a Woulfe's bottle is connected to an elevated reservoir of water, and an outlet near the bottom of the bottle is fitted with a three-way tap leading to a pipette with a pear-shaped bulb of about 0.5 c.c. less capacity than the pipette to be calibrated and a capillary stem above the bulb to bring the capacity up to the required figure. The lower pipette is calibrated from a standard pipette fitted into the other neck of the Woulfe's bottle. The standard pipette is filled with water from the reservoir and the surplus water is removed from its upper extremity by means of blotting paper. The three-way tap is then opened so as to allow the water from the standard pipette to run back into the bottle and force out an equal quantity into the pear-shaped pipette, and after a prearranged time for drainage, the height of the water in the capillary tube is marked thereon. The pear-shaped pipette is then emptied. The standard pipette is then replaced by the pipette to be calibrated, which is filled and emptied in a similar manner, except that when the water has risen to the mark on the capillary tube (after allowing the requisite time for drainage) the level of the water in the higher pipette is marked thereon. The maximum error found when comparing this method with the customary one in which the water run out of the pipette is weighed was 0.027 c.c. in a series of twelve 50 c.c. pipettes, but the new method only requires about 2 minutes per pipette.

—A. B. S.

*Thymolsulphophthalein; Use of — as indicator in acidimetric titrations.* A. B. Clark and H. A. Lubs. *J. Amer. Chem. Soc.*, 1918, 40, 1443—1448.

THYMOLSULPHOPHTHALEIN, an indicator having two useful working ranges (see this J., 1916, 980), can be very successfully employed in many differential acidimetric and alkalimetric titrations. To illustrate the general method of employment, a few typical titrations are described (mixtures of benzoic and hydrochloric acids, acetic and hydrochloric acids, acetic and sulphuric acids, titration of aniline by hydrochloric or sulphuric acid). Any acid with a dissociation equal to or weaker than that of acetic acid may be differentially titrated in the presence of hydrochloric acid, provided that the weaker acid is completely neutralised when the alkaline colour change of the indicator occurs. The use of proper colour screens or, preferably, of a colorimeter or spectrophotometer greatly increases the accuracy of differential titrations but is not absolutely necessary.—H. W.

*Copper; Separation of traces of — from solution.* J. E. Saul and D. Crawford. *Analyst*, 1918, 43, 348.

WATER containing as little as 0.002% of copper gives a voluminous yellow precipitate when treated with 0.1% of quinosol (the potassium salt of 8-hydroxy-quinoline-5-sulphonic acid) and allowed to stand for about 18 hrs. The precipitate is soluble in dilute hydrochloric acid. Other metals in very dilute solution do not give a reaction, but a precipitate is obtained when a concentrated solution of a mercuric salt is treated with the reagent.—W. P. S.

*Zinc dust [for analysis].* O. Binder. *Chem.-Zeit.*, 1918, 42, 458.

THE author has found that zinc dust contains appreciable amounts of chlorine, for which allowance must be made in certain analytical processes.

*Zinc; Volumetric estimation of* —. R. Howden.  
Chem. News, 1918, 117, 322.

Zinc when present as chloride in solutions which contain no heavy metal or ammonium salts may be estimated by first boiling to remove excess of free acid, exactly neutralising with N/10 sodium hydroxide using methyl orange as indicator, and then titrating the acid combined with the zinc with N/10 sodium hydroxide, using phenolphthalein as indicator. Towards the end of the reaction the solution is boiled and the end-point taken as that point at which the pink colour is not destroyed by boiling.—J. F. S.

*Colloidal hydroxides; Adsorption of* —. K. Scheringa. Pharm. Weekblad, 1918, 55, 1070—1074.

The adsorption of ordinary salts by metallic hydroxides is not of much practical significance in analysis. In separating iron, it is not desirable to have a great excess of alkali.—A. J. W.

*Barium and strontium; Separation and determination of* —. F. A. Gooch and M. A. Soderman. Amer. J. Sci., 1918, 46, 538—540.

Strontium chloride is soluble in a mixture of hydrochloric acid and ether, whilst barium chloride is precipitated. About 0.5 gm. of the mixed chlorides, containing not more than 0.3 gm. of strontium chloride, is dissolved by warming with the least possible quantity of water, and the cold saturated solution is treated with a mixture of concentrated hydrochloric acid, 4, and ether, 1 vol., added slowly, with stirring. The precipitated barium chloride is collected on an asbestos filter, washed with the acid-ether mixture, dried at 150° C., and weighed. The error does not exceed 0.0004 gm. —W. P. S.

*Iron; Titanium chloride as a reducing agent in the determination of* — by titration with permanganate in hydrochloric acid solution. L. Brandt. Chem.-Zeit., 1918, 42, 432—434, 450—451.

Titanium trichloride solution readily reduces ferric salts in the cold (Knecht and Hibbert, this J., 1903, 232, 762), the reduction being made either by adding the exact quantity of titanium trichloride, or by adding more than this and then removing the excess by the addition of copper sulphate. The ferrous salt is then titrated in the usual way with permanganate solution. Platinum chloride, potassium bichromate, and arsenic acid do not interfere with the method, but arsenious acid should not be present. When stannous chloride is used for reducing the ferric salt, copper sulphate may be employed in place of mercuric chloride for removing the excess of stannous chloride added.—W. P. S.

*Gasometric [nitrogen] determinations; Simplified* —. A. Renaud. J. Pharm. Chim., 1918, 18, 104—106.

FREQUENT standardisations with pure urea may be avoided in the determination of nitrogen in urine by using a comparison tube exactly similar to the nitrometer. This comparison tube contains a volume of air equal to the volume of nitrogen liberated in the nitrometer from a known quantity of urea; the volume of air then serves as a standard in subsequent determinations. It is necessary to note the pressure and temperature at the time the air is admitted to the comparison tube. (See also J. Chem. Soc., Nov., 1918.)—W. P. S.

*Carbon; Determination of total* — in various biological substances. A. Renaud. J. Pharm. Chim., 1918, 18, 106—108.

THE substance (e.g., urine) is heated in a flask with potassium bichromate and sulphuric acid, the gases evolved are passed into ammoniacal calcium

chloride solution, and the resulting calcium carbonate is separated and determined volumetrically. After the absorption of the carbon dioxide it is usually necessary to heat the ammoniacal solution on a water-bath in order to obtain the calcium carbonate in a condition suitable for titration. If barium chloride is used in place of calcium chloride, the resulting barium carbonate may be collected on a filter at once, converted into barium sulphate, and weighed. (See also J. Chem. Soc., Nov., 1918.)

—W. P. S.

*Magnetic susceptibility of mixtures of liquids.* Alpheus W. Smith and Alva W. Smith. J. Amer. Chem. Soc., 1918, 40, 1218—1224.

THE magnetic susceptibility of liquid mixtures is found to satisfy the simple mixture rule within the limits of experimental errors. Data are recorded for mixtures of acetone and water, acetic acid and water, acetic acid and benzene, acetone and ethyl alcohol, and it is suggested that the measurement of the magnetic susceptibility may conveniently be used in the determination of the composition of binary liquid mixtures.—H. M. D.

*Motor fuels.* Moore. See IIa.

*Chlorinated toluene.* Lubs and Clark. See III.

*Toluene-o- and -p-sulphonic acids.* McKie. See III.

*Sulphur dyestuffs.* Zänker and Schnabel. See IV.

*Celluloid analysis.* Hervé. See V.

*Nitrites and nitrates.* Oelsner. See VII.

*Determining sulphates.* Vansteenbergh and Bauzil. See VII.

*Gas purification masses.* Müller. See VII.

*Radium emanation.* Underwood and Schlundt. See VII.

*Chemical glassware.* Cauwood and Turner. See VIII.

*Refractory materials.* See VIII.

*Calcium soaps.* Sahn and Prager. See XII.

*Fatty acids in soaps.* Bosshard and Comte. See XII.

*Methyl alcohol in ethyl alcohol.* Toplis. See XVIII.

*Milk in pastry.* Grossfeld. See XIXa.

*Caffeine in coffee.* Vautier. See XIXa.

*Hypochlorites in water.* Ling. See XIXb.

*Hypochlorite solutions.* Cullen and Austin. See XIXb.

*Benzaldehyde and benzoic acid.* Geiger. See XX.

*Nucleic acids.* Feulgen. See XX.

*Determining histidine, etc.* Lautenschläger. See XX.

*Separating formic, acetic, and lactic acids.* Onodera. See XX.

*Detecting allyl group.* Furukawa. See XX.

*Mercury fulminate.* Nicolardot and Boudet. See XXII.



## PATENTS.

*Moisture; Means for detecting or measuring* —.

C. A. Parsons and A. H. Law, Newcastle-on-Tyne. Eng. Pat. 119,533, 4.10.7. (Appl. 14,352/17.)

A LAYER of electric insulating material is wound on a metal cylinder, and an open coil of wire is wound on the insulation. The insulation resistance through the material from the wire to the cylinder is measured by any suitable means, and its variation due to the variable humidity of the air is a measure of that humidity. In a modification the terminal elements consist of two coils of wire wound on the insulation, and the surface resistance between them is measured. The method is suitable for testing the air used for cooling dynamo-electric machinery, the insulation employed in the test being similar to that used in the machinery.

—W. F. F.

*Glassware.* Eng. Pat. 119,297. See VIII.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Antonoff. Catalysis. 18,020. Nov. 4.

August. Treating liquids or solids for heating, calcining, vaporising, or decomposing them. 17,799. Oct. 31.

Conradi, Crawford, and Lyall. Apparatus for filtering, scrubbing, or conditioning air, gases, and liquids. 18,290. Nov. 8.

Diebold. Manufacture of dispersions or suspensions of matter. 18,312. Nov. 8.

Dressler Tunnel Ovens, Ltd., and Williamson. Drying or heating apparatus. 17,895. Nov. 1.

Gourlay and Sands. Apparatus for separating immiscible liquids. 17,520. Oct. 28.

Johnston. Dehydrators for drying vegetable, animal, and mineral substances. 17,530 and 18,346. Oct. 28 and Nov. 8.

Mann. Drying, concentrating, or evaporating liquids etc. 18,017. Nov. 4.

Soddy. Improving or restoring absorptive power of charcoal for gases. 17,583. Oct. 28.

Stubley. Separating machine. 17,866. Nov. 1.

Tansley. Apparatus for cooling gases. 17,821. Oct. 31.

Titan Co. Aktieselskabet. Freeing liquids from particles suspended therein. 17,811. Oct. 31. (Norway, 12.12.17.)

Torrance. Disk etc. grinding-mills. 18,127. Nov. 5.

Urruty. 18,343. See XIX.

Welch. Method of concentrating solutions. 17,696. Oct. 30.

West. Kilns. 18,228. Nov. 7.

Winkel. Filtering-tanks. 18,008. Nov. 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,347 (1917). Blom and Gledie. Machines for drying liquids. (110,753.) Nov. 6.

15,974 (1917). Smallwood. Furnaces. (120,259.) Nov. 13.

17,118 (1917.) Price and Sugg. Drying apparatus. (120,283.) Nov. 13.

218 (1918). Hilton and Willis. Apparatus for bringing gases and liquids into intimate contact. (120,304.) Nov. 13.

1154 (1918). Blacker. See VII.

1171 (1918). Soc. d'Exploit. de Proc. Evapora-

toires Système Prache et Bouillon. Evaporating and crystallising apparatus especially adapted for obtaining large crystals. (112,943.) Nov. 6.

2564 and 10,231 (1918). Brownlow. Centrifugal separators. (120,319.) Nov. 13.

3997 (1918). Kilburn (Sulzer frères). See II.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Aitches, Ltd., and Fabini. Incandescent gas mantles, and manufacture thereof. 18,248. Nov. 7.

Baines. Composite fuel. 17,557. Oct. 28.

Barbet et Fils et Cie. Removing tar from products of distillation of wood, coal, etc. 18,026. Nov. 4. (Fr., 3.11.17.)

Bataafsche Petroleum Maatschappij. Rectification of hydrocarbons. 17,741 and 18,270. Oct. 30 and Nov. 7. (Holland, 21.2 and 1.3.18.)

Burnett. Apparatus for washing coal etc. 17,873. Nov. 1.

Burnett. Extracting water etc. from washed coal etc. 17,874. Nov. 1.

Coxon and Williams. Renewal of filaments in metal filament electric lamps. 18,370. Nov. 9.

Davidson. Manufacture of peat fuel from damp peat. 17,843. Oct. 31.

Dellwick, and Techno-Chemical Laboratories, Ltd. Combustion of fuel. 18,392. Nov. 9.

Ellison, Wilkinson, and Woodhouse. Retorts for continuous carbonisation of coal etc. 17,782. Oct. 31.

Griggs. Hydrogen generators. 18,382, 18,383, 18,384. Nov. 9.

Kirke. Burning gaseous fuel. 17,918. Nov. 1.

Michel and Raskin. Manufacture of briquettes from flue dust, sawdust, etc. 17,945 and 17,946. Nov. 2.

Rinman. Process of dry distillation with strong bases of mixtures of carbon acids and mixtures thereof with substances containing lignin. 17,718. Oct. 30. (Sweden, 8.11.17.)

Sleeman. 18,337. See XIX.

Smith. Briquettes and method of making same. 17,693. Oct. 30. (U.S., 15.11.16.)

Soddy. 17,583. See I.

Wade (Le Petit). Production of fuels. 18,039. Nov. 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,335 (1916). Chamier, Craig, and Moeller. Manufacture of power gas and recovery of hydrocarbon oils. (120,051.) Nov. 6.

10,973 (1917). Wellington. Destructive distillation of carbonaceous substances. (120,223.) Nov. 13.

13,954 (1917). Day. Apparatus for producing hydrocarbons. (120,230.) Nov. 13.

15,868 (1917). Dubbs. Treatment of petroleum and other hydrocarbons to produce hydrocarbons having lower boiling points. (112,010.) Nov. 6.

15,885 (1917). Hutchins. Manufacture of carbon blocks. (120,255.) Nov. 13.

16,898 (1917). Day, Macdonald, and Day. Formation of hydrocarbons having low boiling points from hydrocarbons having high boiling points. (113,264.) Nov. 13.

16,899 (1917). Day and Day. Vaporising hydrocarbons and producing hydrocarbons having low boiling points from hydrocarbons having high boiling points. (119,440.) Nov. 13.

16,900 (1917). Day and Day. Treatment and refining of hydrocarbon materials. (119,441.) Nov. 6.

18,578 (1917). Key. Apparatus for distilling or carbonising coal and cannel. (120,139.) Nov. 6.

3997 (1918). Kilburn (Sulzer frères). Cooling hot residues from distillation processes. (120,329.) Nov. 13.

## III.—TAR AND TAR PRODUCTS.

## APPLICATIONS.

Barbet et Fils et Cie. 18,026. *See* II.  
 Barrett Co. Recovery of pyridine bases. 17,663.  
 Oct. 29. (U.S., 17,118.)  
 Batnafsche Petroleum Maatschappij. 17,741 and  
 18,270. *See* II.

## IV.—COLOURING MATTERS AND DYES.

## APPLICATION.

A'Brassard and Crawford, and Singer. Manu-  
 facturing colouring matters. 18,016. Nov. 4.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

## APPLICATIONS.

Dawson and Dyson. Preparing textile fibres for  
 spinning. 17,606. Oct. 29.  
 Gray and Jardine. Preparation of bamboo in  
 paper-making. 17,711. Oct. 30.  
 Spicer (Shirlow). Impregnating and drying  
 strawboard, paper, etc. containers. 17,902 and  
 17,906. Nov. 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

15,467 (1917). Spicer (Wells). Production of  
 paper pulp. (120,086.) Nov. 6.  
 298 (1918). Blyth. Paper-making machines.  
 (120,147.) Nov. 6.  
 2046 (1918). Horrox. Substitute for cardboard,  
 strawboard, etc. (120,157.) Nov. 6.

VI.—BLEACHING; DYEING; PRINTING;  
FINISHING.

## APPLICATIONS.

Bright and Bros., and Evans. Machines for boil-  
 ing, bleaching, dyeing, sizing, etc. yarns or fabrics.  
 17,620. Oct. 29.  
 Poulson. Rendering textile fabrics etc. water-  
 proof and fireproof. 17,683. Oct. 30.

## COMPLETE SPECIFICATIONS ACCEPTED.

1137 (1918). Calico Printers' Assoc., Ashton, and  
 Nelson. Printing cotton fabrics or yarns. (120,152.)  
 Nov. 6.  
 10,299 (1918). Calico Printers' Assoc., Fothergill,  
 and Wilson. Treatment of insoluble natural gums  
 for the production of thickenings suitable for  
 calico and other textile printing and finishing etc.  
 (120,183.) Nov. 6.

VII.—ACIDS; ALKALIS; SALTS; NON-  
METALLIC ELEMENTS.

## APPLICATIONS.

Aanensen. Manufacture of ammonium per-  
 chlorate. 17,700. Oct. 30. (Norway, 22,12,17.)  
 British Dyes, Ltd., Davidson, and Turner. In-  
 creasing production and efficiency in contact plants  
 in manufacture of sulphuric acid. 17,869. Nov. 1.  
 British Dyes, Ltd., Davidson, and Turner. Manu-  
 facture of pure sulphur dioxide. 17,870. Nov. 1.  
 British Dyes, Ltd., Davidson, and Turner. Pro-  
 duction of nitrous anhydride and nitrites from  
 nitrates or waste nitric acid. 17,871. Nov. 1.  
 British Dyes, Ltd., Davidson, and Turner. Con-  
 tinuous process for production of high strengths of  
 oleum. 17,872. Nov. 1.  
 Cain, Couch, and Powell. Manufacture of alkali  
 permanganate. 17,582. Oct. 28.  
 Calvert. Production of cyanide. 18,131. Nov. 5.  
 Craig, and Spence and Sons. Recovery of sul-  
 phur. 17,691. Oct. 30.  
 Craig. Recovering potash etc. from blast fur-  
 naces. 17,974. Nov. 4.

Delage. Manufacture of magnesite. 18,335.  
 Nov. 8. (Fr., 8,11,17.)

Grayson. Manufacture of sulphur dioxide.  
 17,813. Oct. 31.

Grayson. Manufacture of sodium nitrite. 17,814.  
 Oct. 31.

Griggs. 18,382, 18,383, 18,384. *See* II.

Heberlein. Electrolysing solution of nickel salt.  
 17,650. Oct. 29.

Imperial Trust for the Encouragement of Scien-  
 tific and Industrial Research, and Stannard.  
 Recovery of tungstic acid from ores. 18,206.  
 Nov. 6.

Maconochie and de Ros. Oxidation of tin and  
 production of tin oxide, also treatment of zinc  
 and zinc-containing materials for production of zinc  
 oxide. 17,749. Oct. 30.

Norsk Alkali Aktieselskabet. Electrolysis of  
 chlorides of alkaline metals. 18,135. Nov. 5.  
 (Norway, 13,12,17.)

Ridoni, and Soc. Talco e Grafite Val Chisone.  
 Purification of natural graphites. 18,330. Nov. 8.

Sheard. Producing neutral sulphate of ammonia.  
 18,362. Nov. 9.

## COMPLETE SPECIFICATIONS ACCEPTED.

10,780 (1917). Statham. Light precipitated chalk  
 and processes of making same. (120,219.) Nov. 13.  
 12,289 (1917). Huber and Reath. *See* IX.  
 1154 (1918). Blacker. Apparatus for breaking up  
 or pulverising caustic soda and the like. (120,309.)  
 Nov. 13.

## VIII.—GLASS; CERAMICS.

## COMPLETE SPECIFICATION ACCEPTED.

5764 (1917). Glossop and Sherlock. Continuous  
 kilns for burning bricks, terra cotta, etc. (120,052.)  
 Nov. 6.

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

Anderton and Coates. Cement. 18,379. Nov. 9.  
 Bährnhelm. Manufacture of cement. 17,717.  
 Oct. 30. (Sweden, 24,12,17.)  
 Cullis. Concrete etc. 18,190. Nov. 6.  
 Harland and Wolff, and Marks. Concrete.  
 17,901. Nov. 1.

## COMPLETE SPECIFICATIONS ACCEPTED.

5764 (1917). Glossop and Sherlock. *See* VIII.  
 12,289 (1917). Huber and Reath. Producing  
 cement and recovering potassium compounds.  
 (110,540.) Nov. 6.

X.—METALS; METALLURGY, INCLUDING  
ELECTRO-METALLURGY.

## APPLICATIONS.

Bloxam (Stabilimenti Blak—Ing. A. Pouchain).  
 Tin-soldering and tin-coating aluminium and  
 aluminium alloys. 17,739. Oct. 30.  
 Bloxam (Stabilimenti Blak—Ing. A. Pouchain).  
 Aluminium alloys. 17,743. Oct. 30.  
 Coxon and Williams. 18,370. *See* II.  
 Craig. 17,974. *See* VII.  
 Hall, and Rolls Royce, Ltd. Aluminium alloy.  
 17,579. Oct. 28.  
 Imperial Trust, and Stannard. 18,206. *See* VII.  
 Ligotti. Rotary converters. 18,047. Nov. 4.  
 Maconochie and de Ros. 17,749. *See* VII.  
 Marks (Luckenbach Processes, Inc.). Mineral  
 selective and frothing agent, and processes of pro-  
 ducing and using the same. 18,028 and 18,029.  
 Nov. 4.  
 Readett. Method of electrically treating steel  
 etc. and hardening steel etc. 17,932. Nov. 2.



Sherwood, and Vickers, Ltd. Surface-hardening of steel and steel articles. 17,903. Nov. 1.  
Wigley. Crucible furnaces. 18,304. Nov. 8.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,863 (1917). Basset. Treatment of ores in the blast furnace. (109,452.) Nov. 13.  
15,867 (1917). Allen. Processes for coating metal. (113,602.) Nov. 6.  
16,159 (1917). Smallwood. Furnaces for annealing, hardening, or like heat treatment of metals etc. (120,266.) Nov. 13.  
16,585 (1917). Wellman, Seaver, and Head, and Smith. Steel-making furnaces or mixers and the like. (120,114.) Nov. 6.  
17,884 (1917). Penhale and Treloar. Treatment of arsenical and antimonial gold ores. (120,287.) Nov. 13.

### XI.—ELECTRO-CHEMISTRY.

#### APPLICATIONS.

Burgess Battery Co. Manufacture of electric dry batteries. 17,754. Oct. 30. (U.S., 20,10,17.)  
Garbett. Electrodes. 18,339. Nov. 8.  
Heberlein. 17,650. *See* VII.  
Kaplan. Galvanic cells. 17,962 and 17,972. Nov. 2.  
Kent. Electric furnaces. 17,543. Oct. 28.  
Norsk Alkali Aktieselskabet. 18,135. *See* VII.

### XII.—FATS; OILS; WAXES.

#### APPLICATION.

Schuck, and Superior Oil and Process Co. Treatment of solid fatty materials. 18,032. Nov. 4.

#### COMPLETE SPECIFICATION ACCEPTED.

1412 (1918). Winters. Apparatus for extracting oil from seeds, beans, nuts, offal, etc. (120,156.) Nov. 6.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

Kidd, and Wilkinson, Heywood, and Clark. Formation of pigments etc. 17,726. Oct. 30.  
Köhler. Production of shellac substitute. 18,253. Nov. 7. (Sweden, 23,10,17.)  
MacKenzie. Dissolving and breaking down shellac for hat manufacture etc. 17,592. Oct. 28. (Australia, 11,6,18.)  
Nicholson. Dope for aeroplanes. 18,373. Nov. 9.

#### COMPLETE SPECIFICATION ACCEPTED.

10,299 (1918). Calico Printers' Assoc., and others. *See* VI.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### APPLICATION.

North British Rubber Co. (Goodyear Tyre and Rubber Co.). Manufacture of caoutchouc substances. 17,968. Nov. 2.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Devonshire and Ford. Manufacture of plastic compositions from vegetable ivory etc. 18,140. Nov. 5.  
Kay and Platt. Tanning hides etc. 18,305. Nov. 8.  
Lansdown and Magnus. Treatment and utilisation of scrap and waste leather. 17,629. Oct. 29.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

Dederich. Manufacture of fertilising material. 17,851. Oct. 31.

#### COMPLETE SPECIFICATION ACCEPTED.

17,900 (1917). Truffaut. Fertiliser and insecticide. (120,288.) Nov. 13.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATION.

Clark, Clark, and Hood. Decolorising and purifying saccharine materials. 18,042. Nov. 4.

#### COMPLETE SPECIFICATIONS ACCEPTED.

10,629 (1917). Simpson and Lyle. Means for treating sugar liquors. (120,055.) Nov. 6.  
10,299 (1918). Calico Printers' Assoc., and others. *See* VI.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATION.

Greville. Malt products. 18,001. Nov. 4.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Albanese. Treatment of milk. 18,245. Nov. 7.  
Greville. Treatment of flour and production of baking powder, self-raising flour, etc. 17,988. Nov. 4.  
Grossmann. Treatment of sewage sludge. 17,524. Oct. 28.  
Sleeman. Liquor for potable or power purposes and method of producing same. 18,337. Nov. 8.  
Urruty. Disinfectant for cleaning condensers and boilers. 18,343. Nov. 8. (Fr., 20,12,17.)  
Watkins. Butter substitute. 17,633. Oct. 29.

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,828 (1917). Kellogg and Kazmann. Food beverage and process of producing same. (120,121.) Nov. 6.  
16,830 (1917). Kellogg and Kazmann. Food beverage and process of producing same. (120,279.) Nov. 13.  
18,524 and 18,525 (1917). Uecke. Processes and apparatus for softening water. (120,293 and 120,294.) Nov. 13.  
5060 (1918). Makin. Foodstuffs and the manufacture thereof. (120,166.) Nov. 6.  
9224 (1918). De Grouseau and Viconge. Treatment of cocoa and waste products of chocolate manufacture. (120,178.) Nov. 6.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,395 (1917). Tcherniac. Manufacture of oxyalkyl ethers of *p*-acetaminophenol or substitution products thereof. (120,081.) Nov. 6.  
4684 (1918). Bloxam (Elektrizitätswerk Lonza). Manufacture of ethyl alcohol from acetaldehyde. (120,163.) Nov. 6.

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATION.

Aronson. Matches. 17,848. Oct. 31.

### XXIII.—ANALYSIS

#### APPLICATION.

Aktiebolaget Ingeniörsfirma F. Egnell. Registering mechanism for gas-analysing apparatus. 17,912. Nov. 1. (Sweden, 8,11,17.)

## I. GENERAL; PLANT; MACHINERY.

## PATENTS.

*Drying air.* F. O. Rynoe, London. Eng. Pat. 119,590, 8.1.18. Appl. 462/18.

COMPRESSED air is delivered into the top of a cylindrical chamber having a conical bottom. A loaded air-escape valve is provided near the top of the chamber, and a long vertical drain pipe extends downwards from the apex of the conical bottom and terminates in another loaded valve. Owing to the compression, the air deposits its moisture, and this passes down the drain pipe and is discharged through the valve, which opens only at a pressure equal to that of the air-escape valve plus the pressure due to the column of liquid in the drain pipe. The dried air escapes from the upper loaded valve.—W. H. C.

*Dryer.* A. W. Lissauer, New York. U.S. Pat. 1,277,961, 3.9.18. Appl., 14.12.17.

THE air inlet and outlet passages, which are connected with chambers in the dryer, have varying cross-sectional areas. A fan supplies preheated air to the inlet passages at their greatest cross-section and withdraws a controlled proportion of air from the outlet passages at their greatest cross-section.—W. H. C.

*Dryer; Rotary* —. W. O. Arzinger, Nashville, Tenn. U.S. Pat. 1,278,288, 10.9.18. Appl., 1.11.17.

THE apparatus consists of an insulated cylinder provided at the inlet end with electrical heating units, which project inwards from the walls of the casing and propel the material forward. In the other portion of the cylinder the material is propelled forward by means of a series of vanes inclined angularly to the longitudinal axis of the cylinder.—J. H. P.

*Drying apparatus with a column of receptacles movable through a drying shaft.* F. H. Schule G.m.b.H., Hamburg. Ger. Pat. 306,987, 24.2.16.

THE compartments of the apparatus, one above another, are each provided with an inlet for fresh hot dry air on one side and an air outlet on the opposite side. The inlet of one compartment is on the opposite side to that of the next above or below, so that the material is traversed by air alternately in opposite directions.—J. H. L.

*Air blast; Process and apparatus for drying* — by means of calcium chloride. L. Honigmann, Aix-la-Chapelle. Ger. Pat. 307,221, 15.7.17.

CALCIUM chloride solution is distributed over a number of containers in a closed vessel; hot gas or air is blown through the vessel to evaporate the solution and dry the calcium chloride, and then the blast to be dried is passed through. In the apparatus claimed the containers consist of trays situated one above another, with alternate central and peripheral gaps, to allow the solution introduced at the top of the vessel to overflow from one tray to another until all are filled, and to afford a baffled passage for the air or gas passed through the vessel.—J. H. L.

*Furnace walls.* E. Bernitz, South Boston, Mass., U.S.A. Eng. Pat. 119,599, 21.1.18. (Appl. 1188/18.)

A FURNACE wall is provided with an internal air chamber, and the bricks on the furnace side of the chamber are spaced apart to provide a series of openings into the fuel bed. The openings are largest at the middle plane of the fuel bed and decrease in size above and below this level. A

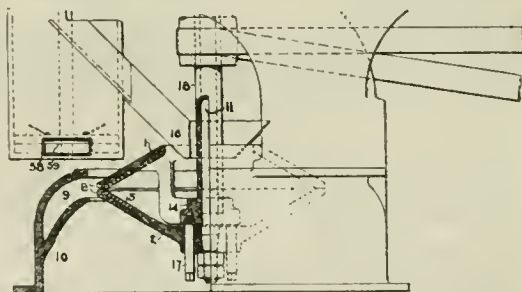
forced draught is delivered to the air chamber through a valve-controlled opening, to cool the furnace wall and prevent the formation of clinker in contact with it. The bridge-wall of the furnace is also provided with air ducts for supplying a regulated quantity of air to the fuel bed.—W. F. F.

*Separators; Centrifugal* —. K. and A. Ward, Stockholm, Sweden. Eng. Pat. 119,642, 11.7.18. (Appl. 11,292/18.)

A hollow horizontal shaft rotating within a stationary outer casing carries a body portion in which a number of separating channels inclined to the axis are provided. The fluid to be separated is fed to the channels at a point between their inner and outer ends. During the separating operation the outer casing is filled with a fluid at such a pressure that the fluid to be separated and its content of particles of less or the same specific gravity are prevented from being thrown out into the outer casing, whilst the heavier particles pass through. —W. H. C.

*Separator; Centrifugal* — for separating solid and liquid mixtures. W. Mauss, Johannesburg. Eng. Pat. 119,706. (Appls. 14,785, 12.10.17, and 5636, 2.4.18.)

IN a centrifugal filter, the filtering drum is composed of two conical members, 1, 2, carried by shafts, 18, 11, respectively. The hub, 14, of the member, 1, carries pins, 17, sliding in the member, 2, so that the two members may be separated axially. Perforated plates, 5, are sunk in the walls of the cones and are covered with filtering cloth, the filtered liquid being discharged through the passages, 8, to the receiver, 9. The two conical members are locked together by a toggle mechanism rotating with the shaft and operated by means of a hydraulic cylinder through suitable levers. The



toggle mechanism is such that the drum and shafts may be lowered as a whole till the outlet holes, 8, are below the rim of the liquid discharge chamber, 9, whereupon further lowering of the shaft, 11, opens the toggle and separates the drum sections, so that the solid matter is discharged into the chamber, 10. The drum sections are then closed and the drum raised to its normal position for a fresh charge, and means are provided to operate the hydraulic cylinder so as to effect this cycle automatically. The pulp to be separated is fed by a pipe, 58, to a reciprocating bucket, 59, which discharges it periodically into the shoot, 16, and thence into the drum. The bucket, 59, is hydraulically operated synchronously with the movements of the drum.—W. F. F.

*Centrifugal separator.* P. T. Sharples, St. Davids, Pa., Assignor to Sharples Specialty Co., West Chester, Pa. U.S. Pat. 1,277,653, 3.9.18. Appl., 6.11.17.

A ROTARY vertical bowl is provided with an inner removable non-perforated liner. The latter



consists of an upper section which is of greater diameter and has inwardly extending ends and is connected with a lower section of less diameter. The mixture is fed into the bottom section and the solid portion is delivered by centrifugal action into the upper section, where it is retained, any entrained liquid draining back into the lower section.—W. H. C.

*Filter; Chemical* —. W. R. Beckley, Assignor to General Filtration Co., Inc., Rochester, N.Y. U.S. Pat. 1,277,832, 3.9.18. Appl., 27.2.15.

THE bottom and sides of the filter tank are lined with plates spaced apart, and a filtering bed of porous tiles, also spaced apart, is formed a short distance above the bottom plates. The spaces between the plates are filled with binding material.—W. H. C.

*Distilling; Process and apparatus for* —. A. B. Foster, Washington, D.C. U.S. Pat. 1,277,895, 3.9.18. Appl., 15.2.13.

IN a cyclic process, the substance to be distilled is caused to traverse a vaporising vessel against a counter-current of gas maintained at a suitable temperature and below atmospheric pressure. The gaseous mixture issuing from the vessel is compressed and cooled, part of the vapour being condensed and separated. The pressure of the remainder is then reduced, and an interchange of heat effected between the compressing and expanding means, provision being made for transmitting kinetic energy from the latter to the compression pump.—W. E. F. P.

*Concentrating apparatus.* N. L. Heinz and J. W. Geib, La Salle, Ill. U.S. Pat. 1,277,922, 3.9.18. Appl., 15.9.15.

A NUMBER of separate furnaces and evaporating pans are set in a row under a common roof, and the fumes are conveyed by a common passage to a series of towers.—W. H. C.

*Evaporating apparatus.* J. H. Fedeler, New York. U.S. Pat. 1,278,127, 10.9.18. Appl., 11.7.11. Renewed 6.11.17.

A STREAM of atomised liquid is projected horizontally into a horizontal chamber, and the spray is dried and held in suspension by jets of heated air projected horizontally below and in the same direction by nozzles arranged along the lower wall of the chamber. The horizontal chamber discharges into the top of a vertical cylindrical chamber, which is provided with openings in its cylindrical walls inclined to the axis, through which air is projected tangentially into the chamber. The top of the horizontal chamber is constituted by a screen formed as an endless band, and the band and air nozzles are oppositely electrified, so that particles are intercepted by the screen and removed.—W. F. F.

*Boiling machine.* E. D. Jefferson, Boston, Mass. U.S. Pat. 1,278,155, 10.9.18. Appl., 19.1.17.

THE machine consists of an outer rotating cylindrical tank containing a perforated cage which rotates with the tank, and steam pipes between the tank and the cage. Exhaust valves and means for controlling the supply of steam, and solution are also provided, and these can all be operated while the tank is rotating.—R. V. S.

*Heat-exchanger; Fractional condensing — and process for fractionating liquids.* E. H. Wright and E. H. Atwood, Olean, Assignors to Vacuum Oil Co., Rochester, N.Y. U.S. Pats. (A) 1,278,279 and (B) 1,278,280, 10.9.18. Appls., (A) 18.5, (B) 19.12.17.

(A) A NUMBER of sections are arranged one above the other in a casing, each section consisting of a number of oil pipes with a collecting pan below for the condensed liquid, a steam preheater, and a steam spray pipe in the pan. The oil flows downwards in a zigzag direction through the sections, and is preheated, and the vapour passes in a zigzag direction upwards and is fractionally condensed. (B) The liquid to be fractionated is distilled and the vapour is passed through the foregoing apparatus, when the different fractions are condensed by the different sections of the condenser. The vapour also superheats steam, which is then sprayed into the collecting pans to re-evaporate fractions lighter than those desired.—J. H. J.

*Fire extinguisher composition.* H. S. Mork., Brookline, Mass., Assignor to American La France Fire Engine Co., Inc., Elmira, N.Y. U.S. Pats. (A) 1,278,714, (B) 1,278,715, and (C) 1,278,716, 10.9.18. Appl., 14.1.18.

THE composition comprises as essential ingredients. (A) a potassium salt of carbonic acid, e.g., potassium carbonate, and an alkali lactate, e.g., sodium lactate, (B) sodium bicarbonate and sodium lactate, or (C) an alkali salt of carbonic acid, e.g., sodium bicarbonate, and an alkali citrate, e.g., potassium citrate.—J. H. P.

*Crystal masses; Method for obtaining large — with well-formed crystals from hot concentrated liquors.* E. Waskowsky, Dortmund, Ger. Pat. 306,173, 10.1.14.

THE liquor is cooled by allowing it to fall freely in a fine state of division. Attaining a state of supersaturation due to supercooling, the drops fall into the crystallising vessel. The concentration at the surface of the liquor in the crystallising vessel is that corresponding to saturation, and the drops being supersaturated, and consequently heavier than the surface liquor, sink to the bottom, so that the crystals are built up on the bottom and sides of the crystallising vessel.—J. S. G. T.

*Spraying liquids; Siphon for* —. H. Petersen, Berlin-Steglitz. Ger. Pat. 307,312, 14.9.17.

IN the lower of two superposed vessels is a float, the hollow stem of which passes up into the upper vessel. The stem of the float carries a ball-valve which when in position, determined by the height of the float, shuts off communication between the two vessels except by way of the hollow stem. The liquid entering the upper vessel, reaches the level of the top of the hollow stem and passes into the lower vessel by way of the stem. The float rises, the ball valve is opened, and the liquid in the upper vessel passes with a rush into the lower. The lower vessel is emptied of liquid by a siphon tube with a "rose" sprinkler attached to its end. For good spraying the total area of the holes in the sprayer should be less than the area of cross-section of the siphon tube.—J. S. G. T.

*Transferring gases from cylinders to the experimental vessel; Arrangements for — in which liquid is used to force the gas out of the cylinder.* Hanseatic Apparatebau Ges., vorm. Bremen u. Co., Kiel. Ger. Pat. 307,489, 15.3.17.

A VERY dilute solution of an electrolyte such as a caustic alkali, or an alkali salt, or mixtures

thereof, is added to the liquid employed in order to prevent rust formation in the interior of the cylinder.—J. S. G. T.

*Air or other gases; Apparatus for purifying* —. H. Ider, Zürich. Ger. Pat. 307,579, 20.5.17. Int. Conv., 11.5.17.

PURIFICATION is effected by saturating the air or other gas with vapour and subsequently cooling. The current of air or gas passes through a horizontal cylinder which is divided into four sections in the following order. First a chamber in which are arranged a series of vapour jets above and below the axis of the cylinder. This is followed by a mixing chamber. In the third section the current of gas is cooled by water sprays. The last section is composed of a coke filter bed over which water drips. The water used in the apparatus runs off through drains in the floor of the apparatus.—J. S. G. T.

*Gas or air ports for regenerative furnaces.* J. Rees, West Hartlepool, and South Durham Steel and Iron Co., Ltd., Stockton-on-Tees. Eng. Pat. 119,826, 15.4.18. (Appl. 6376/18.)

*Drying and aerating machine.* C. E. and W. M. Clark and J. A. Sparks, Elk City, Kans. U.S. Pat. 1,278,593, 10.9.18. Appl., 22.6.17.

See Eng. Pat. 108,520 of 1916; this J., 1917, 996.

*Cooling and evaporating apparatus.* Ger. Pat. 307,483. See VII.

*Electro-osmosis.* Ger. Pat. 307,242. See XI.

## IIa. —FUEL; GAS; MINERAL OILS AND WAXES.

*By-product coking practice; Some characteristics of American coals in* —. F. W. Sperr, jun. J. Franklin Inst., 1918, 186, 133–163.

THE growth of by-product coking in the United States is dealt with and statistics are given showing the distribution of the world's supply of bituminous coals and iron ores. The by-product coking process is much more elastic than the beehive process and consequently a considerably wider range of coals is rendered available for coke production. In practice the tendency is to mix a coal of high volatile content with 15% to 30% of a coal low in volatile matter, the mixture containing 30 to 35% volatile matter; a better coke is thus produced for blast-furnace work. The coking quality of a coal is best determined by practical scale tests, several of which may be necessary to find the most suitable conditions of coal preparation, oven construction, and coking regulation. The geological age of a coal is no criterion as to its coking quality. As good by-product yields and as good coke are obtained from the comparatively recent coals of Colorado as from the early carboniferous strata of Pennsylvania. The coking quality seems to bear some relation to the oxygen content of the coal. The highly oxygenated coals of Illinois may have less volatile matter than coal from a portion of the Pittsburgh seam and yet produce a much inferior coke. As a rule coals of high oxygen content require more careful adjustment and regulation of conditions in order to produce a satisfactory coke than is necessary in the case of other coals containing the same percentage of volatile matter but less oxygen. Coke made in by-product ovens retains approximately 10 to 20% less of the sulphur

originally in the coal than that made in beehive ovens.—T. F. E. R.

*Benzol and naphthalene recovery [from coal gas].* H. E. Copp. Mid. Assoc. Gas Eng. and Managers, Oct. 31, 1918. Gas J., 1918, 144, 311–313.

THE benzol plant is situated at the inlet to the purifiers and comprises scrubber, heat exchangers, oil superheaters, crude benzol still using live steam, analyser, main condenser, separator, and oil coolers. The process is continuous. The washing medium is creosote, prepared in a tar dehydration plant alongside the benzol plant. When fresh, the creosote contains 20 to 25% of naphthalene and gives the following fractions on distillation: up to 200° C. 8%, 200° to 230° 32%, 230° to 280° 20%, 280° to 300° C. 15%, and residue 25%. After passing through the scrubbers, this creosote contains 2% benzol and retains 0.5% after passing through the still. In order to free the creosote from naphthalene and thereby enable it to wash all the naphthalene out of the gas, a small supplementary condenser or analyser has been inserted between the still and the main condenser and is maintained at a constant temperature of 92°–98° C. The liquids condensed at this point consist chiefly of water and naphthalene, which are collected separately and the latter is recovered for sale. The effect of the analyser is to reduce the naphthalene content of the creosote to 6%, with the result that the gas is entirely free from naphthalene after washing; it contains 16.8 grains per 100 cub. ft. at the inlet to the scrubbers. In addition, the creosote lasts for a longer period and the crude benzol is considerably improved.

—T. F. E. R.

*Electric heater for use in the analytical distillation of gasoline.* E. W. Dean. J. Ind. Eng. Chem., 1918, 10, 823–826.

NICKEL-CHROMIUM wire is wound round a mandrel of about  $\frac{1}{4}$  in. diameter, the helix thus formed is stretched out to a length of about 26 in. and then wound as a spiral on a wooden cone 2 in. high and  $1\frac{1}{2}$  in. diameter at the base. The spiral is held in position by brads and a layer of alundum cement is applied over the whole to a thickness of  $\frac{3}{8}$  in. When the cement is set, the wooden cone is removed, the device dried at 100° C., and then baked at 900° C. It is attached to a stout asbestos board which forms the lid of a brass box, so that the cement cone is in an inverted position in the box when the lid is in place. The terminal wires from the cone are connected with binding screws on the lid and the dead space in the box is filled with kieselguhr, shredded asbestos, or magnesia, a silica tube being used to insulate the wire leading from the lower end of the cone. The flask to be heated rests over a hole in the lid immediately over the cone. Sliding wire rheostats are used for regulating the heat.—W. P. S.

*Drilling oils and their substitutes; Examination of* —. J. Marcussen. Mitt. K. Material-prüf., 1918, 36, 107–112.

VARIOUS substitutes have been used in Germany to replace the so-called water-soluble mineral oils used in the drilling of armour plate, etc. Those which are free from oil include sulphite lyes, extracts of vegetable mucilage, and solutions of glue, and are characterised by dissolving in water without forming an emulsion. Sulphite lyes when treated with excess of dilute hydrochloric acid, yield a precipitate of ligninsulphonic acid, which is insoluble in ether or benzene, and is decomposed by strong hydrochloric acid, with the liberation of sulphur dioxide. They reduce Fehling's solution



and solutions of dyestuffs such as Methylene Blue, and give the naphthol-sulphuric acid reaction for carbohydrates. On evaporation sulphite lyes leave about 10% of residue containing 10 to 15% of ash, 1 to 10% of sulphur, and about 0.4% of nitrogen. When used as a substitute for drilling oil they require the addition of alkali as a rust-preventive. The brown specks formed on the metal when strongly alkaline sulphite lyes are used are due to tannin constituents of the lyes. Vegetable mucilage extracts are prepared from seeds such as linseed, and especially from carrageen moss, which is first soaked and then boiled with soft water. Good preparations should give a nearly fluid jelly with 16 to 20 parts of water. They are characterised by the presence of vegetable particles which are rendered more distinct by staining with iodine. They yield a precipitate with alcohol, which does not dissolve readily in hot water. Fehling's solution is not reduced, unless the liquid has previously been heated with dilute acid. In practice the formation of rust on the steel may be prevented by adding sufficient alkali. Glue solutions for drilling purposes are prepared by dissolving 40 parts of glue in 50 parts of water and 10 parts of potassium hydroxide solution (50° B., sp. gr. 1.53). Glue is precipitated almost quantitatively by tannin, and contains about 15% of nitrogen. The other group of drilling oil substitutes consists of mixtures of the above-mentioned preparations with mineral oil or tar oil, as, for example, a mixture of 80 parts of sulphite lye, 5 parts of potassium hydroxide solution (40° B., sp. gr. 1.38), and 15 parts of tar oil or mineral oil. They should be tested as to their emulsibility with water and rust-preventing properties. For the analysis of these products 10 grms. is dissolved in 100 c.c. of petroleum spirit, and repeatedly shaken with alcohol. The alcoholic extracts, which will contain any emulsifying soaps present, are evaporated, and the residue dried and weighed. If ammonium soaps are present the ammonia will be driven off during the evaporation, and the residue will consist only of the fatty acids, etc., of the soap. The residue is decomposed with dilute acid and the separated acids are tested for fatty acids, rosin acids, and naphthenic acids. To detect the last-named, the acids are converted into copper salts and treated with petroleum spirit, which dissolves copper naphthenate forming a green solution. Naphthenic acids may be distinguished from fatty acids by the greater solubility of the magnesium salts in water. They have also a much higher sp. gr. (e.g., 0.998 as compared with 0.898 to 0.920 at 20° C.). Another characteristic of naphthenic acids is their ready formation of sparingly soluble "formolites." The acid under examination is mixed with an equal vol. of strong sulphuric acid and shaken with about one half vol. of 40% formaldehyde. After standing for about 15 mins. the liquid is diluted with water and treated with ether, which dissolves the decomposition products of oleic acid, etc., leaving an insoluble precipitate of the naphthenic acid compound. Finally the preparations are tested for volatile constituents such as alcohol, petroleum spirit, and ammonia.

—C. A. M.

*Action of coal gas on plants.* Wehmer. See XVI.

#### PATENTS.

*Coal or the like; Apparatus for washing — and for separating it from its impurities.* The Grange Iron Co., Ltd., and H. P. Hoyle, Durham. Eng. Pat. 119,835, 22.5.18. (Appl. 8539/18.) Addition to 15,827 of 1915 (this J., 1916, 1209).

In coal-washing apparatus of the type described in the principal patent, the coal is fed from a hopper

on to an inclined perforated plate in a tank, and the water is circulated by a parallel plate below, which is pivoted at one end, and reciprocated at the other by an eccentric and rod. The reciprocating plate is provided with flap valves which open only on the downward stroke. At the lower end of the perforated plate, pockets for collecting dirt are provided, which are closed by pivoted and counter-weighted doors at the bottom. The doors are opened by the weight of the material, and are connected with dash-pots to control their movements. The perforated plate is kept free from clogging by a scraper which is slowly reciprocated over it by means of an eccentric and pawl and ratchet. The washing water overflows into a settling tank provided with vertical baffle-plates having openings which are out of line with one another. The slurry settling in this tank is delivered by a conveyor on to an elevator which receives the washed coal from the perforated plate.—W. F. F.

*Combustible gases; Device for producing —.* Chinese-American Co., Boston, Mass., Assignees of W. B. S. Whaley, Malden, Mass., U.S.A. Eng. Pat. 111,102, 13.10.17. (Appl. 14,850/17.) Int. Conv., 19.10.16.

CRUDE gas, e.g., coal gas, enters a separator chamber in which solid impurities and heavier coal distillation products are retained; the gas and lighter liquid products go forward into the distributing box of a chamber containing a large number of parallel jet pipes. The gas passes down through these pipes at high velocity, and on emerging, impinges on a metal partition heated from underneath by the exhaust gases from an internal combustion engine which afterwards pass into the air. The vapours in the coal gas are thereby completely gasified or "cracked." The hot gases pass up round the pipes (thereby heating the incoming gas) to the outlet of the chamber, and after cooling are ready for use in a gas engine without further purification. The device may also be used as a carburettor, in which case, oil is run into the space above the hot plate and air forced through the jet tubes. The air in bubbling through the heated oil becomes carburetted.—T. F. E. R.

*Gas-producers.* S. Glover, St. Helens, J. West, Southport, and West's Gas Improvement Co., Ltd., Manchester. Eng. Pat. 119,723, 23.10.17. (Appl. 15,396/17.)

The producer gas is taken off directly from the hot zone, by ports leading into an annular chamber wherein dust is deposited, and the gas then passes to the gas main. The ash is discharged at the bottom by the action of a rotating inverted cone having on its interior a projecting spiral rib. The ash falls on to a table placed at such a distance from the bottom of the cone as to prevent the producer emptying itself; a rotating vane continually discharges some ash from this table. The joint between the revolving cone and the producer is water-sealed. By a suitable arrangement of pipes at this point, steam or other gases can be admitted. Air is admitted to the producer at the lower end of the cone.—T. F. E. R.

*Gas for internal combustion engines and for other purposes; Method and means for electrically producing —.* T. McClelland, Glasgow. Eng. Pat. 119,885, 24.9.17. (Appl. 13,697/17.)

CARBON, similar to arc-lamp carbon, in granules about pea size, is loosely packed in separate parallel grooves, or one sinuous groove, in the bottom of a porcelain vessel. The carbon is covered with water alone, or water containing

acid or alkali. Electric conductors pass through the gas-tight cover of the porcelain vessel and make contact with the rows of carbon granules. By passing a suitable current through the granules an explosive gas is evolved partly by electrolytic action and partly by the smothered arcing action between the granules. An opening is provided for withdrawing this gas for use in a gas engine. As an alternative to the grooves, the carbon may be contained in perforated porcelain tubes closed at the ends by carbon conducting blocks of lower electrical resistance than the granules.—T. F. E. R.

*Gas; Apparatus for making* — J. U. McDonald, La Grande, Oreg., Assignor to American Gas and Incinerator Co., Tacoma, Wash. U.S. Pat. 1,278,180, 10.9.18. Appl., 22.11.16.

THE apparatus comprises a vertical retort above which is a charge chamber closed top and bottom by gas-tight valves. The charge chamber is filled with the material to be carbonised from a superposed hopper by opening the top valve. When full this valve is shut and the bottom one opened, and the charge is fed into the retort at any desired rate through a chamber containing an endless screw. The residue is removed from the bottom of the retort by a horizontal endless screw. The gas is taken off at the top of the retort, below the feed chamber. Pressure-equalising pipes connect the bottom of the retort with the bottom of the charge chamber, and the top of this chamber with the hydraulic main.—T. F. E. R.

*Gas producers; Method for the control of* — A. Riedel. Ger. Pat. 308,259, 4.1.16.

IN those parts of the producer in which a low temperature is desirable, a spray of water is introduced along with the blast and steam. The fineness of the spray is controlled by an arrangement of jets. In any cross-section of the producer the centre and outer portions are kept cool. Between these there is a region of higher temperature. By adjustment of the jets it is possible to obtain a uniform rate of gas production in any cross-section despite differences of temperature which exist.

—J. S. G. T.

*Cracked oils; Purification of* — A. E. Dunstan, Sumbury-on-Thames. Eng. Pat. 119,751, 28.11.17. (Appl. 17,613/17.)

OLEFINS and diolefines, which impart a yellow colour and objectionable odour to petroleum cracked by the usual methods, are removed by slowly distilling the cracked oil in the presence of about 1% of an anhydrous chloride (e.g., anhydrous aluminium chloride). To render it more effective, the chloride may be applied dissolved in an anhydrous solvent (e.g., absolute alcohol), or mixed with a dry inert material; the mixture may also be stirred during distillation.—L. A. C.

*Heavy fuel oils [mixtures of heavy oil with pitch]; Treatment of* — preparatory to burning. J. W. Westwood, Birmingham. Eng. Pat. 119,977, 9.2.18. (Appl. 2372/18.)

PITCH is melted in a tank heated by steam or superheated water, and is heated to a temperature below the flash point of the oil with which it is to be mixed. It is then forced into heavy oil contained in another tank heated by similar means. The mixture is pumped around a system of pipes connected with the burners, any excess oil being returned through a return pipe to the mixing tank. Agitation of the oil and pitch in the mixing tank is effected by feeding in the pitch and excess mixture from the burners through a pair of perforated

pipe systems or spargers so disposed that the issuing streams effectively mix and agitate the contents of the tank.—L. A. C.

*Charging vertical retorts or ovens; Apparatus for* — Apparatus for discharging coke or the like. J. Pieters, Paris. Eng. Pats. 113,435 and 113,436, 22.1.18. (Appls. 1272 and 1273/18.) Int. Conv., 10.2 and 16.2.17.

*Burning solid fuel; Process of* — F. Thuman, London. From O. B. Evans, Lansdowne, Pa., U.S.A. Eng. Pat. 120,011, 20.6.18. Appl. 10,206/18. SEE U.S. Pat. 1,267,646 of 1918; this J., 1918, 499 A.

*Heat exchanger for fractionating.* U.S. Pats. 1,278,279 and 1,278,280. See I.

*Separating sulphur from gas.* Ger. Pat. 306,988. See VII.

*Hydrogen sulphide from gases.* Ger. Pat. 308,107. See VII.

*Blast-furnace gas.* Ger., Pat. 306,853. See X.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

### PATENTS.

*Wood; Carbonisation of* — L. E. Tissier, Batna, Algeria. Eng. Pat. 110,360, 8.10.17. (Appl. 14,541/17.) Int. Conv., 6.10.16. Addition to 105,915 (this J., 1918, 184 A).

WOOD contained in a kiln is carbonised by the exhaust gases of internal combustion engines, e.g., those worked with poor gas. The exhaust gases are passed into the kiln through a grating at the bottom, and the gaseous products from the wood, including tar, are withdrawn at the top through a removable sheet metal hood provided with a water seal, and then passed into a condenser. The charcoal is withdrawn through a door at the side and quenched in a pit immediately below.

—W. F. F.

*Carbonisable materials; Distillation of* — A. L. Pearse, London. Eng. Pat. 119,700, 11.10.17. (Appl. 14,725/17.)

CARBONACEOUS material such as coal, lignite, oil shale, or the like, is fed continuously from air-tight hoppers into a series of vertical retorts, and the solid residue is continuously withdrawn at the bottom. The retorts are heated by superposed horizontal flues in the dividing walls, each flue being provided with a gas burner at one end which can be separately regulated. The air supply is preheated by passing it through passages in the walls at the bottom, and the carbonised material is thereby cooled. The horizontal flues are connected by short vertical passages which are provided with dampers and increase in size with their distance from the burner. The lowest horizontal flue is connected to a discharge stack, so that the hot gases move horizontally along their respective flues and also downwards through the lower flues to the stack. Cold air may also be supplied to each flue. The retorts are thus heated in zones the temperature of which increases progressively towards the bottom. Discharge conduits are provided for the gases at two different levels, and are formed by increasing the width of the retort in steps downwards at those places, and allowing the end wall of the upper narrower zone to project



downwards into the lower zone, thus forming a vertical discharge conduit which is not subject to choking. In distilling coal, the upper zone is maintained at 900°–1000° F. (480°–540° C.), the middle zone at 1350° F. (730° C.), and the lower zone at a higher temperature. Gas from the upper and middle zones, containing benzene and toluene, is withdrawn through the upper conduit, and gas from the lower zone consisting of hydrogen, carbon monoxide and dioxide, and ammonia, is withdrawn from the lower conduit.—W. F. F.

*Retorts for distilling, carbonising, roasting, and the like, of coal, carbonaceous and other materials.* A. Bradley, London. Eng. Pat. 119,911, 18.10.17. (Appl. 15,134/17.)

HORIZONTAL or slightly inclined retorts are arranged in superposed pairs and are surrounded by heating flues and separately heated by regulated gas burners. An endless chain conveyor travels through each pair of retorts, and the fuel or ore is fed from separate hoppers at opposite ends of the two retorts and discharged at the other end of each retort. A band conveyor may be used for finely divided material not liable to cake during heating. The conveyor passes through a closely fitting slot at the entrance to the retort to remove adhering material and prevent loss of heat. The material is discharged from the conveyor by a fixed V-shaped plate into lateral shoots. For a material which cakes during heating, a conveyor carrying cup-shaped scoops may be used. The apparatus is applicable for carbonising coal, shale, peat, and wood, and for roasting minerals or the like.

—W. F. F.

*Incandescence bodies; Preparation of — composed of a conductor embedded in rare earths.* J. Frank Rahtjen, Berlin-Schöneberg. Ger. Pat. 308,036, 30.11.13.

A HELICAL conductor is covered with fibre which is then saturated with salts of the rare earths. The incandescence body so obtained is then burnt off, covered with shellac or similar substance, shaped, and hardened in a neutral atmosphere or *in vacuo*.—J. S. G. T.

*Charging and discharging of retorts for the distillation of coal, shale, or the like.* J. Robertson, Glasgow. Eng. Pat. 119,715, 22.10.17. (Appl. 15,284/17.)

### III.—TAR AND TAR PRODUCTS.

*Coal-tar industry; Methods of analysis used in the —. II. Distilled tars and pitches.* J. M. Weiss. J. Ind. Eng. Chem., 1918, 10, 817–823.

WATER, sp. gr., matter insoluble in benzene, and viscosity of distilled tar are determined by the methods mentioned for the analysis of crude tar (this J., 1918, 648A). Consistence is determined by the Schutte penetrometer or by the float apparatus, and the distillation test is made under definite conditions in an apparatus of standard dimensions. In the case of pitch, the sp. gr. may be determined by the methods mentioned and also by the suspended cube (in water) method. The melting point is determined by noting the temperature at which a cube of the pitch drops from a metal hook when suspended in water, and the melting point in air is determined in a similar way. The evaporation test consists in heating 10 grms. of the pitch in a nickel basin for 7 hrs. at 163° C. and noting the loss in weight. The

sliding test is made by placing a cube of the pitch in a groove in a plate inclined at 30° and heated at 40° C., the extent of the flow being noted after 7 hrs.—W. P. S.

*Hydroxyazo-compounds; Electrolytic reduction of —.* E. Puxeddu. Gaz. Chlm. Ital., 1918, 48, 11., 25–30.

IN alcoholic solution containing hydrochloric acid, *p*-hydroxyazobenzene undergoes electrolytic reduction to *p*-aminophenol, whereas, dissolved in sulphuric and acetic acids, it gives no definite result.

—T. H. P.

*Benzol and naphthalene recovery.* Copp. See IIA.

#### PATENTS.

*Heavy fuel oils.* Eng. Pat. 119,977. See IIA.

*Artificial stone.* Ger. Pat. 306,975. See IX.

### IV.—COLOURING MATTERS AND DYES.

*Formic acid; Action of — on triarylcabinols.* [Triphenylmethane dyestuffs.] A. Kovache. Ann. Chlm., 1918, 10, 184–248.

A MORE detailed account of work already published (see this J., 1913, 528).—W. G.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

*Wool fibres; Influence of humidity upon the strength and elasticity of —.* J. I. Hardy. J. Agric. Res., 1918, 14, 285–295.

THE breaking strength of wool fibres was determined under controlled conditions of temperature and moisture by means of Reeser and Mackenzie's fibre-testing machine (Matthews, "Textile Fibres," 1913, p. 254). The tests were made in a special room, the temperature of which was controlled by a thermograph electrically connected with overhead lamps, whilst the humidity was controlled by means of a hydrograph connected with atomisers distributing a fine spray of water into a large tank. It was found that the wide variations in the size of individual fibres caused the determination of the breaking-strength to be an unsatisfactory method of measuring the strength of wool. Owing to irregularity in the shape of the fibres, microscopical measurement of the diameter gave inaccurate results, but rapid and accurate measurements were made by means of a micrometer caliper substituted for the lower jaw of the testing machine. The tensile strength (ratio of breaking strength to area of cross-section) was calculated from the diameter of the fibre as measured at the smallest point. Comparison of the tensile strength at five relative degrees of humidity (40, 50, 60, 70, and 80%) showed a decrease in strength with the increase in the humidity, and *vice versa*. The percentage elasticity of wool fibres increased with the rise in the humidity. For example the average elasticity of four samples of different kinds of wool fibres increased from 27.81 at a relative humidity of 40% to 39.08 at a humidity of 80%.—C. A. M.

*Drilling oils, etc.* Marcusson. See IIA.

## PATENTS.

*Paper: Method of coating — and articles produced thereby.* H. Gesell, Montclair, N.J. U.S. Pat. 1,277,904, 3.9.18. Appl., 15.2.15.

PAPER is coated with a solution of a resinous condensation product and a wax, such as paraffin, and then heated sufficiently to volatilise the solvent and convert the residual coating into a homogeneous mixture of wax and infusible, insoluble condensation product. For making paper transparent, a fusible phenol resin is employed, without wax, in the same manner.—W. E. F. P.

*Plastic substances: Manufacture of —.* F. Ringer, Vienna. Ger. Pat. 306,025, 28.6.17.

FINELY divided paper or wood, with or without previous treatment with sulphuric acid, is thoroughly mixed with a binding material made by treating a mixture of gelatin or glue, alum, fatty acids, and varnish with formaldehyde. The material so prepared is air-dried and shaped by pressure. Water-glass solution may be added. The product is stronger and more water-resistant than so-called "vulcan-fibre."—F. C. T.

*Cellulose acetates: Process of making —.* H. Dreyfus, Basel, Switzerland. U.S. Pat. 1,278,885, 17.9.18. Appl., 23.9.15.

SEE Fr. Pat. 478,023 of 1914; this J., 1916, 1152.

*Cellulose compounds: Manufacture of —.* L. P. Wilson, Coventry. Assignor to The Viscose Co., Marcus Hook, Pa. U.S. Pat. 1,279,200, 17.9.18. Appl., 22.1.15.

SEE Eng. Pat. 14,675 of 1914; this J., 1915, 606.

*Cellulose compounds: Manufacture of —.* W. H. Glover, Braintree, and L. P. Wilson, Coventry. Assignors to The Viscose Co., Marcus Hook, Pa. U.S. Pats. 1,279,328 and 1,279,329, 17.9.18. Appls., 22.1.15 and 2.8.18.

SEE Eng. Pat. 13,055 of 1914; this J., 1915, 606.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

*Bleaching and washing materials: Determination of loss of oxygen during the use of oxygen-yielding —.* A. Grön and J. Jungmann. Chem.-Zeit., 1918, 42, 473—475.

THE method described consists in heating a known weight of fabric with a definite quantity of the bleaching agent in a flask under reduced pressure. The flask is connected through a condenser with a gas burette, in which any gases given off during the heating are collected. Subsequent analysis of the gases gives the quantity of oxygen evolved from the bleaching agent and not combined with the fabric, etc. The quantity of nitrogen found in the gases is taken as a measure of the air present in the apparatus and an allowance is made for the oxygen derived from this source. Results of experiments with perborates show that from 60 to 80% of the active oxygen escapes in the gaseous form, but if soap is also present the loss is under 12%. The greater part of the active oxygen is absorbed by the oxidisable constituents of the soap.—W. P. S.

*Cotton fibre: Dyeing of — with basic dyes.* R. Haller. Kolloid Zeits., 1918, 23, 100—106.

THE mechanism of dyeing cotton with basic dyes is shown to be the following in the case of Methylene

Blue. On immersing the fabric in a tannin solution, this by virtue of its highly disperse state penetrates into the interstices, and when the tannin-charged fabric is placed in a solution of tartar emetic this substance as a molecular disperse solution penetrates very rapidly and forms in the interstices an insoluble antimony-tannin compound. When now the fabric is placed in the Methylene Blue bath this substance, being also highly disperse, penetrates and forms a complex lake, tannin-antimony-Methylene Blue, which is fixed in the fibre. A similar course is followed when aluminium sulphate is used instead of tartar emetic, but in this case the tannin-aluminium compound is soluble and consequently the colours are not so fast as in the former case.—J. F. S.

## PATENTS.

*Bleaching fibres: Process for —.* A. Schaidhauf, Frankfurt, Germany. Assignor to The Roessler and Hassacher Chemical Co., New York. U.S. Pat. 1,278,389, 10.9.18. Appl., 25.8.14.

SEE Fr. Pat. 460,959 of 1913; this J., 1914, 76. The bath is maintained at about 87° C. during the entire bleaching operation.

*[Machines for] printing fabrics and yarns.* The Calico Printers' Assoc., Ltd., E. B. Clegg, and F. Farnworth, Manchester, and F. Roberts, Accrington. Eng. Pat. 119,947, 30.11.17. (Appl. 17,721/17.)

*Vomiting-kier.* E. D. Jefferson, Boston, Mass. U.S. Pat. 1,278,154, 10.9.18. Appl., 29.12.16.

SEE Eng. Pat. 112,260 of 1917; this J., 1918, 576 A.

*Dyeing machine.* H. M. Dudley, Philadelphia. U.S.A. Eng. Pat. 113,094, 28.1.18. (Appl. 1592/18.) Int. Conv., 31.1.17.

SEE U.S. Pat. 1,258,546 of 1918; this J., 1918, 265 A.

*Boiling tank.* U.S. Pat. 1,278,155. See I.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Cottrell processes in the sulphuric acid industry.* A. A. Heimrod and H. D. Egbert. Chem. and Met. Eng., 1918, 19, 309—314.

THE Cottrell process is used in the manufacture of sulphuric acid for cleansing the hot gases as they leave the pyrites burners, and for removing the mist of acid carried in the gases from concentrators and absorption towers. In the former case the temperature of the gases and character of the suspended matter are governing factors, and at temperatures of 1000° F. (540° C.) and over, and for the removal of dust rather than fume, precipitators of the plate type are preferable in which the gases pass horizontally between collecting and discharge electrodes. With gases cooled to about 600° F. (320° C.) and for the removal of fume, the pipe type of precipitator is preferable. In a Wisconsin plant for roasting zinc ore, 17,500 cub. ft. of gas per minute is cleansed at about 500° F. (260° C.) by an installation of two units, each containing 36 steel pipes as collecting electrodes, 12 in. in diameter and 15 ft. high, with discharge electrodes in the form of chains arranged axially. The electrical equipment consists of a 200 to 65/70/75/80/85000 volt transformer, and the power consumption is about 18 kilowatts; 7600 lb. of



material containing 22% zinc and 30% sulphur is collected every 24 hours. In a precipitator at Baltimore working at 1100°–1400° F. (590°–760° C.), 17,500 cub. ft. of gas per minute at 1100° F. (590° C.) is treated, and 900 to 1200 lb. of dust per day is obtained from the roasting of 40 to 50 tons of pyrites. The installation is made in two sections, each of which may be shut off by dampers. The electrodes are heavy steel plates, suitably reinforced, suspended, and insulated, and a voltage of about 50,000 volts is used, the power consumed being not over 2 kilowatts. For the removal of acid mist from concentrator gases, the pipe type of precipitator is employed. Precipitators of this type are described, in one of which 3000 cub. ft. of gas per minute at 135° F. (57° C.) is treated from three concentrators of the tower type yielding 50 tons of acid of 66° B. (sp. gr. 1.84) per 24 hours, and 3000 lb. of acid of 18° B. (sp. gr. 1.14) is recovered per 24 hours from the mist. A potential of 75,000 volts is employed, with a power consumption of 3½ kilowatts. Another unit, similarly equipped electrically, of 12 pipes each 12 in. by 15 ft., treats 2500 cub. ft. of gas per minute at 200° F. (93° C.) from two tower concentrators producing 40 tons of acid of 66° B. (sp. gr. 1.84) per 24 hours, and 3500 lb. of acid of 33° B. (sp. gr. 1.296) is recovered. One of the largest installations cleanses 30,000 cub. ft. of gas at 180° F. (82° C.) per minute from 22 concentrators of the cascade type, each producing 10 tons of 66° B. acid per 24 hours. Four units, each of 12 pipes, 12 in. by 15 ft., are employed, and 25 tons of 45° B. acid (sp. gr. 1.45) is recovered per 24 hours. The high-potential parts of precipitators for collecting acid mist must be provided with insulators having a large safety factor as regards voltage rating, and must be protected from acid fumes. Fans are mostly used to create the necessary draught, but steam jets are used in some cases, though they are not so satisfactory.—B. N.

*Sulphur in pyrites; Source of error in the determination of* —. O. Binder. Chem.-Zeit., 1918, 42, 503.

To ensure thorough mixing of the sodium carbonate and potassium nitrate in preparing a stock of fusion mixture, it is recommended that the sodium carbonate be suitably coloured and the mixing then continued until the colour of the whole mixture is uniform.—W. P. S.

*I. Hydrochloric acid; Detection and separation of — in presence of bromic and iodic acids.*  
*II. Bromic and iodic acids; Detection and quantitative determination of — in presence of hydrochloric, hydrobromic, and hydriodic acids.*  
 A. Purgotti. Gaz. Chim. Ital., 1918, 48, II., 63–66.

THE methods described are based on the property possessed by potassium or sodium hydroxide of attacking silver bromate and iodate, whereas silver chloride, bromide, and iodide remain unchanged. The solution to be examined is treated with silver nitrate solution in presence of nitric acid, the precipitate being collected and washed and then digested for some minutes in the cold with the alkali hydroxide solution; silver bromate and iodate are thus transformed into the corresponding soluble alkali salts. The liquid is filtered and the precipitate thoroughly washed, and tested for chloride, bromide, and iodide in the usual way. To test the filtrate for bromate and iodate ions, it is acidified with excess of hydrochloric acid and treated in a test-tube with one or two c.c. of carbon bisulphide, sulphurous acid solution being then carefully added, drop by drop, and the whole vigorously shaken. In presence of bromic acid the carbon bisulphide

becomes yellow or orange-yellow, and in that of iodic acid, violet. If both bromic and iodic acids are present, careful addition of the sulphurous acid reveals first the yellow colour of the bromine and afterwards the violet colour of the iodine. The presence in the original silver precipitate of salts of the cyanogen acids does not affect the reaction, since silver thiocyanate and ferrocyanide are not attacked by cold alkali hydroxide, whilst the cyanide is slightly attacked and the ferricyanide decomposed; when cyanide and ferricyanide are present, rather more sulphurous acid is added. The determination of bromic and iodic acids in presence of hydrobromic and hydriodic acids may be effected by employing the means of separation described above, the alkaline solution containing sodium or potassium bromate and iodate being then reduced with either zinc dust, or aluminium or Devarda's alloy (Cu 50, Al 45, Zn 5), and the bromide and iodide precipitated with silver nitrate in acid solution. If cyanogen derivatives are present, they must be removed, the procedure being as follows: the silver precipitate is calcined, the residue treated with dilute sulphuric acid and zinc, and the solution reprecipitated with silver nitrate; the precipitate then contains only bromide and iodide.—T. H. P.

*Selenious and selenic acids; Volumetric determination of* —. L. Moser and W. Prinz. Z. anal. Chem., 1918, 57, 277–305.

THE most accurate results in the determination of selenious acid are obtained when the substance is distilled with hydrochloric acid and an excess of potassium iodide (four times the quantity required by theory), and the liberated iodine then titrated both in the receiver and in the residue. The indirect iodometric method described by Gooch and Pierce (Z. anorg. Chem., 1896, 11, 249), in which the selenious acid is heated with a definite and known excess of potassium iodide in the presence of potassium hydrogen arsenate and sulphuric acid, and the arsenious acid formed then titrated with iodine solution after neutralisation, also gives accurate results if carried out as a distillation process. In the oxidation of selenious acid by permanganate in alkaline solution there is always some loss of oxygen when the hot mixture is acidified previous to the titration of the excess of permanganate, and the results obtained are, therefore, inaccurate. There is formation of hydrogen selenide when selenious acid is titrated with titanium trichloride solution and this method is, consequently, unsatisfactory. The best method for the determination of selenic acid consists in reduction with hydriodic acid. The selenic acid solution (100 c.c.) is treated with 10 c.c. of concentrated hydrochloric acid and an excess of potassium iodide (5 grms. for each 0.1 gm. of selenium), the mixture distilled in a current of carbon dioxide, and the iodine titrated in the receiver and also in the residue.—W. P. S.

*Ammonia-oxidation and similar reactions; Starting and stability phenomena of* —. F. G. Liljenroth. Chem. and Met. Eng., 1918, 19, 287–293.

IT has been shown by the "conversion curves," giving the percentages of ammonia oxidised at various temperatures, and the "heat of reaction line," which gives the temperature of the gauze or catalyst for various percentages of conversion, that the latter line is straight and cuts the conversion curve at two points, one only of which is stable. This position is the one where the line cuts the curve from below. It is shown that there is a particular ignition temperature to which the mixture must be heated to start the reaction. It is of

importance to keep the composition and velocity of the gas constant, in order to keep the reactions stable, and in the presence of catalyst-poisons a higher working temperature must be employed either by the use of a heat exchanger or the supply of additional heat. The stability of the reaction is probably increased with increase in the percentage of ammonia in the mixture used. Increase of gas velocity decreases the conversion and the stability, and increase of thickness or number of gauzes has the same effect, though the introduction of a heat exchanger may stabilise the reaction in the former case with an increased output. It is suggested, however, that it may be possible to use a low-grade catalyst for the first gauze and a platinum one for completing the oxidation, and that, by obtaining conversion curves for different gas strengths and velocities for ammonia-air and ammonia-oxygen mixtures, it may be possible to adjust or design a burner for a maximum commercial efficiency.—B. N.

*Potassium chloride; Heat technology applied to solution and cooling in the preparation of — [from crude potassium salts].* Krull. Kali, 1917, 11, 307–316. Z. angew. Chem., 1918, 31, Ref., 303.

The separation of potassium chloride from crude potassium salts, depending upon the different solubilities of potassium chloride and sodium chloride at different temperatures, is essentially a problem of applying and extracting heat. According to the constitution of the crude salts, extraction is effected at 90°–130° C., and the solution is cooled to 15°–25° C. Enclosed steam is usually employed in the extraction process, the liquor being warmed to 70° C. in preheaters, and then heated to 90°–115° C. in the extraction vessels. Low-pressure exhaust steam is used. In cooling the liquors, the main object is to produce a high yield of well-defined crystals. Heat may or may not be recovered during the process; preferably, the liquor is cooled to a low temperature without the recovery of heat. Salts precipitated at lower temperatures are somewhat contaminated with sulphate, but these may be profitably employed in the manufacture of sulphates; on the other hand, the quality of the liquor is improved for use again in the extraction process by the removal of sulphates. The cooling boxes formerly employed yielded a product excelled by no other method; on the other hand, they possessed great disadvantages in their low output and the necessity for removing the product by hand. Cooling by stages only partly remedied this defect.—L. A. C.

*Nitrites and hydrogen peroxide; Determination of small quantities of —, separate or together. [Estimation of sulphur in thiocyanates.]* A. Quartaroli. Gaz. Chim. Ital., 1918, 48, I., 102–111.

The bromine water usually employed for the oxidation of the sulphur present in thiocyanates to sulphuric acid, to be estimated as barium sulphate, may be conveniently replaced by nitric acid and sodium nitrite: to 5 c.c. of thiocyanate solution mixed with 50 c.c. of water was added 2 c.c. of concentrated nitric acid, the liquid being heated over a small flame, and a solution of 1 gm. of sodium nitrite in 20 c.c. added, drop by drop and with stirring, before the temperature reached about 60° C.; the solution was then heated to boiling and barium chloride added immediately. Percentage of thiocyanate thus found in solution was 6.15, whilst with bromine the result was 6.48.

The following reagent gives a blood-red coloration with a nitrite, the reaction being capable of

detecting about 1 part of nitrite per 200,000,000 of solution: to 100 c.c. of the above potassium thiocyanate solution are added 0.5 gm. of crystallised ferrous sulphate and 2 c.c. of pure, colourless nitric acid diluted with an equal volume of water, the liquid being then stirred until the ferrous sulphate is completely dissolved. Two c.c. of the reagent is added to 25 c.c. of the solution under examination. A similar reaction, though less intense, is given by hydrogen peroxide (1:30,000,000), potassium persulphate, and probably other compounds of the type of hydrogen peroxide, but not by chlorates or perchlorates. With hydrogen peroxide, the maximum coloration is reached almost instantaneously, but with nitrites, slowly and with characteristic striation. If the reaction is positive and the coloration does not change at all when the liquid is heated rapidly to boiling with hydrochloric acid and urea, hydrogen peroxide is present, but no nitrite, and the peroxide may easily be determined colorimetrically. If the reaction is positive and the liquid becomes paler but not colourless when boiled with hydrochloric acid and urea, both nitrous acid and hydrogen peroxide are present, the former in excess. If the reaction is positive and the coloration disappears or, rather, becomes similar to that obtained with distilled water, on boiling with hydrochloric acid and urea, only nitrous acid or excess of this and a small proportion of peroxide may be present.—T. H. P.

*Sulphites, thiosulphates, and polythionates; Studies on —.* A. Sander. Z. angew. Chem., 1918, 31, 197.

In connection with his work on the quantitative determination of sulphites, thiosulphates, and polythionates (this J., 1915, 225, 277; 1916, 307, 421), the author has studied the action of these substances on mercuric chloride. By the interaction of mercuric chloride with alkali thiosulphates and polythionates, very unstable mercury thiosulphates and polythionates are formed, which immediately break down into mercuric sulphide and free sulphuric acid (together with free sulphur in the case of tetra- or pentathionates). Alkali sulphites and bisulphites, on the other hand, form with mercuric chloride stable complex compounds, salts of the disulphonic acid,  $\text{Hg}(\text{SO}_3\text{H})_2$ , and of the chlorosulphonic acid,  $\text{HgClSO}_3\text{H}$ . The quantitative analytical processes worked out by the author depend upon this difference in behaviour towards mercuric chloride.—E. H. R.

*Phosphates; Behaviour of — at the anode.* F. Fichter and J. Müller. Helv. Chim. Acta, 1918, 1, 297–305.

By the anodic oxidation of a 2M solution of dipotassium hydrogen orthophosphate containing 2N potassium fluoride and 0.32 gm. of potassium chromate per litre, the potassium salts of monoperphosphoric acid ( $\text{K}_2\text{P}_2\text{O}_8$ ) and perphosphoric acid ( $\text{K}_2\text{P}_2\text{O}_7$ ) have been obtained. The electrolytic oxidation is most efficiently carried out at 5° C. between platinum electrodes in an undivided cell and with a current density of 0.01 amp. per sq. cm.—J. F. S.

*Hypophosphates; Preparation of —.* R. G. Van Name and W. J. Huff. Amer. J. Sci., 1918, 46, 587–590.

HYPHOPHOSPHATES are best prepared by suspending a number of sticks of phosphorus cast round glass rods in a stout glass jar containing a litre of water and 250 grms. of sodium carbonate, which need not be dissolved. The sticks of phosphorus should be about  $3\frac{1}{2}$  in. long and  $\frac{3}{4}$  in. diameter, and should protrude about  $\frac{1}{2}$  in. from the solution. The whole



is then placed in a cool position and left to react until a few drops of the solution just turns Congo red. The phosphorus is then removed and placed in a similar solution. The product, sodium hydrogen hypophosphate, is found in part as a crystalline precipitate at the bottom of the jar, whilst the remainder is obtained by concentrating the solution. It is recrystallised to remove the accompanying phosphates and phosphites. The best temperature to carry out the reaction is  $10^{\circ}\text{C}.$ – $15^{\circ}\text{C}.$ , and the yield is 10%–16% of the theoretical value.

—J. F. S.

*Iron disulphide ( $\text{FeS}_2$ ); Formation of — by a wet method.* V. Rodt. Mitt. K. Materialprüf., 1918, 36, 93–107.

THE iron trisulphide formed as the first product of the interaction of hydrogen sulphide and iron hydroxide, readily becomes partially insoluble in hydrochloric acid, as for example, when the reaction is carried out at a higher temperature. The insoluble substance has the composition  $\text{FeS}_3$ , sp. gr. 4.58 at  $18^{\circ}/4^{\circ}\text{C}.$ , and is formed in accordance with the equation  $\text{Fe}_2\text{S}_3 \rightarrow \text{FeS}_2 + \text{FeS}$ . It may also be produced by the addition of sulphur to iron sulphide, in the absence of substances with an alkaline reaction, which also interfere with its formation from iron trisulphide. It is probably formed in this way in soils containing iron hydroxide and putrescent matter, but free from alkaline-earth carbonates. By boiling solutions of iron with alkali polysulphides it may be found possible to separate iron quantitatively in the form of iron disulphide. (See also Alkali Inspector's Reports, this J., 1912, 717; 1913, 787; 1914, 861; also J. Chem. Soc., Dec., 1918.)—C. A. M.

*Tungsten compounds; Analysis of — by volatilisation in a current of carbon tetrachloride vapour.* P. Jannasch and R. Leiste. J. prakt. Chem., 1918, 97, 141–153.

THE method depends on the formation of a mixture of volatile chlorine derivatives of tungsten when tungstic acid or a tungstate is heated in a current of carbon dioxide saturated with carbon tetrachloride vapour. The volatilised compounds are decomposed by dilute acids and the tungstic acid is precipitated. A temperature below red heat is required for the volatilisation, which takes about 1 hr. for completion. The method may be used for the determination of tungsten in scheelite and wolframite.—W. P. S.

*Molybdenum compounds; Analysis of — by volatilisation in a current of carbon tetrachloride vapour.* P. Jannasch and O. Laubi. J. prakt. Chem., 1918, 97, 154–181.

THE substance, such as molybdic acid, molybdates, molybdenum ores, etc., is heated in a current of carbon dioxide saturated with carbon tetrachloride vapour; the molybdic acid volatilises, and is collected in a receiver, then evaporated with nitric acid, ignited, and weighed. To determine molybdenum, 0.4 gm. of the sample is heated at  $430^{\circ}$ – $630^{\circ}\text{C}.$  in the carbon dioxide-carbon tetrachloride vapour and the volatilised substances collected in a suitable receiver; a trace of substance may remain unvolatilised and this may be heated with hydrochloric acid, the solution filtered, and the filtrate added to the contents of the receiver, which are then evaporated. After silica has been separated in the usual way, the solution is heated with an excess of ammonia, the ferric hydroxide collected on a filter, and the filtrate evaporated to dryness with the addition of *aqua regia*; the residue of molybdic acid thus obtained still contains traces of silica and is once more volatilised in a current of carbon tetrachloride.—W. P. S.

*Carbides of the rare earths of the cerium group.* A. Damiens. Ann. Chim., 1918, 10, 137–183.

THE carbides of the cerium group of metals when decomposed by water give off a gas which is, in all cases, a mixture of hydrogen, saturated hydrocarbons consisting of ethane, propane, and isobutane but no methane, ethylene and its homologues, and acetylene and its homologues. The different constituents of this gaseous mixture, whilst invariably present, vary in their relative proportions according to the velocity of the reaction, which is dependent on the physical state of the carbide. The metallic hydroxides produced are those corresponding to the sesquioxides. Cerous hydroxide was isolated in the pure state as a white compound, which fixes oxygen in the cold with the evolution of heat and the formation of ceric hydroxide.

—W. G.

*Mesothorium to thorium; Ratio of —.* H. N. McCoy and L. M. Henderson. J. Amer. Chem. Soc., 1918, 40, 1316–1326.

THE term "1 mgrm. of mesothorium" is applied to the quantity of  $\text{Ms}_1 + \text{Ms}_2$  in equilibrium which has a  $\gamma$ -ray activity equal to that of 1 mgrm. of radium in equilibrium with its first four products. The  $\gamma$ -activity was measured through 2.03 mm. of lead+1.32 mm. of brass. The results of the research show that "1 mgrm. of mesothorium" is in equilibrium with 19 kilos. of thorium in minerals, or 1 gm. of thorium is in equilibrium with  $0.524 \times 10^4$  mgrm. of mesothorium. This last ratio is only one sixth as great as the ratio of radium to uranium, namely  $3.23 \times 10^4$  mgrm. of radium per 1 gm. of uranium. (See further J. Chem. Soc., Dec., 1918.)—F. S.

*Silicic acid gels.* H. N. Holmes. J. Phys. Chem., 1918, 22, 510–519.

INSTRUCTIONS are given for preparing silicic acid gels from water-glass and a long series of acids. In addition the concentrations and quantities of the various acids necessary to produce gels which will set in a definite period are also determined. (See J. Chem. Soc., Dec., 1918.)—J. F. S.

*Gallium.* Dennis and Bridgman. See X.

*By-product lime.* Kosmann. See XVI.

#### PATENTS.

*Sulphuric acid; Manufacture of —.* A. B. Foster, Washington, D.C. U.S. Pat. 1,277,896, 3.9.18. Appl., 23.7.14.

SULPHUR-BEARING material is burned in a limited supply of air, and the gas—on its way to and from the Glover tower—is subjected to the action of atomised chamber acid which is injected by means of air blasts, and in the direction of the gas flow, at a number of points along the connecting flues, and then removed. The chamber gases are similarly treated.—W. E. F. P.

*Sodium and aluminium hypochlorites; Process for the manufacture of —.* E. E. and P. C. Dutt, Jubbulpore, India. Eng. Pat. 119,261, 22.8.17. (Appl. 12,058/17.)

A MIXTURE of bauxite (or other suitable aluminous material), calcium hydroxide, sodium chloride, and carbon is heated to redness in the presence of arsenic trioxide, whereby sodium aluminate, calcium carbonate, arsenic trichloride, hydrogen, and carbon dioxide are produced. The product is lixiviated with water, and one-half of the solution

obtained is treated with carbon dioxide to precipitate aluminium hydroxide which, after separation, is suspended in the remaining half of the liquid. This mixture is treated with chlorine until the suspended aluminium hydroxide is dissolved, after which the solution is first neutralised with sodium hydroxide (to precipitate sodium chloride, which is removed) and then treated with a further quantity of this reagent to produce a solution of sodium hypochlorite and precipitate the whole of the aluminium as hydroxide. The latter precipitate is separated, mixed with water, and treated with chlorine to produce a solution of aluminium hypochlorite.—W. E. F. P.

*Nitre cake; Method for the utilisation of* — [in the production of hydrochloric and nitric acids]. J. Grossmann, Manchester. Eng. Pat. 119,290, 28.9.17. (Appl. 13,975/17.)

In the production of nitric and hydrochloric acids by heating sodium nitrate and sodium chloride with nitre cake to about 250° C. and 450° C. respectively, the finely powdered materials are mixed with coke or other carbonaceous material to render the residual salt-cake friable and easily removable. The reducing action of the coke is minimised by regulating the proportion and size of the fragments employed.—W. E. F. P.

*Manganese dioxide; Process of separating* — from ore containing the same. Separator for separating manganese dioxide from ores containing the same. L. E. Sowers and P. S. Phillips, Livermore, Cal. U.S. Pats. (A) 1,277,144 and (N) 1,277,145, 27.8.18. Appls. 12.9 and 30.10.17.

(A) The dry, powdered ore is fed into a vessel in which an upward stream of water is maintained, and the lighter particles are removed by peripheral overflow. (B) The apparatus consists of a funnel-shaped vessel provided with a water inlet at the bottom, a peripheral trough at the top, and a central spindle upon which are fixed in succession, from the water level downwards, a conical distributor (partly submerged), a pair of horizontal paddles or vanes, and a helix. The latter is disposed within the funnel stem, and serves to promote uniformity of flow in the upward stream of water.—W. E. F. P.

*Alkali-metal-bearing and aluminiferous minerals; Decomposition of refractory* —. [Extraction of potash from felspar.] W. H. Swenarton, Montclair, N.J. U.S. Pat. 1,277,773, 3.9.18. Appl. 23.3.12.

A mixture of the material with an "alkali flux" is heated, and the mass is lixiviated with water and the insoluble matter separated. The solution and the residue are then treated separately with sufficient acid to effect decomposition and produce a neutral reaction, whereby alumina and silica are precipitated simultaneously and a solution of a potassium compound obtained in each case.

—W. E. F. P.

[Sodium] cyanide; Crude — and process of producing the same. Electric furnace [for making sodium cyanide]. Process of producing sodium cyanide. H. Freeman, Niagara Falls, Ont., Assignor to American Cyanamid Co., New York. U.S. Pats. (A) 1,277,898, (N) 1,277,899, and (C) 1,277,900, 3.9.18. Appls., 29.11.16, 20.4, and 23.4.18.

(A) An intimate mixture of calcium cyanamide, sodium chloride, and finely divided (chemically active) carbon—the latter having been previously heated to not more than 1000° C.—is heated to a temperature sufficient to produce sodium cyanide.

Crude products obtained by this means, and containing more than 20% and more than 32% by weight of sodium cyanide respectively, are claimed. (B) An electric furnace for making sodium cyanide from "lime nitrogen" (crude calcium cyanamide) and sodium chloride has a hearth of conducting material (which forms the lower electrode) and a funnel-shaped shaft, at the apex of which a small crucible chamber is situated within the hearth. The upper graphite electrode depends within the shaft, is adjustable vertically, and serves to restrict the passage of the charge from the shaft to the crucible chamber. A tap-hole is provided for removing the sodium cyanide produced. (C) A mixture of calcium cyanamide, sodium chloride, and carbon or calcium carbide is fed continuously into the shaft of the furnace described under (B), and through the restricted portion of the mixture within the crucible chamber a current density is maintained sufficient to produce sodium cyanide without causing foaming of the charge.—W. E. F. P.

*Sulphur; Separation of* — from gases. F. Muhler, Göttingen. Ger. Pat. 306,988, 14.4.16.

The gas, freed from cyanogen and ammonia, is treated with a dilute solution of a copper salt (e.g. copper sulphate), copper sulphide and dilute sulphuric acid being formed. The copper sulphide is separated and heated strongly in a current of air with the production of copper oxide and sulphur dioxide. The copper oxide is dissolved in the dilute acid produced in the first reaction, and the resulting solution used for the treatment of further quantities of gas.—L. A. C.

*Hydrogen sulphide; Method for the removal of* — from gases. W. Diekmann, jun., Duisburg. Ger. Pat. 308,107, 6.10.16.

The purifying material is used in the form of compact cubical, spherical, laminar, etc. portions, these being formed at a temperature which does not destroy the purifying property of the material. The sulphur is washed out of the purifying material by a solvent or vapour. The advantages claimed for the method are that it is extremely simple, the purifying agent is not removed and does not deteriorate in value, the material can be used until saturated with cyanogen, and the sulphur absorption and recovery take place in the same vessel.

—J. S. G. T.

*Liquors containing potassium salts, etc.; Apparatus for cooling and evaporating* —. E. Plinkenburger, Mülheim. Ger. Pat. 307,483, 20.9.13.

The liquors are allowed to trickle over hurdles, laths, or the like stacked in a closed apparatus and arranged around one or more shafts closed at the top. The interior of the apparatus is readily accessible from the shafts, and the hurdles, laths, etc., which are removable, can be easily cleaned.

—L. A. C.

*Cryolite; Manufacture of* — from sodium bisulphate, silica, and fluorspar. Chem. Fabr. Goldschmieden H. Bergius and Co., Goldschmieden. Ger. Pat. 307,525, 23.1.17.

Silicofluoride, produced by decomposition at red heat of fluorspar and silica by means of sodium bisulphate, is absorbed by solid hydrated sodium sulphate, and, after separation of sulphuric acid or sodium bisulphate, the sodium silicofluoride is decomposed by fusing it with crystalline sodium carbonate in the proportion of 1 mol. of sodium silicofluoride to 4 mols. of sodium carbonate, with the production of sodium fluoride, sodium bicarbonate, and silica. The silica is filtered off, and



cryolite precipitated by addition of sodium aluminate to the solution. The following equations represent the reactions which take place: (1)  $2\text{CaF}_2 + 4\text{NaHSO}_4 + \text{SiO}_2 = \text{SiF}_4 + 2\text{Na}_2\text{SO}_4 + 2\text{CaSO}_4 + 2\text{H}_2\text{O}$ ; (2)  $3\text{SiF}_4 + 2\text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O} = 2\text{Na}_2\text{SiF}_6 + 2\text{H}_2\text{SO}_4 + \text{SiO}_2$ ; (3)  $\text{Na}_2\text{SiF}_6 + 4\text{Na}_2\text{CO}_3 + 10\text{H}_2\text{O} = 6\text{NaF} + \text{SiO}_2 + 4\text{NaHCO}_3 + \text{SH}_2\text{O}$ ; (4)  $6\text{NaF} + 4\text{NaHCO}_3 + \text{NaAlO}_2 = (\text{NaF})_3\text{AlF}_3 + 4\text{Na}_2\text{CO}_3 + 2\text{H}_2\text{O}$ .  
—L. A. C.

*Diaphragms for electrolysis.* Ger. Pat. 307,471.  
See XI.

### VIII.—GLASS; CERAMICS.

*Refractory ware; Production of highly — from non-argillaceous materials, particularly zirconia.*  
H. Arnold. Chem.-Zeit., 1918, 42, 413—414, 426—428, 439—440.

Pure zirconia melts at about 2600° C., has a coefficient of expansion of  $8.4 \times 10^{-7}$  (quartz,  $7 \times 10^{-7}$ ), is highly insensitive to sudden changes in temperature, and has an electrical conductivity of 0.0008 at 1200° C. Zirconia readily dissolves other oxides, and the electrical conductivity at 1200° C. is increased to 0.00255 recip. ohm. by adding 1 mol.  $\text{Al}_2\text{O}_3$  to 9 mols.  $\text{ZrO}_2$ , to 0.0075 at 1000° C. if the alumina is replaced by  $\text{CeO}_2$ , and to 0.0358 at 1287° C. if replaced by  $\text{Fe}_2\text{O}_3$ . Crude zirconia is a good conductor. The author finds that the use of any fluid in pressing zirconia ware is detrimental, and recommends (Ger. Pat. 285,934; U.S. Pat. 1,121,889; this J., 1915, 231) pressing the cold zirconia dry at 300 atm. or the hot material at 50 atm. A mixture of raw and highly calcined zirconia should be used (as it shrinks less than the raw zirconia, which polymerises at 1600°—1760° C.), and as soon as the articles are dry they may be burned in an electric furnace at 2000°—2300° C., only about 1 hour being required for crucibles 2 in. high and  $1\frac{1}{2}$  in. diam. The addition of a small percentage of alumina or clay is advantageous, but magnesia and silica make the ware too porous. The importance of grading the zirconia into particles of suitable sizes appears to have been overlooked. Articles made of zirconia may be cast in plaster moulds, using a slip composed of graded zirconia and colloidal zirconia sol, the latter being prepared by repeatedly evaporating zirconium nitrate with 20 times its weight of water. The speed of translation of the particles of colloidal zirconia sol in an electric field was measured by the author: with a potential difference of 110 volts it averaged  $2.2 \times 10^6$  cm. per second, for 1 volt with electrodes 1 cm. apart. The speed is seriously affected by electrolytes in the zirconia, and material to be used for casting should be washed free from negative electrolytes. According to Podszus (Eng. Pat. 11,771 of 1912; this J., 1913, 792) the particles of colloidal zirconia must not exceed 0.005 mm. diameter, and some particles not exceeding 0.1  $\mu$  to 1  $\mu$  should be present. The plasticity of zirconia slip and the ease with which it leaves the mould are related to its electrical properties. The proportion of water in the slip must be regulated according to the rate at which it is to be absorbed by the mould. Thus, for articles of complex shape a "thin" slip is preferable, as it is absorbed more slowly and there is less risk of the article splitting. Non-porous moulds may be used if they have a low coefficient of expansion and are lined with wax or other readily fusible or soluble material which may be removed before the article. For complex shapes a nickel mould coated internally with paraffin wax is suggested; to release the article from the mould the wax is melted and poured out. Ware with walls only a fraction of a mm. thick may be cast in this manner. Zirconia ware should be supported

on discs of carbon or zirconia and burned in an electric resistance furnace at 2000° C.; if burned at a higher temperature, its porosity is increased by the volatilisation of a further part of the impurities. Ware burned at 2000° C. is bluish-grey, at 2200° C. reddish-brown, and at 2400° C. bluish-black owing to absorption of carbon and the formation of carbide. The blue colour appears to be due to titanium oxide, the reddish colour to zirconium nitrocarbide. Pieces of zirconia ware which have been burned under slight reducing conditions have a handsome purple brown colour, and, when polished, resemble black marble. They are very strong, and may be dropped on a hard floor with impunity. If reheated at 1000° C. in a muffle, the colour changes to a yellowish-white, and the expansion due to reoxidation makes the ware porous and liable to spall. Vessels of clay or other refractory materials may be lined or coated externally with zirconia, but such a method of protection has only a limited applicability. Zirconia ware has not hitherto been glazed satisfactorily; a glaze composed of felspar 22.4, alumina 40.8, zirconia 147.6, magnesite 50.4, and quartz 69.6 parts burned on the ware at 1900°—2000° C., is fairly satisfactory and does not craze, but is rather matt and liable to blister. There is a strong tendency for the zirconia to combine with glazes melting at a lower temperature, though when the product is sufficiently refractory they may be used.—A. B. S.

#### PATENTS.

*Plate glass; Pot for the manufacture of — and method of making the same.* C. H. Kerr, Tarentum, Pa., Assignor to Pittsburgh Plate Glass Co. U.S. Pat. 1,278,164, 10.9.18. Appl., 18.12.16.

PLASTIC refractory clay is thoroughly mixed with from 15% to 50% of silica, which is so finely divided that the major portion of it consists of grains less than 0.005 in., preferably less than 0.0025 in. diameter.—B. V. S.

*Kiln for ceramic ware.* O. von Horstig, Saarbrücken. Ger. Pat. 306,726, 3.7.14.

In a zig-zag ceramic kiln the end chambers are connected by a canal, which passes over or under the other chambers, and is provided with firing doors and adapted to receive articles to be baked.  
—J. H. L.

*Porcelain and other clay wares; Half-gas firing for smokeless burning of —.* J. Popp, Bunzlau. Ger. Pat. 307,902, 29.9.16.

THE ware is burned in an oven fitted with deep vertical fire-boxes with sloping, water-cooled grates which project beyond the fire-boxes. The grates are covered by narrow slides which are moved reciprocally by mechanical means. By this means the production of smoke and clinker is avoided even when low-grade fuel is used. The absence of irregular stoking, heavy poking, and soot results in a considerable saving in fuel and labour.—A. B. S.

### IX.—BUILDING MATERIALS.

*Calcium aluminium sulphate as a concrete destructor.* Nitzsche. Z. angew. Chem., 1918, 31, 195—196.

A DISCUSSION of the formation of the double sulphate of calcium and aluminium in cement and its deleterious action. The compound is always formed when a solution of a sulphate is brought into contact with lime water and precipitated alumina, but it is

only stable in presence of free lime. Most natural waters contain sulphate and consequently attack cement, but sea water appears not to lead to the formation of the double calcium aluminium sulphate. The preventive measures recommended are the coating of the cement with bituminous material, or the modification of the constitution of the cement in such a manner as to prevent the formation of the double sulphate.—E. H. R.

*Silicic acid gels.* Holmes. See VII.

#### PATENTS.

*Cement shaft-kilns; Apparatus for loading — and pre-drying the material.* Lothringer Portland Cement Werke, Strassburg, Alsace. Ger. Pat. 306,514, 23.1.17.

Inside the superstructure of the kiln, and exposed to the hot gases from the latter, is a hopper situated centrally above the kiln shaft. The material delivered by this hopper falls against the sloping sides of a hollow cone, also situated centrally, base downwards, at a somewhat lower level. The lower portion of the sides of this cone is formed of grids which allow the smaller particles to pass through, so that they fall in the central part of the shaft, whilst the larger pieces are deflected by the cone towards the kiln walls. This prevents caking of the finer material on the walls.

—J. H. L.

*Portland cement; Production of — from raw materials with hydraulic properties.* N. S. Borch, Copenhagen. Ger. Pat. 306,653, 15.12.17. Int. Conv., 24.2.17.

In the wet process of making cement from hydraulic materials, the slurry is prevented from setting by the addition of certain substances, e.g., 0.25% of sucrose or various hydroxy-acids, aldehydes, or ketones, which of course are subsequently destroyed in the kiln. The process is applicable to the manufacture of cement from blast-furnace slag or water-granulated blast-furnace slag.—J. H. L.

*Stones and plates; Production of artificial — containing cement.* H. Petersen, Eckenforde-Borby. Ger. Pat. 306,952, 5.8.17.

CEMENT is mixed with air-dry finely ground marl.

—J. H. L.

*Stones; Production of artificial polishing — for lithographic stones, marble, etc.* R. Friedrich and J. Wimmer, Munich. Ger. Pat. 306,975, 25.7.17.

COAL tar pitch is heated with powdered pumice and glass and the pasty mixture cooled in moulds.

—J. H. L.

*Portland cement; Process of manufacturing — of raw materials with hydraulic properties.* N. S. Borch, Copenhagen. Eng. Pat. 113,785, 19.2.18. (Appl. 2958/18.) Int. Conv., 24.2.17.

See Ger. Pat. 306,653: preceding.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Slag control in the iron blast furnace by means of slag viscosity tables.* A. L. Feild. Chem. and Met. Eng., 1918, 19, 294—300.

THE diminution of slag viscosity with increase of temperature is shown in the form of a curve, and

by means of this it may be demonstrated that a minor change in percentage of one of the principal constituents—lime, alumina, and silica—may cause a great change in the temperature-viscosity relations. A gradual increase in the amount of lime, instead of producing a gradual change in viscosity, produces maxima and minima. Changes in percentages of minor constituents of a slag, such as oxides of magnesium, manganese, iron, etc., do not appreciably affect temperature-viscosity relations if the percentage of magnesia, the most important minor constituent, does not exceed 8%. The temperature relations of the slag are said to be most suitable with a viscosity ranging from 10 in the furnace to a viscosity of 4 where the slag leaves the furnace. In controlling the slag between these limits, it is shown that all calculations of slag analyses must be made on a basis of 100% of lime, alumina, and silica; lime must be accurately determined by analysis and not by difference, as slag properties are particularly susceptible to slight differences in the lime content. With a given supply of raw materials—ore, coke, and limestone—the economy in coke consumption for a specified grade of iron is chiefly dependent on the maintenance of a proper slag composition. Examples are given of the necessary calculations, with ores of known percentage composition, showing how the amount of limestone per pound of pig iron of a definite composition may be calculated so as to arrive at a slag of the proper viscosity by making use of the slag-viscosity tables. (See also this J., 1918, 61 A, 246 A, 548 A.)—B. N.

*Cast iron; Wearing and antifrictional qualities of —.* J. E. Hurst. Iron and Steel Inst., Carnegie Scholarship Memoirs, 1918, 9, 59—76.

WEAR in commercial grey cast iron appears to be brought about by a process of surface disintegration, individual grains of hard eutectic being detached bodily. This debris may be largely instrumental in bringing about "seizing" in moving parts. Subsequently to this stage the plastic constituents flow into the cavities formed, and yield a well-glazed surface of excellent antifrictional qualities, with harder particles standing up in slight relief. The iron should not be too "open" in texture. There does not appear to be any relationship between wearing properties in cast iron and its Brinell hardness. The casting temperature of the iron is said to affect the antifrictional qualities, but in what way is not stated. Two machines designed to afford laboratory tests on the wearing properties of samples of cast iron are described.

—F. C. Th.

*Steel manufacture; Balanced reactions in —.* A. McCance. Faraday Soc., Nov. 12, 1918. [Advance proof.] 11 pages.

DURING the melting process in the open-hearth furnace, as the temperature becomes lower there is greater oxidation of the scrap and more FeO in the slag, as following analyses show:—

	FeO	SiO <sub>2</sub>
" Cold heat " ... ..	36.3	36.4
" Hot heat " ... ..	20.8	45.0

It is suggested that this oxidation is largely due to steam in the burnt producer gases:  $3\text{Fe} + 4\text{H}_2\text{O} = \text{Fe}_3\text{O}_4 + 4\text{H}_2$ . Thus to minimise loss due to scaling it is necessary to melt down quickly and at a high temperature and to have a low steam content in the burnt gases. So long as the bath of steel lies under a covering of slag containing ferrous oxide, this will be present in solution in both steel and slag, the ratio of the two concentrations being always constant. The ferrous oxide in solution in the steel reacts with the carbon also present forming



carbon monoxide, which, when the metal has become supersaturated, is evolved. So long as no new ore additions are made to the bath a straight line is obtained when the logarithm of the carbon in the bath is plotted against the time. The volume of gas present in deoxidised steel is greater than that in steel not so treated, and further the  $\text{CO}_2 : \text{CO}$  ratio is much greater in the deoxidised material.—F. C. Th.

*Steel; Gases occluded in* — T. Baker. Faraday Soc., Nov. 12, 1918. [Advance proof.] 4 pages.

THE gases evolved when samples of steel were heated *in vacuo* to about  $1000^\circ \text{C}$ . were collected, measured, and analysed, the results being summarised in the following table:

Description	Vol. of gas in c.c. per grm. of steel	Composition				
		$\text{CO}_2$	$\text{H}_2$	$\text{CO}$	$\text{CH}_4$	$\text{N}_2$
Sound steel, 0.9% C ...	1.32	1.68	52.00	45.53	0.72	0.07
Do. reheated ...	1.40	1.16	49.55	45.99	2.71	0.59
Steel with blow-holes ...	0.66	0.88	54.56	42.36	1.73	0.47
Soft ingot ...	1.03	1.18	52.12	45.64	0.73	0.33
Bar from soft ingot ...	0.53	0.91	49.08	43.12	0.11	1.77

The steel with blow-holes contains less gas than does the sound steel. Reheating to  $1200^\circ \text{C}$ . does not appreciably alter either the volume or the composition of the gases. Forging reduces the volume of occluded gas considerably, but does not result in any marked alteration in the composition. With hard steels hydrogen reaches a maximum rate of evolution at  $600^\circ \text{C}$ ., and below this temperature constitutes the greater part of the gas given off. Carbon monoxide is slowly evolved from the start, and reaches its maximum rate of evolution at  $688^\circ \text{C}$ . With soft steels hydrogen again forms the greater part of the gas collected up to  $660^\circ \text{C}$ ., and presents a maximum at  $609^\circ \text{C}$ . The carbon monoxide is evolved most quickly at  $786^\circ \text{C}$ ., at which temperature a second maximum in the rate of evolution of the hydrogen occurs.—F. C. Th.

*Strain in iron and steel; Chemical detection of* — by their reaction with nitric acid. J. H. Whiteley and A. F. Hallimond. Iron and Steel Inst., Carnegie Scholarship Memoirs, 1918, 9, 1—56.

THE final reduction products obtained when iron is dissolved in dilute nitric acid are chiefly nitrogen peroxide, nitric oxide, nitrogen, and ammonia, depending on the acid-strength and the degree of cold work put upon the iron. At an acid-strength of 24 grms. per 100 c.c. the reaction undergoes a marked change, much of the nitrogen and ammonia being replaced by oxides of nitrogen. By plotting as ordinate the weight of iron consumed in forming nitrogen and ammonia and as abscissa the acid-strength, a characteristic "reaction curve" is obtained. Progressively increasing strain in the sample results in a progressive shifting to the right of the "reaction curve." Strained iron subsequently annealed at  $520^\circ \text{C}$ . becomes from this point of view unstrained. Differences in grain size in annealed pure iron do not produce perceptible differences in the reaction curves.—F. C. Th.

*Iron; Protection of* — with paint against atmospheric corrosion. J. N. Friend. Iron and Steel Inst., Carnegie Scholarship Memoirs, 1918, 9, 77—123.

ACCELERATION tests are of little value. Reliable results can only be obtained from tests carried out under practical conditions. Addition of pigment to

oil increases the efficiency of the latter as a protective agent up to a maximum, after which further addition of pigment causes deterioration. The best results are obtained from paints possessing as high a percentage of good oil as is compatible with a good body. Linseed oil expands on setting, which results in crinkling and cracking. Linoxyn is permeable to moisture, the permeability being reduced by heating in absence of air. Polymerised linseed oil affords better protection than raw oil. A thick coat of paint, so long as it is not so thick that crinkling occurs, protects the metal better than a thin one. The best results are obtained by multiple coats, two thin ones being better than one thick one. Turpentine has little effect on the efficiency of a paint. The most permanent paints are those containing black and red pigments. Fine pigments afford more efficient protection than coarse pigments. Iron should be painted while the scale is still on, with loosely adherent flakes scraped off. The paint will then last longer than if applied to the pickled or sand-blasted surface. Experiments with rusty plates, though not conclusive, suggest that rust need not be so completely removed prior to the painting as is usually thought to be necessary.—F. C. Th.

*Tinplate; Metallographic examination of* — L. Mayer. Stahl u. Eisen, 1918, 38, 960—962.

THE softness of metallic tin made it necessary repeatedly to etch and polish the surface to be examined before a satisfactory specimen could be obtained. Very dilute (2%) nitric acid is the best etching medium; picric acid does not attack tin, but may be used on the tin-free iron surface. The surface of the specimen was first electroplated with copper; a portion of the surface was then polished, etched with the dilute nitric acid to display the tin crystals, and finally with an alcoholic solution of picric acid to display the ferrite and pearlite in the iron. The tin crystals were small and elongated; no indication of any compound of tin and iron was found, though the thickness of the tin coating varied from 0.002 to 0.01 mm. Small iron cubes measuring  $10 \times 15 \times 20$  mm. were polished, pickled in dilute hydrochloric acid, washed with distilled water, dried in a bath of zinc chloride, and then tinned by immersion for half an hour at  $300^\circ$ ,  $500^\circ$ ,  $750^\circ$ , and  $950^\circ \text{C}$ . respectively. On examining the specimens under the microscope, there appeared to be a layer of intermediate crystals which increased in thickness as the temperature of the bath increased. At  $750^\circ \text{C}$ ., with low-carbon iron and at  $500^\circ \text{C}$ . with high-carbon iron, a reduction in the amount of pearlite occurred in the layer adjacent to the tin coating. At  $950^\circ \text{C}$ ., pearlite again appeared at the edges of the tin coating; with the purest iron, the formation of small crystals within the larger ferrite crystals was observed in the neighbourhood of the tin coating. The diffusion of tin into iron may be shown indirectly by the behaviour of pearlite and ferrite at high temperatures. (See also J. Chem. Soc., Dec., 1918.)

—A. B. S.

*Lead in tin-plate; Volumetric determination of* — J. B. Deininger. Z. Unters. Nahr. Genussm., 1918, 36, 66—67.

ABOUT 0.5 gm. of the metal is heated with 10 c.c. of concentrated sulphuric acid until metallic particles are no longer visible; after cooling, 20 c.c. of 5% ammonium oxalate is added and the mixture again heated in order to dissolve remaining traces of iron. Alcohol is then added, the lead sulphate collected, washed with dilute alcohol, then dissolved in 15 c.c. of hot 50% sodium acetate solution, and bromine water added, drop by drop, to the warm

solution until a slight excess is present. After 5 mins., the precipitated lead peroxide is collected, washed with hot water, rinsed into a beaker with cold saturated sodium acetate solution, and treated with 20 c.c. of 5% potassium iodide solution and 10 drops of acetic acid; the lead peroxide decomposes rapidly, and the liberated iodine is titrated with thiosulphate solution. Each c.c. of  $N/10$  thiosulphate solution is equivalent to 0.01036 gm. Pb. The lead sulphate obtained by the treatment with sulphuric acid contains tin, and if weighed directly the results obtained are much too high.

—W. P. S.

*Soft iron and copper; Hardness of — compared.*  
F. C. Kelley. Amer. Electrochem. Soc., Sep.-Oct., 1918. [Advance copy.] 4 pages.

By annealing samples of American "ingot iron" and ordinary commercial cold-rolled copper similarly, in hydrogen and in a vacuum, at temperatures between 770° and 950° C., the Brinell hardnesses of the metals as received were reduced from 95 and 80 to 60 and 40, respectively. It is suggested that such dead soft iron—which, in the present case, could be whittled with a knife—might find uses in place of pure soft copper.

—W. E. F. P.

*Brass melting; Present status of electric —.*  
H. M. St. John. Chem. and Met. Eng., 1918, 19, 321—328.

It was estimated by Gillett, in 1914, that the value of the metal employed in brass and bronze melting in the United States was \$120,000,000 per annum, whilst the value of the metal lost during the melting operation was \$3,000,000. Most of the developments in electric furnace melting have been designed to eliminate much of this loss. The advantages which may be expected to accrue from electric melting are metal saving, improved quality, exact temperature control, increased production, elimination of crucible cost, other incidental savings, and better working conditions. The principal requirements in electric melting are uniform heating of the metal, uniformity in composition on pouring, and a high thermal efficiency consistent with good metallurgical results; and the electrical characteristics of the furnace must be such as to make it a desirable load for the central station company or the factory power plant. Of the four types of electric furnace available, the vertical ring induction furnace is of high efficiency, limited in its application, but within its limited range gives satisfactory results. The indirect-resistance, indirect-radiation furnace is less efficient, has a lower rate of production in proportion to its holding capacity, but is more flexible and better suited to general foundry use, though neither form is entirely satisfactory. The indirect-arc is more efficient than the indirect-resistance, and more flexible within its own field; it may be advantageously used for alloys with no zinc, or with only a small percentage. The most promising possibilities are stated to be in an induction furnace not only efficient but flexible and of wide-spread application, or in a form of arc furnace which will be applicable to yellow brass and other high-zinc alloys, as well as to the less sensitive alloys. Electric crucible furnaces, though reliable and effective, are hardly likely to be profitable. The application of electricity to brass-melting, from the central station viewpoint, is discussed, and it is stated that the power company should be familiar with the electrical and operating characteristics of the different furnaces in use, so as to prevent mis-applications, whilst pointing the way to correct use and ensuring profitable development.—B. N.

*Antimony; Electrolytic refining of —.* [Extraction from low-grade ores.] Y. C. Wong. Chem. and Met. Eng., 1918, 19, 509.

LOW-GRADE ores are not a commercial source of antimony at the present time; but during the last 30 years processes depending on the treatment of such (sulphide) ores with a solvent (sodium sulphide, etc.), and the recovery of metallic antimony from the solution by electrolysis, have been used more or less successfully. Siemens and Halske (Eng. Pat. 18,966 of 1892; this J., 1893, 847) in 1892, and von Engelhardt and Nettel in 1896, employed as solvents solutions of alkaline-earth sulphides which were subsequently regenerated with only small losses; and from these solutions 0.621 kilo. of antimony per kilowatt-hour (representing an efficiency of 76%) was deposited with a current density of 0.8 amp. per sq. cm. at 0.8 volt. In laboratory tests made by the author on the electrolysis of calcium thioantimony salts in aqueous solutions, an anode of platinum wire in the form of a helix was surrounded by a cylindrical cathode of platinum foil having an exposed surface of 39.3 sq. cm. With a current of 0.4 amp. at 1–1.2 volts, 90% of the antimony was deposited in one hour from about 450 c.c. of hot solution containing 0.962 gm. of the metal. The cost of extraction by this process is estimated at 5c. (2½d.) per lb. of metal recovered.—W. E. F. P.

*Pyrophoric alloy industry; The American —.* A. Hirsch. Chem. and Met. Eng., 1918, 19, 510—512.

The history of the industry is briefly outlined, with special reference to the production of metallic cerium and pyrophoric alloys in the U.S.A. by the New Process Metals Co., operating under U.S. Pat. 1,273,223 (see this J., 1918, 591A). According to this process, so-called metallic cerium ("Mischmetall") is prepared from the mixed rare-earth metal oxides obtained as a by-product in the extraction of thorium from monazite. The mixed oxides (mainly cerium oxide) are dissolved in hydrochloric acid and the solution after purification is evaporated to dryness; the residue is fused and electrolysed in cast iron pots which are heated when necessary during the operation. The pot constitutes the cathode of the cell, the anode being a rod of carbon or graphite; each of the latter materials has a critical current density (5½ and 6–7 amp. per sq. in. of surface, respectively) above which current may pass without deposition of metal. To secure a desirable electrical heating effect, the current density maintained at the anode is from 3 to 4 times that at the cathode. At the conclusion of the electrolysis, the charge is allowed to cool in the pot, and the latter is broken to remove the button of "Mischmetall," which is then alloyed with about 30% of other metals (chiefly iron) to produce the commercial sparking metal or pyrophoric alloy.—W. E. F. P.

*"Ulco" hard metal [lead alloy].* F. C. Frary and S. N. Temple. Chem. and Met. Eng., 1918, 19, 523—524.

"Ulco" hard metal, originally designed as a substitute for antimonial lead, is an alloy of lead with considerably less than 1% of calcium or/and other alkaline-earth metals. It is much harder than other commercial lead alloys, and expands on solidification, forming castings free from blow-holes. The Brinell hardness of antimonial lead is about 17; that of chilled castings of Ulco metal varies from about 22 to 26, according to the temperature of pouring, and the alloy can be remelted several times without appreciable loss of hardness. Ulco metal has the general appearance and mechanical properties of ordinary lead, and may



be diluted with the latter, or with tin or bismuth, to produce softer alloys the hardness of which increases on "ageing" for a few days; but the addition of antimony causes separation of the alkaline-earth metal. In comparative tests made by the U.S. Bureau of Standards on machinery bearings of genuine Babbitt metal and Ulco hard metal, the latter gave highly satisfactory results. The Babbitt metal had a tensile strength of 10,000 lb. per sq. in. and, on 1 inch, gave an elongation of 15% and a reduction of area of 8%. The corresponding figures for the lead alloy were 13,000 lb., 5%, and 1%, respectively, the average elastic limit in compression being 11,525 lb. per sq. in. The alloy is also recommended for use in the production of small castings at present made from brass.—W. E. F. P.

*Alloys; Determination of the composition of binary — by specific gravity determination.* M. von Schwarz. *Bayr. Ind. u. Gewerbebl.*, 1918, 104, 61–63. *Z. angew. Chem.*, 1918, 31, Ref., 310.

In determining the specific gravity of alloys by Matthiessen's formula,  $d = 100 / \left( \frac{A}{a} + \frac{B}{b} \right)$ , where A

and B are the percentages of the two metals in the alloy and *a* and *b* their specific gravities, an allowance must be made for the alteration in density due to contraction and in a few cases for that due to expansion. The method of using the sp. gr. as a means of determining the composition finds practical application principally for tin-lead and antimony-lead alloys, of which samples of definite volume can be cast and their weight compared with those of similar substances of known composition on the so-called tin assay balance. Instead of the usual disc or conical samples, balls are now used which can be obtained uniform and of an exact volume. Using balls of a volume of 1.62 c.c. and weighing to 0.01 grm., the composition of tin-lead or antimony-lead alloys can be obtained from the sp. gr. curves with an accuracy within the limits of 0.5–1.0%.—W. G.

*Gallium.* L. M. Dennis and J. A. Bridgman. *J. Amer. Chem. Soc.*, 1918, 40, 1531–1561.

SPARK spectra are suitable for detecting amounts of gallium above 0.0046 mgrm. and amounts of indium above 0.0013 mgrm. In the presence of gallium, 0.06% of its weight of indium can be detected by means of the spark spectrum and 0.18% of gallium can be detected in the presence of indium. Fractional electrolysis, after 14 operations, yields pure gallium from a mixture of gallium and indium, whilst fractional distillation of a mixture of the chlorides of gallium, indium, and zinc yields pure gallium chloride. Gallium is best estimated by precipitation from slightly acid solutions as hydroxide by means of sodium sulphite. Mixtures of gallium and zinc are separated and estimated by adding an excess of potassium mercuric thiocyanate to a slightly acid ( $H_2SO_4$ ) solution of the sulphates. After keeping for several hours the precipitate of zinc mercuric thiocyanate is collected in a Gooch crucible, dried at 105°–110° C., and weighed. The filtrate is acidified with hydrochloric acid and the mercury removed by hydrogen sulphide; after boiling, the gallium is precipitated as hydroxide by means of sodium sulphite. Mixtures of gallium and indium are separated and estimated as follows: Solutions containing small amounts of both elements are largely diluted and treated with a small amount of hydrochloric acid and exactly neutralised with sodium hydroxide, an excess of 1.5 grms. of sodium hydroxide is added, and the solution boiled for

several minutes. The precipitated indium hydroxide is well washed, dissolved in hydrochloric acid, and the process repeated. Finally it is dissolved in hydrochloric acid, precipitated by ammonia, washed, dried, ignited, and weighed as oxide. The filtrate and washings from each precipitation are combined and the gallium precipitated as before. The separation of gallium and aluminium is effected in the following way: The aluminium is precipitated as hydrated chloride by adding 60 c.c. of hydrochloric acid to the solution and then 60 c.c. of ether. The flask is immersed in cold water and the solution saturated with hydrogen chloride, when hydrated aluminium chloride is precipitated. This is collected in a Gooch crucible, washed with a mixture of 30 c.c. of hydrochloric acid and 30 c.c. of ether and then dissolved in water and the aluminium estimated in the usual way. The combined filtrates and washings are treated with a small amount of sulphuric acid and boiled to expel ether and most of the hydrochloric acid, and the gallium estimated as before. Methods are given for the estimation of mixtures of gallium, indium, and zinc, and gallium, indium, zinc, and aluminium. (See also *J. Chem. Soc.*, Dec., 1918.)—J. F. S.

*Electric welds.* E. E. Thum. *Chem. and Met. Eng.*, 1918, 19, 301–308.

MACHINES are described for use in butt and spot welding, in which increased speed is obtained in the operation, and the formation of undesirable oxides is avoided. Considerable trouble was at first experienced in spot welding a small inset of high-speed steel into a carbon-steel tool, through the breaking away of the tool steel, but this was afterwards prevented by slowly annealing the material in lime in a hot oven and later hardening. To study the chemical and physical changes induced by the welding process, pieces of drill steel, 0.97% carbon, were butt welded and afterwards turned, tested for ultimate strength, and examined metallographically. The unannealed welded bars were found to have a higher ultimate strength than the original bar, and in the annealed bars the marked increase in ductility near to the weld suggested a change in the carbon content; metallographic examination showed a decarburised zone, and this was confirmed by analysis. A welded bar, not heat treated, will break at some distance from the joint under static load, but at the weld if tested under sharp, eccentric impact. After heat treatment, exactly the opposite will occur, as the joint will then be the weakest and most ductile part of the bar due to local decarburisation. Photomicrographs are shown illustrating the changes that take place in various types of steel in spot and butt welding, and showing the development of a eutectiform structure in spot welds. The eutectiform appearance has been shown to be identical in nature with the Widmanstätten bands, consisting of a concentration of excess ferrite along the octahedral cleavages of austenite.—B. N.

*Drilling oils, etc.* Marcusson. See IIA.

*Tungsten compounds.* Jannasch and Leiste. See VII.

*Molybdenum compounds.* Jannasch and Laubi. See VII.

*Platinum substitutes.* Nicolardot and Boudet. See XXIII.

## PATENTS.

*Manganese steel; Producing wrought shapes of* — W. S. Potter, Pittsburgh, Pa. U.S. Pat. 1,278,207, 10.9.18. Appl., 11.5.14. Renewed 1.6.18.

The steel ingot is heated to a temperature within the range of 200° C. above the critical temperature (about 825° C.), for a sufficiently long time to allow of re-absorption by the mix crystals (solid solution) of the metal of the excess carbides of the eutectic and any carbides which may have separated; the metal is subsequently heated to a temperature suitable for the ductility required, and then subjected to reduction.—T. H. B.

*Ferro-molybdenum; Process of making* — E. H. Westling (Assignor to N. W. Stern) and C. Andersen, San Francisco, Cal. U.S. Pat. 1,278,408, 10.9.18. Appl., 18.11.16.

An acid solution of molybdic acid, containing a ferrie salt, is neutralised with an alkaline basic compound, the precipitated ferrie molybdate separated, and heated in presence of a reducing agent.—T. H. B.

*Iron and steel scrap; Utilisation of* — Preparation of briquettes of iron and steel scrap in admixture with carbon. Deutsch-Luxemburgische Bergwerks- u. Hütten-A.-G., Bochum. Ger. Pats. (a) 304,872, 24.2.16, and (a) 307,834, 2.6.17.

(A) TURNINGS and planings of iron and steel are heated to incandescence and then made into briquettes, which are subsequently smelted. Scrap from special steels can be worked up in this way and then charged direct into an open-hearth furnace. (B) A mixture of scrap iron or steel and carbon is briquetted as described under (A) or is charged into a rotary kiln wherein it is heated by a reducing flame.

*Tin and wolfram ores; Concentration of* — W. W. Webster, E. Edser, and L. A. Wood, London. Eng. Pat. 119,050, 14.6.17. (Appl. 8562/17.)

BEFORE concentration by means of tables, vanners, etc., the ore is subjected to the action of sodium or potassium hydroxide, silicate, carbonate, or phosphate, ammonia, borax, or silicic acid sol. The process is more particularly applicable to the treatment of slimes.—W. E. F. P.

*Smelting and refining metallic swarf and the like; Furnaces for* — J. Tylor and Sons, Ltd., H. T. White, J. Gaunt, and D. Brookfield, London. Eng. Pat. 119,779, 14.1.18. (Appl. 803/18.)

In a furnace of the type having a metal preheater (see Eng. Pat. 118,750; this J., 1918, 659 A), the preheater communicates with an inclined tubular extension, which contains a trough of semi-circular cross-section so arranged that the flue-gases rising from the furnace through the tubular extension have free access to all sides of the trough. Swarf is charged into the trough through suitable openings in the tubular flue and is melted down, and discharged into the crucible from the lower end of the trough.—C. A. K.

*Furnaces for heating metal articles and the like.* W. H. Southorne, North Shields. Eng. Pat. 119,787, 22.1.18. (Appl. 1230/18.)

The furnace, which is specially applicable to the hardening or annealing of steel tools, gauges, and

the like, has a removable, revolving bottom, the upper surface of which is corrugated or roughened to permit free access of air or gases to the articles resting thereon. The furnace is heated by burners so arranged that the flames do not impinge directly upon the articles.

*Furnaces for heating tools and other articles; Gas-fired* — H. J. Yates, Birmingham, and S. N. and E. R. Brayshaw, Manchester. Eng. Pat. 119,817, 19.3.18. (Appl. 4797/18.)

The improvements described in Eng. Pats. 22,522 of 1907 and 23,066 of 1911 in relation to methods of operating the doors of furnaces, and in Eng. Pat. 110,960 (this J., 1917, 1266) in relation to burners, are combined in a single furnace.

*Hot-blast stove; Four-pass* — J. I. Larimer, Joliet, Ill. U.S. Pat. 1,278,173, 10.9.18. Appl., 7.11.16.

The first and fourth passes are symmetrically disposed on opposite sides of the stove; the intervening space is bisected by a wall to form the second and third passes. Metal checker-work is used near the end of the last pass and is composed of a number of units, each consisting of spaced webs providing narrow vertical passages, "vertically adjacent units being relatively angularly disposed." (See also U.S. Pat. 1,295,923; this J., 1918, 473 A.) —C. A. K.

*Aluminium or its alloys; Autogenous welding of* — A. Carpmacel, London. From A.-G. für Autogene Aluminium Schweissung, Zürich. Eng. Pat. 120,005, 29.5.18. (Appl. 8874/18.)

A FLUX for use in the autogenous welding of aluminium or aluminium alloys consists of a mixture of alkali halides, having the required fusibility, and comprising at least three different members of the halogen group, of which fluorine must be one. A suitable mixture contains: KCl 40%; NaCl 30%; LiCl 10%; KBr 10%; NaF 10%. The melting point of this mixture is about 600° C.; for use in the welding of alloys, the melting point of the flux may be reduced by an alteration in the proportions of the mixture, or by the addition of an alkali iodide.—C. A. K.

*Ore concentration.* F. Gooch, Cobalt, Ont., Canada. U.S. Pat. 1,276,753, 27.8.18. Appl., 25.5.16.

In an apparatus for concentrating ores by the agitation-froth process, a centrifugal agitator and distributor, comprising radial impeller blades enclosed by upper and lower plates, is provided in the agitation and froth-decantation chamber. The agitator is mounted upon a hollow shaft through which the apparatus is charged with ore pulp and air.—W. E. F. P.

*Subliming and sintering ores; Method and apparatus for* — W. H. Kilbourn, Pueblo, Colo., Assignor to United States Smelting, Refining, and Mining Co. U.S. Pat. 1,278,166, 10.9.18. Appl., 7.11.17.

The ore or similar material containing volatile metals is mixed with a large proportion of carbon and fed on to a horizontal travelling grate passing under an ignition arch in a combustion chamber. A series of depressions is formed in the upper surface of the bed of ore, and a blast of air is passed through the grate under the ignition arch.

—B. V. S.



*Nickel-matte or nickel-copper matte: Method of treating* —. O. Lellep, New York. U.S. Pat. 1,278,176, 10.9.18. Appl., 11.10.17.

NICKEL-CONTAINING matte is subjected to the action of an oxidising agent at a temperature between the melting points of the matte and of nickel oxide. The furnace conditions should be non-reacting (non-acidic).—C. A. K.

*Electric welding; Process of* —. J. B. Murray, Brooklyn, N.Y. U.S. Pat. 1,278,193, 10.9.18. Appl., 22.3.18.

THE parts to be welded are electrically heated to a welding temperature, and at the same time subjected to pressure. The heating is then stopped, but the pressure is maintained until the critical cooling range has been passed.—C. A. K.

*Alloy.* H. S. Cooper, Assignor to The General Alloys Co., Cleveland, Ohio. U.S. Pat. 1,278,304, 10.9.18. Appl., 23.2.18.

AN alloy containing a preponderating amount of nickel, and 0.5–15 or 0.5–20% of zirconium, with relatively small amounts (1–20% in all) of aluminium and silicon.—T. H. B.

*Alloy, and method of purifying same.* P. Poetschke, Assignor to L. D. Caulk Co., Milford, Del. U.S. Pat. 1,278,744, 10.9.18. Appl., 7.2.18.

COMMUNUTED alloys are placed in trays having permeable bottoms, and are purified by treatment with acidified alcohol, then washed in water, dehydrated with alcohol, and dried in a current of air.—T. H. B.

*Electric [metallurgical] furnace and method of supplying current thereto.* J. H. Gray, New York. U.S. Pat. 1,278,635, 10.9.18. Appl., 20.7.18.

A CURRENT of comparatively high E.M.F. is supplied to the electrodes of an electric furnace during the melting down period, to produce a long arc, and during the refining period a current of lower E.M.F. is substituted to avoid surges on the power line. A reactance coil is fitted in circuit with the primary windings of the transformer to effect a higher E.M.F. on the secondary winding, and after melting the metal down this reactance coil is cut out of circuit so as to reduce the E.M.F. in the secondary coils of the transformer.—C. A. K.

*Reduction of metallic oxides, especially iron oxides; Process for the* —. G. Gröndal, Djursholm, Sweden. Ger. Pat. 306,261, 25.11.15.

A LAYER of the finely divided metallic oxide, covered with a charge of finely divided coal or other fuel, is first heated by combustion of the latter in a current of air drawn or forced downwards through the charge; when the desired temperature has been attained the air current is so diminished as to produce carbon monoxide, by which the metallic oxide layer is reduced. At 700° C. the reduction of iron oxide by carbon monoxide takes place with practically no absorption of heat. If necessary a layer of lime may be interposed between the fuel and the metallic oxide, to desulphurise the gases before they reach the latter. The process may be carried out in a suitably lined cast-iron holder, open at the top, provided with a suction pipe for the gases at the bottom, fitted inside with a grate near the bottom to support the charge, and mounted on trunnions to enable it to be inverted.—J. H. I.

*Zinc; Production of — of high purity from impure metal and alloys in vacuo.* E. Herter, Berlin. Ger. Pat. 306,725, 6.6.15.

IN the vacuum distillation of zinc the condensed liquid metal is continuously drawn off, without impairing the vacuum, by maintaining a column of the liquid metal of sufficient height to balance the atmospheric pressure.—J. H. I.

*Blast-furnace gas; Apparatus for purifying* —. A. M. Fasel, Mülhofen. Ger. Pat. 306,853, 11.3.16.

FOR the removal of dust the gases are conducted, through inclined ducts, across the open tops of vertical chambers or fall-pipes from the lower ends of which the deposited matters can be discharged at intervals. The ducts are connected by inclined curved end-tubes from which fall-pipes extend downwards. At suitable points the passage of the gases may be obstructed by dampers extending across the lower half of the cross-section of the ducts or end-tubes.—J. H. I.

*Lead-tin alloys.* W. Stockmeyer, Minden, and H. Hanemann, Charlottenburg. Ger. Pat. 306,892, 14.9.15.

CLAIM is made to alloys of lead and tin containing sodium or magnesium or both and if desirable some copper. Small quantities of sodium or magnesium appreciably increase the hardness of lead, and the addition of tin to such alloys renders them less brittle and chemically more resistant; small quantities of copper further increase their hardness.—J. H. I.

*Annealing furnaces [; Means for moving articles through—].* Greenwood and Batley, Ltd., and W. Clegg, Leeds. Eng. Pat. 119,841, 24.6.18. (Appl. 10,379/18.)

*Tin-plates or sheets and other like metal coated plates or sheets; Appliances used in the manufacture of* —. H. S. Thomas, Llandaff, and W. R. Davies, Whitechurch, Glamorgan. Eng. Pat. 119,969, 21.1.18. (Appl. 1140/18.)

*Smelting furnace.* E. K. H. Lundberg, Bruzaholm, Sweden. U.S. Pat. 1,278,696, 10.9.18. Appl., 14.7.16.

SEE Eng. Pat. 108,498 of 1916; this J., 1917, 1013.

*Ingot-mould for manufacture of steel.* B. Talbot, Middlesbrough. U.S. Pat. 1,279,037, 17.9.18. Appl., 13.3.18.

SEE Eng. Pat. 118,488 of 1917; this J., 1918, 628 A.

*Drying air blast.* Ger. Pat. 307,221. See I.

*Retorts for roasting.* Eng. Pat. 119,911. See IIa.

## XI.—ELECTRO-CHEMISTRY.

*Electric heater.* Dean. See IIa.

*Hydroxyazo compounds.* Puxeddu. See III.

*Collrell process.* Heimrod and Egbert. See VII.

*Phosphates at anode.* Fichter and Müller. See VII.

*Platinum substitutes.* Nicolardot and Boudet.  
See XXIII.

#### PATENTS.

*Electro-osmosis; Apparatus for removal of water by means of* —. Bergmann Elektrizitäts-Werke A.-G., Berlin. Ger. Pat. 307,242, 16.5.17.

The walls of the cells are constructed of insulating material, e.g., concrete, in which the electrodes can be mounted without special insulation. The cells may be cylindrical holes in a block of concrete.

—J. H. L.

*Diaphragms for electrolytic purposes.* Siemens u. Halske A.-G., Berlin. Ger. Pat. 307,471, 22.8.16.

DIAPHRAGMS particularly suitable for use in chlorine-alkali electrolytic cells with horizontal electrodes, are made from a mixture of powdered cement and cellulose or material containing cellulose, mixed to a pulpy mass with water. The diaphragms so obtained are strong and somewhat pliable and possess excellent porosity. The porosity can be varied by varying the proportion of cellulose employed. They possess the advantage of cheapness over the Billiter asbestos diaphragms, and are fairly stable. They are more simply prepared than asbestos-cement diaphragms or diaphragms containing cow-hair or wool.

—J. S. G. T.

*Diaphragms used in electrolysis; Supports for* —. Farb. vorm. Meister, Lucius, u. Brünig. Ger. Pat. 307,524, 18.12.17.

In electrolytic processes employing pulpy diaphragms, the diaphragms are usually supported on a grid of wire gauze. In the course of the electrolysis, owing to causes such as variations in the lengths of the individual wires of the grid, the grid buckles and the material of the diaphragm accumulates on the lower parts of the grid, with consequent unequal distribution of current in the electrolyte, and unequal destruction of the anode. According to the present patent a diaphragm support formed of rods is used, which, despite alterations of length, remains plane and in its original position; for example the rods may be left free at the ends to allow for expansion.—J. S. G. T.

*Producing gas.* Eng. Pat. 119,885. See IIa.

*Electric furnace.* U.S. Pat. 1,277,899. See VII.

*Electric furnace.* U.S. Pat. 1,278,635. See X.

## XII.—FATS; OILS; WAXES.

"Sélé," "Cocorico," and *Ximania americana*: African oil-yielding plants. J. Pieraerts. Ann. Musée Colon. Marseille, 1916, 4, Pt. 2, 1—21. Bull. Agric. Intell., 1918, 9, 1060.

Sélé is the local name of an oil popular among the natives in certain parts of the Belgian Congo. A specimen of the oil prepared locally at Mombasa (Bengala district) consisted of the glycerides of oleic, "linoleic," stearic, palmitic, and lauric acids, in addition to a small quantity of an unidentified acid of higher molecular weight. Sélé oil may be classed as semi-drying, and is an excellent edible oil. It has good keeping qualities, is suitable for soap and glycerin manufacture, but unsuitable as a source of stearin owing to its low content of solid acids. *Cocorico* is a variety of *Citrullus vulgaris*. A specimen of the oil prepared locally at Yangambi

(Stanleyville district) had all the desirable qualities of sélé oil, but the low unit yield and the slow and difficult decortication of the seed render the latter useless for commercial purposes. *Ximania americana* is a bush of the *Oleaceæ* family, abundant in tropical America and the West Coast of Africa. The *Ximania* is known by many local names peculiar to the different countries in which it is found, e.g. Jamaica, "mountain plum" or "sea plum"; Gabon, "elozy," "zégucé," or "sea lemon"; South Africa, "zuur prulm" (acid plum). The "sea plum" is particularly valuable on account of its high oil content. The particular interest in the oil lies in its high content of linolenic acid and the special properties of its solid acids.—A. de W.

*Elais poissonii*, a new species of oil palm, in the Cameroons. Fauchère. Bull. P'Office Colon., 1918, 11, 80—83. Bull. Agric. Intell., 1918, 9, 1061.

THE fruit of this oil palm is peculiar in that it is enclosed in a sort of fleshy sheath formed by the development of six staminodes contained in the female flower, which in the varieties of *Elais* hitherto described are always atrophied. The fruit weighs from 10 to 20 grms. The following table gives comparisons of the new species (of which two varieties are to be distinguished—var. *tenera* and var. *dura*) with the var. *Lisombe* of *Elais nigrescens* (the best form of oil palm known at present).

	<i>Tenera</i> %	<i>Dura</i> %	<i>Lisombe</i> %
Oily pulp ... ..	76	44	61.5
Nuts ... ..	24	56	38.5
Oil yield of the pulp... ..	70.25	58.6	63.15
Oil yield of the whole fruit	53.50	55.8	38.35

The following figures were obtained from an examination of ten fruits of each of the chief varieties of *Elais* in the Cameroons:—

	Fruit	Weight in grms.:		Oil
		Pulp	Kernel	
<i>Dibope</i> ... ..	123.0	51.5	23.0	27.0
<i>Lisombe</i> ... ..	121.5	76.5	20.5	47.0
<i>Dura</i> ... ..	171.0	74.2	18.1	44.0
<i>Tenera</i> ... ..	168.5	128.3	19.4	90.0

—A. de W.

*Saponifiable fat and total fat.* J. Prescher. Z. Unters. Nahr. Genussm., 1918, 36, 71—73.

THE true fat content of a material (fatty acid plus neutral fat) is best ascertained from determinations of the free fatty acids, the total fatty acids, and the saponif. value.—W. P. S.

[*Lipolytic*] enzyme action; Influence of certain salts on —. I. S. Falk. J. Biol. Chem., 1918, 36, 229—247.

CALCIUM chloride inhibits the lipolysis of olive oil, and in its presence the point of equilibrium of the reaction mixture is reached much earlier than in its absence. The addition of sodium chloride tends to prevent the appearance of the calcium effect, although sodium chloride by itself exerts scarcely any action on the rate or equilibrium point of lipolysis.—H. W. B.

*Drilling oils, etc.* Marcusson. See IIa.

#### PATENTS.

*Oil from vegetable fruits; Extraction of* —. H. R. Greenhalgh, Rock Ferry, Chester, Assignee of N. A. Gavin, Kinshasha, Belgian Congo. Eng. Pat. 111,676, 28.11.17. (Appl. 17,577/17.) Int. Conv., 28.11.16.

IN a process for extracting oil from the pericarp of fruits, such as palm-oil and similar nuts, the fruit



is subjected to a preliminary heat treatment in a jacketed kettle, and then transferred to a centrifugal extractor. If desired, the residual mat of combined fibre and nuts may be screened and the fibre pressed for the extraction of the remaining oil.—W. E. F. P.

*Sulphur soap.* T. Tanaka, Hongo-ku, Japan. U.S. Pat. 1,278,518, 10.9.18. Appl., 16.4.18.

See Eng. Pat. 117,060 of 1918; this J., 1918, 553 A.

### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Pigments for printing inks.* T. M. Tyson. Oil and Colour Chem. Assoc., Nov., 1918, 1, No. 5.

PRINTING INKS may be divided into two classes, viz., "litho" and "letterpress." The pigments used for the former class must be completely free from acidity, quite insoluble in water, possess good "solidity," moderate transparency, and not too high a specific gravity. Those for use in the manufacture of letterpress inks must be brilliant and opaque and when for use in process or three-colour work have low specific gravity. Printing ink pigments are preferably composed of dyes fixed on alumina, either alone or co-precipitated with *blanc fixe*, the lake and mineral pigments being simultaneously precipitated. Barytes or other abrasive material is quite useless as a base for either class of ink. The desiderata of a good printing ink pigment are principally brightness and strength, the latter property ensuring that sufficient printed impression will be obtained by the use of a very attenuated film, thus guarding against liability to "set-off" on the adjacent superposed sheet. Other properties of the pigment are required for special purposes, e.g., transparency and resistance to heat for tin-printing pigments, insolubility in alcohol for labels which are subsequently spirit varnished, resistance to alkali for use on soap wrappers. A classification into degrees of fastness to light and the adaptation to different purposes of pigments of varying stability to light, alkali, etc., is given, e.g., the special suitability of a fugitive, water- and spirit-soluble pigment, non-resistant to acid and alkali, for printing on cheques, etc.

—A. de W.

*Protecting iron with paint.* Friedl. See X.

#### PATENTS.

*Oil pastes from wet precipitated pigments; Manufacture of — and separation of free water therefrom.* C. R. Rogers, Sydney, N.S.W. Eng. Pat. 118,289, 14.8.18. (Appl. 13,236/18.) Int. Conv., 24.7.17.

A SMALL quantity of a soluble crystallisable salt having a slightly alkaline reaction, e.g., borax, is added during the milling of wet precipitated pigments with oil, in order to cause separation of water and facilitate incorporation of the paint stock with the oil.—A. de W.

*White [antimony oxide] pigments; Manufacture of —.* E. F. Morris, Roby, and T. N. C. Nevill, St. Helens, Lancs. Eng. Pats. (a) 119,711, (b) 119,712, and (c) 119,713, 17.10.17. (Appls. 15,022—4/17.)

(a) THE acidity of commercial antimony oxide due to contained sulphur compounds is neutralised by treating a mixture of antimony oxide in water with

acid-binding substances such as sodium carbonate, borax, or precipitated chalk or mixtures of these. *Example.* A thin mixture of antimony oxide and water is treated with about 0.5% (on the dry antimony oxide) of milk of lime, and boric acid, in proportions to leave the resulting solution faintly alkaline to litmus, and then drying the precipitate. (b) The contamination of antimony oxide with acid sulphur compounds, rendering it objectionable for use in paints, enamels, etc., is removed by treating the antimony oxide or oxide-containing pigment in a dry or comparatively dry state with an alkali or acid-binding substance, preferably whilst both are suspended in air. As examples, the oxide as made, before collection, is treated with gaseous ammonia, atomised alkaline substance (calcium borate), or an organic amine. The acid-binding substance may also be added to the oxide in powder form, preferably in the dampest state allowing fine grinding. (c) A pigment cheaper than antimony oxide but possessing some of its properties may be obtained by treating antimony sulphate with barium carbonate, so as to obtain a mixture of antimony oxide and barium sulphate. *Example.* 100 lb. of antimony oxide is treated with 112 lb. of sulphuric acid (in 12% solution) and run into a small excess of barium carbonate (5% paste). The barium carbonate must be free from sulphide. The pigment is dried below the temperature of formation of antimony tetroxide.—A. de W.

*Carbon-black and lampblack; Process for the manufacture of —.* J. M. Gerard, New York. U.S. Pat. 1,278,137, 10.9.18. Appl., 8.10.17.

THE gaseous products resulting from the incomplete combustion of a hydrocarbon, and containing 2 or 3 lb. of suspended unoxidised carbon particles per 1000 cub. ft., are caused to impinge on a cool surface until substantially 1 lb. of the said carbon particles per 1000 cub. ft. of the gaseous products is collected, the remainder of the carbon particles being electrically precipitated at a voltage ensuring collection in a finely-divided form.—A. de W.

*Coating paper.* U.S. Pat. 1,277,904. See V.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### PATENTS.

*Indiarubber, vulcanite, and like substances; Substitutes for —.* J. F. Bennett, Dronfield, and F. W. Mellows, Sheffield. Eng. Pat. 119,878, 10.10.17. (Appl. 4998/17.)

PLASTIC masses are obtained by heating a mixture of fatty oil, sulphur, and stearin or other hard fat. The fatty oil may be replaced by mineral oil or jelly, and other ingredients such as tar, resin or pitch, lime or iron oxide, and various forms of cellulose fibre, may be introduced to modify the consistency or strength of the product. This is stated to dissolve in most of the common rubber solvents.—D. F. T.

*Leather scrap or refuse; Consolidation of — together with crude or waste rubber.* J. L. Watkins, London. Eng. Pat. 119,902, 16.10.17. (Appl. 14,965/17.)

IN rubber substitutes of the type composed of scrap rubber, scrap leather, new rubber, and cotton or other vegetable fibre, kapok fibre is used as the vegetable fibre. The ingredients are mixed together with oil, asbestos dust or fibre, additional fibrous material if required, a metallic oxide, and a

vulcanising agent such as caustic soda or sulphur; the resulting dough is rolled into sheets or bars and vulcanised in any suitable manner. The product has excellent stitching and flexible qualities.

—D. F. T.

## XV.—LEATHER; BONE; HORN; GLUE.

*Glue; Determination of the adhesiveness of* —. M. Rudeloff. Mitt. K. Materialprüf., 1918, 36, 2—49.

The solution of the glue is applied to the planed end surfaces of two pieces of red beech wood 185 mm. long, 125 mm. broad, and 50 mm. thick, and these are placed so that the glued surfaces cross at right angles. The glue film is then allowed to dry under definite pressure, and the force required to tear the pieces of wood apart is measured by means of a suitable machine. In a series of determinations made in this way it was found that for glue solutions up to 200% of water (referred to weight of glue dried at 100° C.) the tenacity of the film decreased in proportion to the extent to which the wood was heated prior to glueing, but that in the case of solutions with 300% of water the greater degree of heating had a favourable influence. The tenacity of the glue film does not decrease in direct proportion to the rise in the amount of water. In the case of solutions with 100 to 150% of water the pressure under which the glue film is dried has no appreciable influence on the tenacity of the film, but with higher amounts of water the drying pressure has considerable influence, especially when the wood has been previously heated. The most suitable conditions for testing samples of glue by this method are the use of solutions containing 150% of water, previous heating of the pieces of wood to 40° C. in dry air, and drying of the film under a pressure of not less than 0.84 kilo. per sq. cm. Solutions containing 100 to 150% of water give concordant results by this method, which may also be used for comparison with the results obtained by determining the viscosity at 35° C. of glue solutions with 556% of water.—C. A. M.

*Drilling oils, etc.* Marcusson. See IIa.

### PATENTS.

*Tannin; Extraction of pine-bark* —. L. Bruml and R. Silberberger, Klattau, Bohemia. Ger. Pat. 306,529, 22.8.16. Int. Conv., 28.6.16.

Prior to the extraction of tannin, resins are removed from the bark by a solvent boiling at about 100° C. An increased yield of tannin results.

—F. C. T.

[*Tanning agents.*] *Water-soluble condensation products of the naphthalene series; Production of* —. O. Schmidt and F. Günther, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,278,229, 10.9.18. Appl., 29.1.17.

A WATER-SOLUBLE product containing fewer sulphonie acid groups than naphthalene nuclei is obtained by condensing formaldehyde with an insoluble naphthalene hydrocarbon and a naphthalenesulphonie acid, e.g., by the interaction of formaldehyde,  $\beta$ -naphthalenesulphonie acid, and naphthalene in presence of sulphuric acid of non-sulphonating strength; the product is capable of precipitating glue from acid solutions.—D. F. T.

*Hides and skins; Bating and deliming of* — [with waste liquors from manufacture of tartaric acid]. E. Luksch, Vienna. Ger. Pat. 305,898, 2.11.16. Int. Conv., 27.5.16.

The waste liquors obtained in the manufacture of tartaric acid after precipitation of calcium tartrate form an excellent bating and deliming liquor.

—F. C. T.

*Hides and skins; Tanning* — by means of hydrolysed [chrome] tanning liquors. E. Kanet, Agram. Ger. Pat. 306,015, 16.10.15.

Strongly basic chrome solutions which deposit chromium hydroxide at ordinary temperatures can be used when cooled. In some cases it is advantageous to warm the hides after they are completely penetrated by the liquor.—F. C. T.

*Leather substitute; Manufacture of a* — suitable for the production of models. F. Krohmer, Pforzheim-Brützingen, and A. Schätzle, Pforzheim. Ger. Pat. 306,104, 19.9.16. Addition to 302,194 (this J., 1918, 344a).

SUITABLE wide-meshed fabric is saturated with an oil-varnish or similar substance, and pressed together with a filling material consisting of leather dust, glue, tale or the like, and colouring matter if desired. Strips so prepared are laid in warm water at 40°–50° C. before use, until completely softened and free from air bubbles.—F. C. T.

*Utilising leather scrap, etc.* Eng. Pat. 119,902. See XIV.

## XVI.—SOILS; FERTILISERS.

*Bacterium lactis viscosum; Occurrence of* — in soil. C. R. Fellers. Soil Sci., 1918, 5, 487–488.

ORGANISMS corresponding to written descriptions and laboratory cultures of *B. lactis viscosum* were isolated from two different soils, neither of which had received applications of cow dung for several years. It is probable that the soil is a natural habitat of this organism.—W. G.

*Nitrogen-assimilating bacteria; Influence of reaction on* —. E. B. Fred and A. Davenport. J. Agric. Res., 1918, 14, 317–336.

THE most noticeable difference in the behaviour of *Rhizobium leguminosarum* derived from different leguminous plants is that certain strains develop much more rapidly than others. To determine the effect of acid and alkali upon these bacteria and upon azotobacter, cultivations were made on mannitol solutions, the reaction of which was changed by the addition of sulphuric acid or sodium hydroxide, and the cultures incubated for four weeks at 28° C. The number of bacteria present was then determined, and the hydrogen-ion content of the cultures was measured colorimetrically. It was found that sulphuric acid in culture media is far more injurious to alfalfa bacteria than to lupin bacteria. The leguminous bacteria may be divided into the following groups as regards their sensitiveness to acid: (1) Critical,  $P_H$  4.9, alfalfa and sweet clover; (2)  $P_H$  4.7, garden pea, field pea, and vetch; (3)  $P_H$  4.2, red clover and common beans; (4)  $P_H$  3.3, soya beans and velvet beans; (5)  $P_H$  3.15, lupins. Leguminous bacteria are more resistant to alkali than to acid, about ten times as much N/1 alkali as acid being required to produce a similar injury. No pronounced difference in sensitiveness to alkali was shown by



different leguminous bacteria. *Azotobacter* is extremely sensitive to slight changes in the reaction of the culture media. The acid limit for the growth of *azotobacter* in mannitol solution is about  $N/1333.3$  and the alkaline limit about  $N/1000$ , or the critical  $P_{\alpha}$  acid value is 6.5 and the alkaline value 8.6. Nodule bacteria from different leguminous plants show pronounced differences as regards the hydrogen-ion concentration of the medium. There appears to be some relationship between the degrees of resistance to acid shown by the bacteria and by the higher plant.—C. A. M.

*Potash: Availability of — in some common soil-forming minerals. Effect of lime upon potash absorption by different crops.* J. K. Plummer. J. Agric. Res., 1918, 14, 297–315.

The common soil-forming minerals, biotite, muscovite, orthoclase, and microcline, show little difference as regards the solubility of their contained potassium in water; but biotite and muscovite yield considerably more potassium to solutions of carbonic acid than do orthoclase and microcline. The solubility of the potassium in any of these minerals is not increased by lime in the form of calcium bicarbonate. Pot experiments have shown that oats, soya bean, rye, and cow-pea take up different amounts of potassium from these minerals. For example, the increase in the yield of the dry matter of oats following the application of biotite is four times as great as with microcline and 66% as much as with potassium sulphate, whilst the increase with muscovite is nearly twice as much as with orthoclase. Analogous results are obtained with rye. The addition of calcium carbonate does not cause any material increase in the yield of dry substance or in the amount of potassium removed from the soil by oats or rye, but when used in conjunction with biotite it causes an increase in the yield of the dry matter of soya bean by about 33%. Calcium carbonate used in association with muscovite causes a pronounced increase in the growth of the plant, and a smaller increase when used in association with orthoclase or microcline. Soya beans and cow-peas removed more potassium from soils treated with calcium carbonate in association with potassium sulphate, biotite, and muscovite, which may be attributed to the development of more favourable conditions in the soil for the growth of the plants, and not necessarily to more potassium having been dissolved. The addition of calcium carbonate does not appear to increase the solubility in  $N/5$  nitric acid of potassium in soils treated with any of these minerals.—C. A. M.

*Ammonium sulphate; Influence of — on the germination and the growth of barley in sand and soil cultures kept at different moisture contents and at various osmotic concentrations of the soil solution.* M. I. Wolkoff. Soil Sci., 1918, 5, 421–479.

The fertilisers applied to the sand or soil were ammonium sulphate, potassium dihydrogen phosphate, calcium carbonate, magnesium sulphate, and ferrous sulphate in different proportions, and the effects of different amounts of potassium chloride, sodium chloride, sodium nitrate, calcium sulphate, aluminium sulphate, and sodium silicate added to the principal mixture were also studied. The moisture content of the sand or light sandy loam in the pots was kept constant at either 20, 40, 60, or 80% of the water-holding capacity of the sand or sandy loam used. In the germination tests two other series having moisture contents of 10 and 15% respectively were used. The moisture content of the soil had a very marked influence on the growth and development of the plants. In the sand cul-

tures the plant yield increased with the moisture content right up to the 80% series, but on the sandy loam the maximum yield of dry matter of barley was obtained in the 60% series. The plant growth either on the soil or sandy loam where the moisture content was kept at 20% of saturation was very small. With a constant moisture content the crop yields increased in the case of the sand cultures with an increase in the application of either ammonium sulphate, calcium carbonate, or potassium phosphate, the effect diminishing in the order given. There was a similar response to applications of nitrogen to the sandy loam, but not to those of lime or phosphate. The difference in plant growth at the various moisture contents is attributed either to the difference in concentration of the soil solution or to the aeration of the soil. The osmotic concentration of the soil solution following the normal application of a fertiliser is not great enough to influence plant growth if the moisture content of the soil is at its optimum (about 60% of saturation). It only becomes an important factor when the moisture content of the soil is considerably reduced. With certain nutrient solutions in the sand cultures with a moisture content of 60%, beneficial results were obtained by the additional application of magnesium sulphate and ferrous sulphate, and also by small applications of potassium chloride, sodium chloride, sodium nitrate, calcium sulphate, and sodium silicate. This beneficial effect is attributed to an improvement in the balance of the ions of the component salts in the soil solution. Aluminium sulphate under similar conditions caused some injury to the plants. A proper balance in the nutrient solution is essential for the rigidity of the straw; the "lodging" effect of applications of large amounts of nitrogenous material may be entirely subdued by modifying the proportions between the component salts in the nutritive solution in sand cultures. The germination of barley seeds is influenced by the same general agencies that affect the growth of plants, though not to the same extent. In sand, germination takes place when the moisture content is as low as 10% or as high as 80% of the water-holding capacity, but in the soils studied it would not take place when the moisture content was below 20%, and was retarded when it reached 80% of the saturation. The treatment of the sand or soil with a nutrient solution at the high moisture contents had very little, if any, effect on the germination of barley seeds. W. G.

*Potassium ammonium nitrate, a new top-dressing for sugar-beet.* M. Hoffmann. Deuts. Zuckerind., 1918, 43, 149–150. Z. angew. Chem., 1918, 31, Ref., 313.

POTASSIUM ammonium nitrate prepared from ammonium nitrate and potassium chloride by the Badische Anilin und Sodafabrik has been used in some places for small experiments. It is easy to distribute over the land, and contains about 13% N, 25%  $K_2O$ , about 3–4%  $H_2O$ , and 27–30% Cl. This material can be used with perfect safety as a top-dressing. It has the advantage over ammonium nitrate, that it can be spread more uniformly and is much less hygroscopic and explosive. According to Schneidewind, potassium ammonium nitrate has exactly the same action as an equivalent amount of sodium nitrate. It is proposed to put a sodium ammonium nitrate on the market. Ammonium chloride, containing 23–25% N, has been put on the market this year, and has the same effect as the sulphate, but care must be taken in using it for plants sensitive to chlorine.—W. G.

*By-product lime [Endlaugenkalk].* P. Kosmann. Tonind.-Zelt., 1918, 42, 579–580.

A VERY brief summary of the various processes patented for preparing a lime fertiliser from the

waste liquors obtained in the extraction of potassium from the Stassfurt salts. Essentially they consist in treating the liquor with an excess of calcium oxide. Magnesium hydroxide and calcium chloride are formed, and these, together with excess of lime, form the principal constituents of the product. An analysis of one sample is given, which contained 9% of magnesium hydroxide, 50% of calcium hydroxide, and 34.26% of calcium chloride ( $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ ).—W. G.

*Plants; Cause of the poisonous action of coal-gas on —.* C. Wehmer. *Z. angew. Chem.*, 1918, 31, 205—209.

EXPERIMENTS carried out by the author prove that the injurious action of coal-gas on the growth of plants and germination of seeds (this J., 1917, 1021, 1106, 1187) is due to hydrocyanic acid which is present in minute quantity in coal-gas.—W. P. S.

*Plants; Behaviour of organic compounds in —.* G. Ciamician and C. Ravenna. *Gaz. Chim. Ital.*, 1918, 48, I., 253—304.

RESULTS are given of experiments on the action of various compounds on the germination and development of plants, on the oxidation of organic compounds by the agency of enzymes contained in spinach leaves and apple pulp, and on the inoculation into living maize plants of pyridine and nicotine, these bases being partly eliminated through the leaves and possibly partly transformed by the plant. (See also *J. Chem. Soc.*, Dec., 1918.)

—T. H. P.

#### PATENT.

*Manure from house, town, and other refuse; Manufacture of —.* A. D. Furse, Beckenham, Kent. Eng. Pat. 119,834, 10.5.18. (Appl., 7843/18.)

THIS invention relates to improvements in the process described in Eng. Pat. 5847 of 1913 (this J., 1913, 953). After the refuse has passed through the first disintegrator, it is spread over a drying floor or passed through an automatic drying machine, in order to reduce the number of disintegrators required for the subsequent fine pulverisation. Fluffy material is removed after the pulverisation is completed by passing the refuse over a shaking sieve.—J. H. J.

### XVII.—SUGARS; STARCHES; GUMS.

*Aldehydes; Determination of — by means of iodine in alkaline solution.* H. Colin and O. Liévin. *Bull. Soc. Chim.*, 1918, 23, 403—405.

THE authors have slightly modified Bougault's method (this J., 1917, 899) and use an alkaline solution containing 35 grms. of sodium phosphate and 50 c.c. of N/1 sodium hydroxide per litre in place of the mixture of sodium carbonate and sodium bicarbonate. An excess of N/10 iodine solution is used, and the volume of alkali added is twice that of the iodine solution. The reaction is complete at the end of one hour, and the excess iodine is titrated back after making the solution just acid with sulphuric acid. Satisfactory results were obtained with the roots and rhizomes of chicory and artichokes, but the results with the leaves were somewhat uncertain.—W. G.

*Potassium ammonium nitrate.* Hoffmann. See XVI.

*Wheat, rye, and potato starches.* Unna. See XIXA.

#### PATENT.

*Beet slices; Process and apparatus for the continuous extraction of —.* H. Eberhardt, Wölbentel. Ger. Pat. 306,517, 5.2.16.

IN a continuous counter-current diffusion process, the suitably pre-heated slices are conveyed downwards at an adjustable rate, through a closed diffuser in which the liquid travels upwards. By heating the juice below, the upper concentrated zones of juice are prevented from sinking. The conveying mechanism for the slices in the diffuser may revolve in opposition to a rotatory movement of the juice. From the bottom of the diffuser the almost exhausted slices are conveyed for a considerable distance against the inflowing water current, to complete the extraction of sugar. In working under pressure, the slices are conveyed into and out of the diffuser through tall pipes to maintain the requisite head of liquid. The conveying mechanism in the diffuser may comprise perforated cylinders through which the slices are transported downwards by spiral conveyors, or it may comprise fixed plates and rotating scrapers or rotating plates and fixed scrapers. Fixed and rotating heating coils may be provided inside the diffuser.—J. H. L.

### XVIII.—FERMENTATION INDUSTRIES.

*Yeast growth.* A. Sclator. *Biochem. J.*, 1918, 12, 248—258.

A METHOD of measuring rates of growth of yeast by direct observation under the microscope at constant temperature is described. The generation times of individual cells of a yeast growing in malt-wort at 30° C. vary within limits which are 10% on each side of the average value of 73 minutes. When old yeast cells are introduced into a fresh medium they remain quiescent for some time (lag-phase). They then start growing at the normal unrestricted rate. Yeast growth proceeds in alternate periods of apparent rest and growth. Little or no lag is observed in yeast cells grown from spores. Air is necessary for yeast growth; the cells can utilise oxygen chemically combined in the wort as well as dissolved oxygen; carbon dioxide inhibits growth. If yeast is seeded into malt-wort, the following stages of growth may be observed: the lag-phase, the logarithmic phase or period of unrestricted growth, retardation in growth due to carbon dioxide, retardation due to failure of oxygen. The latter factor is the main one which finally stops growth. By altering the conditions of seeding, aeration, temperature, etc., one or more of these stages of growth may be caused to disappear. Regarding the influence of temperature, it is found that at a temperature above that at which maximum growth occurs, some cells grow at about the maximum rate, some at lower rates, and some die. By these and similar studies it appears possible to put the problem of yeast crops on a sound mathematical basis.

—H. W. B.

*Wine; New method for determining the wateriness of —.* U. Pratolongo. *Staz. Sper. Agrar. Ital.*, 1918, 51, 56—60. *Bull. Agric. Intell.*, 1918, 9, 1104.

NATURAL wine forms a saturated solution of potassium bitartrate and calcium tartrate. If a wine, when treated with these two salts, dissolves further quantities, the presence of added water is indicated. Portions of the wine under examination are treated separately with excess of potassium bitartrate and calcium tartrate, respectively; the mixtures are kept at 50° to 60° C. for 30 mins., then cooled, the



undissolved salts removed, and determinations made of the two salts in the wine both before and after saturation.—W. P. S.

*Enzyme action.* Falk. See XII.

*Antineuritic substances.* Sugiura. See XX.

#### PATENTS.

*Malt; Process for kilning* — E. Weymar, Mühlhausen, Thuringia. Ger. Pat. 306,751, 5.11.16.

MALT is subjected to the kilning (curing) temperature, not as whole corns, but in the form of grist; or the grist may be separated into husks, grits, and meal, and the process applied to some or all of the fractions. Caramel malt and colour malt may be produced in this way.—J. H. L.

*Brewing [wort boiling]; Process of* — Brauerei zum Wagnerbräu H. Wagner, Munich. Ger. Pat. 306,839, 29.4.17.

THE first wort from the mash is boiled by itself to a gravity of 28–35° Balling, and then diluted with later and weaker wort and boiled again. The process intensifies the malt-like flavour and aroma of the wort and is therefore especially suitable for the preparation of thin beers.—J. H. L.

#### XIXa.—FOODS.

*Milk; Cholesterol in* — W. Denis and A. S. Minot. J. Biol. Chem., 1918, 36, 59–61.

Cow's milk contains from 0.010 to 0.018% of cholesterol; human milk, up to 0.038%. The amount probably varies with the cholesterol content of the food.—H. W. B.

*Whey proteins and curd; Method for distinguishing between* — O. Lüning and W. Tönies. Z. Unters. Nahr. Genussm., 1918, 36, 63–65.

CASEIN, or curd, dissolves in 1% ammonium oxalate solution and is re-precipitated by acetic acid, whilst whey proteins do not yield any substances which are precipitated by acetic acid from the ammonium oxalate solution. This test does not, however, afford a means of directly identifying ordinary whey proteins since casein is rarely removed completely from the whey. The products may be distinguished by determining the total soluble nitrogen: 2.5 grms. of the sample is ground in a mortar with hot 1% sodium oxalate solution, the mixture rinsed into a 250 c.c. flask with about 200 c.c. of the same solution, heated to boiling, allowed to stand for 24 hrs., then diluted to the mark, filtered, and the nitrogen determined in the filtrate. Curd yields 22 to 23% of nitrogenous substances, whilst whey proteins give from 1 to 3%.

—W. P. S.

*Margarine; Determination of water in* — by heating in aluminium beakers. J. Prescher. Z. Unters. Nahr. Genussm., 1918, 36, 70–71.

THE usual method of determining water in margarine by heating the sample in an aluminium beaker until the water has been evaporated presents some difficulty in the case of margarine as now sold in Germany. Excessive spitting takes place probably owing to the presence of traces of soap (from the purification of the crude fats) and of potato starch; the soap appears to coat the starch

granules and the steam escapes violently. Distillation with xylene or drying on sand in the water-oven is recommended in the case of such samples.

—W. P. S.

*Wheat, rye, and potato starches; Micro-colorimetric method for the identification of* — in the presence of each other. E. Unna. Z. Unters. Nahr. Genussm., 1918, 36, 49–53.

TEN grms. of the flour is steeped in 3% phenol solution for 24 hrs., and a small portion is then transferred to a microscope slide and air-dried for 30 mins. It is then treated with a mixture of 1 gm. of Water Blue-Orcein solution (Water-Blue, 1. Orcein, 1, glacial acetic acid, 5, glycerol, 20, alcohol, 50, and water, 100 parts) and 6 drops of 1% eosin solution (in 60% alcohol); after 10 mins., the preparation is washed with water, treated for 20 mins. with 1% Safranine solution, again washed with water, and treated for 30 mins. with 0.5% potassium bichromate solution. The preparation is finally washed with water, alcohol, and xylene, mounted in Canada balsam, and examined under the microscope. Potato starch is coloured bright red, whilst wheat and rye starches are brownish-yellow, the rye being darker than the wheat. Gluten is coloured bright blue.—W. P. S.

*Vegetables; Antiscorbutic property of desiccated and cooked* — M. H. Givens and B. Cohen. J. Biol. Chem., 1918, 36, 127–145.

RAW cabbage added to a scorbutic diet prevents the onset of scurvy in guinea pigs. Cabbage dried at 40°–50° C. retains a small proportion of its antiscorbutic quality, but heating at 75°–80° C. or cooking for 30 minutes and then drying at 65°–70° C. completely destroys the anti-scorbutic vitamin. Potatoes cooked and dried at 65°–70° C. also possess no anti-scorbutic value.—H. W. B.

*Vegetables; Investigation of the methods employed for cooking* — with special reference to the losses incurred. I. Dried legumes. H. Masters. Biochem. J., 1918, 12, 231–247.

BUTTER beans, haricot beans, and dried peas should be soaked overnight in 1% sodium bicarbonate solution, and, after straining, cooked in a 0.25% salt solution either in a steamer for 1½ hours or by boiling for about an hour. This method ensures the least loss of material and the shortest time of boiling.—H. W. B.

*Banana; Nutritive value of the* — K. Sugiura and S. R. Benedict. J. Biol. Chem., 1918, 36, 171–189.

THE banana is unable to supply adequate material for the growth or maintenance of albino rats, being deficient in protein and in the water-soluble accessory substance. The addition of casein and yeast or carrot extract to the bananas renders the diet sufficient for growth and maintenance purposes. The casein in the diet cannot be satisfactorily replaced by beef protein.—H. W. B.

*B. botulinus; Bacterial precipitins and the detection of* — in preserved foods by the thermo-precipitation method. M. Bornand. Trav. chim. aliment. et d'hyg., 1918 9, 87–98. Bull. Agric. Intell., 1918, 9, 1113–1114.

A RABBIT was inoculated repeatedly with extracts of a culture of the *B. botulinus*, and when bled, the serum was found to have precipitating properties specific for the *B. botulinus*. Either the filtered, serum or the serum preserved with toluene could be used, the latter having the stronger action. The

precipitogen in the serum was stable on heating. Boxes of preserved beef, herrings, and peas were inoculated with the *B. botulinus*, and after five months were opened, and cultures made from the contents showed the presence of the *B. botulinus*. 5 grms. of each specimen was added to physiological salt solution and boiled in a water-bath for 5 mins. The solution was filtered and added to the prepared anti-serum, when a precipitate was formed in the case of the beef and the herrings, but not in the case of the peas.—J. H. J.

*Determining aldoses.* Collin and Liévin. See XVII.

*Antineuritic substances.* Sugiura. See XX.

#### PATENTS.

*Cereals, seeds, and flour, especially maize; Process for regenerating damaged* —. L. Callebaut, Sas van Gent, Holland. Eng. Pat. 110,364, 11.10.17. (Appl. 14,752/17.)

The damaged cereals are washed in water, and extracted for 60 hrs. at 50° C. with water containing dilute soda lye and sulphur dioxide, or sulphur dioxide (0.5%) alone, in order to remove deleterious substances and to sterilise the cereals. The extracted cereals are washed and rapidly dried in a vacuum.—J. H. J.

*Flour or meal.* J. Sleeman, Stonehouse, Gloucester. Eng. Pat. 119,923, 26.10.17. (Appl. 15,624/17.)

Fruits or vegetables are cleaned, sliced or pulped, dried to below 12% of moisture, ground to a meal, and passed through a sieve to separate the coarser particles. The product may be mixed with wheat flour or maize malt flour. The coarse particles may be used as a cattle food. Pomace from cider and perry manufacture may be treated by this process. (Reference is directed in pursuance of Sect. 7, Sub-sect. 4, of the Patents and Designs Act, 1907, to Eng. Pats. 21,038 of 1892, 2981 of 1903, 26,794 of 1905, 1074 and 16,651 of 1909, 17,723 of 1913, and 8371 of 1915; this J., 1893, 942; 1909, 903; 1914, 230.)—J. H. J.

*Food preparation.* Mapleton's Nut Food Co., Ltd., and H. Mapleton, Liverpool. Eng. Pat. 120,018, 31.7.18. (Appl. 12,499/18.)

ALMONDS, filberts, and pea-nuts are blanched, dried at 150° F. (65.5° C.), and ground to a paste to which water is added to bring the mixture to the consistency of cream. 10% of milk powder or an equivalent of fresh milk is added, together with a pure culture of lactic acid organisms, and the mass is fermented at 60°–70° F. (15.5°–21° C.) for 20 hrs. The excess of water is then separated, and 2% of salt is kneaded in.—J. H. J.

*Foodstuffs; Extraction of — from bones and other animal substances.* A. Tummer, Berlin-Hohenschönhausen. Ger. Pat. 307,411, 12.10.17.

During the extraction of bones and other animal substances by steam in the usual manner, they are repeatedly treated with the broth, after the latter has been freed from fat, and are also subjected to mechanical pressure.—L. A. C.

#### XIXB.—WATER PURIFICATION; SANITATION.

*Sewage purification; Importance of geological factors in —.* E. A. and A. E. Cooper. Biochem. J., 1918, 12, 275–281.

Is the dissolved oxygen absorption test for the standardisation of a sewage effluent, it is essential that the latter should be diluted with water

collected from the river or stream into which the effluent is discharged instead of with laboratory tap-water. The results also suggest that waters from sedimentary rocks, and those associated with the detritus of eruptive rocks, where the active chemical constituents have been or are released, may be expected to affect profoundly the biological processes of oxidation.—H. W. B.

*Phosphorus; Determination of white — in mouse poisons.* F. Mach and P. Lederle. Chem.-Zeit., 1918, 42, 491.

Ten grms. of the material is mixed with 5 grms. of plaster of Paris, the powdered mixture is transferred to a 200 c.c. cylinder and shaken for 1 hour with 100 c.c. of carbon bisulphide. After settling, 10 c.c. of the clear solution is withdrawn and added to 50 c.c. of saturated bromine solution. The oxidation of the phosphorus by the bromine is complete in 1 hour at the ordinary temperature and the resulting phosphoric acid is precipitated as ammonium magnesium phosphate after the carbon bisulphide and the excess of bromine have been evaporated.—W. P. S.

#### PATENTS.

*Sterilising apparatus.* R. E. Case, South Norwalk, Conn., Assignor to The R.U.V. Co., New York. U.S. Pat. 1,277,378, 3.9.18. Appl., 13.3.16.

In an apparatus for sterilising liquids by means of a "purifying chemical," prepared electrolytically, which is discharged continuously into the liquid as the latter flows through a conduit, the production of the "purifying chemical" is regulated automatically by, and is proportional to, the flow of liquid through the conduit.—W. E. F. P.

*Sterilising liquids; Process of —.* L. M. Wood, Spokane, Assignor to J. R. Gumm, Kelso, Wash. U.S. Pat. 1,278,278, 10.9.18. Appl., 19.12.17.

The liquid in its container is submitted to the action of ultra-violet rays of varying frequency from an electric lamp suspended in, and moved throughout the liquid.—J. H. J.

*Air; Continuous sterilisation and purification of — [by means of ozonised water].* A. Wolff, Berlin. Ger. Pats. (A) 307,433, 15.8.16, and (B) 307,434, 25.1.17.

(A) A QUANTITY of water is circulated continuously through two towers, being pumped from the bottom of one tower to the top of the other. In each tower the water flows downwards in the form of a fine spray, and a separate current of air is pumped into the bottom of each tower, passing out at the top. The air entering one tower, the "saturation tower," has been passed previously through an ozoniser, and ozonises the water with which it comes in contact; in the other tower, the current of air is scrubbed by the spray of ozonised water and is thus sterilised and purified. If desired, the air issuing from the saturation tower, which still contains a certain proportion of ozone, may be mixed with the sterilised air. (B) In an improved form of the above apparatus, the saturation tower is omitted, ozonisation of the water being effected by leading it into one end of a pipe at the top of the sterilising tower and ozonised air into the other end of the pipe. The water enters the tower through a series of nozzles fixed on the pipe, and from the bottom of the tower is pumped back to the top through a system of pipes in which it is utilised to drive a dynamo and an air compressor, and to cool the air under treatment. Air is forced by means of the compressor through the cooler,



which it leaves by a forked pipe, part passing through an ozoniser and then to the top of the tower, where it ozonises the water, and the remainder direct to the bottom of the tower. The sterilised air leaves the tower at the top, and means are provided whereby the humidity and temperature of the air may be altered as desired. Air so treated remains free of germs and is valuable for therapeutic purposes, *e.g.*, for the treatment of open wounds, etc.—L. A. C.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Glucosides; New synthetic* — F. Mauthner. J. prakt. Chem., 1918, 97, 217—224.

TETRA-ACETOGLUCOACETOVANILLONE,  $C_{24}H_{24}O_{12}$ , colourless crystals, m.pt.  $156^{\circ}$ — $157^{\circ}$  C., obtained by the condensation of acetovanillone and acetobromoglucose by sodium hydroxide in aqueous acetone solution below  $18^{\circ}$  C., is converted by 6% barium hydroxide solution into glucoacetovanillone,  $C_{15}H_{20}O_8$ , needles, m.pt.  $223^{\circ}$ — $224^{\circ}$  C. Methyl tetra-acetogluco-salicylate (tetra-acetogaultherin),  $C_{22}H_{26}O_{12}$ , leaflets, m.pt.  $154^{\circ}$ — $155^{\circ}$  C., methyl tetra-acetogluco-*p*-coumarate,  $C_{24}H_{28}O_{12}$ , m.pt.  $160^{\circ}$ — $161^{\circ}$  C., and methyl tetra-acetoglucoferulate,  $C_{25}H_{30}O_{13}$ , needles, m.pt.  $125^{\circ}$ — $126^{\circ}$  C., have been similarly prepared from methyl salicylate, *p*-coumarate, and ferulate respectively. The last two yield by hydrolysis gluco-*p*-coumaric acid,  $C_{15}H_{18}O_8$ , needles, m.pt.  $194^{\circ}$ — $195^{\circ}$  C., and glucoferulic acid,  $C_{16}H_{20}O_9$ , needles, m.pt.  $186^{\circ}$ — $187^{\circ}$  C. (See also J. Chem. Soc., Dec., 1918.)—C. S.

*Santonin; Oxidation of* — by means of organic peracids. G. Cusmano. Gaz. Chim. Ital., 1918, 48, I., 248—253.

OXIDATION of santonin by means of perbenzoic or peracetic acid yields isoartemisin or  $\delta$ -oxysantonin and  $\epsilon$ -oxysantonin. Treatment of the latter with concentrated hydrochloric acid in the cold yields a new chlorosantonin,  $C_{15}H_{17}O_4Cl$ . (See also J. Chem. Soc., 1918, i., 434.)—T. H. P.

*Antitoxic sera; Concentration of* — by the salting-out of the heat-denatured serum proteins with sodium chloride. A. Homer. Biochem. J., 1918, 12, 190—209.

THE concentration of anti-toxic sera can be successfully conducted by regulation of the heat-denaturation of the serum proteins, followed by a direct treatment of the heated sera with brine and salt. The end products thus obtained are clear and easily filterable, but they are more deeply coloured than those furnished by the Banzhaf and Homer methods. Moreover, they contain a higher percentage of serum proteins and have a lower degree of concentration of the antitoxin. (See also this J., 1917, 734; 1918, 107 A.)—H. W. B.

*Antineuritic substances from carrots and yeast; Preparation of* — K. Sugiura. J. Biol. Chem., 1918, 36, 191—196.

ALCOHOLIC and aqueous extracts of yeast and of carrots cure polyneuritis in pigeons. A colourless crystalline substance of considerable curative value can be obtained from dried yeast by dissolving in salt solution and subjecting it to per-crystallisation through a collodion bag by Kober's method (this J., 1917, 1038; see further J. Chem. Soc., 1918, i., 532).—H. W. B.

*Uric acid in blood; New volumetric method for the determination of* — L. J. Curtman and A. Lehrman. J. Biol. Chem., 1918, 36, 157—170.

THE method consists in removing the proteins from the blood by coagulation, precipitating the uric acid in the filtrate by nickel acetate in the presence of sodium carbonate, and estimating the uric acid in the precipitate by means of a standard solution of iodine. (See further J. Chem. Soc., Dec., 1918.)—H. W. B.

*Lactic acid; Thiophen test for* — A colour test for aldehydes. Biochemical colour tests. 1. W. R. Fearon. Biochem. J., 1918, 12, 179—183.

THE thiophen reaction for lactic acid is due to the production of acetaldehyde from the lactic acid, which interacts with the thiophen in the presence of excess of sulphuric acid to give the cherry-red colour. This discovery forms the basis for the following general reaction for aldehydes: A couple of drops of a 0.2% alcoholic solution of thiophen are mixed with 5 c.c. of concentrated sulphuric acid and then a drop of a weak solution of an aldehyde is added. A red colour develops and spreads through the acid. On adding a few drops of water, the colour is discharged, but can be reproduced by adding more sulphuric acid. The test can detect 1 part of formaldehyde in 100,000; substituted aldehydes such as chloral, *p*-hydroxybenzaldehyde, etc. also give a positive reaction. The presence of aldehydes in moist ether can be demonstrated by this test, which is important because ether is frequently used in the extraction of lactic acid, prior to estimation. (See also J. Chem. Soc., Dec., 1918.)—H. W. B.

*Iodoform; Alteration of* — alone or in solution, in direct light. E. Comanducci and G. Meduri. Gaz. Chim. Ital., 1918, 48, I., 238—247.

THE action of direct sunlight on solutions of iodoform in 19 different organic solvents, kept in sealed vessels almost filled with the solutions, has been studied. The solution (1%) in olive oil remains unchanged after a year. In absence of solvent, iodoform undergoes very little decomposition under the above conditions.—T. H. P.

*Nitriles; Direct transformation of acid chlorides into* — by catalysis. A. Mailhe. Bull. Soc. Chim., 1918, 23, 380—381.

ACID chlorides may be converted directly into the corresponding nitriles by passing their vapours mixed with ammonia over alumina at  $490^{\circ}$ — $500^{\circ}$  C. Excellent results were obtained with benzoyl, isovaleryl, isobutyryl, and propionyl chlorides.—W. G.

*Esters; Influence of temperature and constitution on the velocity of hydrolysis of* — by hydrogen ion catalysis. F. Bürki. Helv. Chim. Acta, 1918, 1, 231—250.

THE hydrolysis of several esters has been studied in the presence of hydrochloric acid, nitric acid, and sulphuric acid respectively at  $25^{\circ}$ ,  $30^{\circ}$  and  $40^{\circ}$  C. The catalytic action increases rapidly with rising temperature, most in the case of hydrochloric acid and least with sulphuric acid. The substitution of one chlorine in the acid of the ester reduces the velocity of hydrolysis to  $\frac{1}{2}$  of its initial value, whilst two chlorine atoms reduce it only to  $\frac{1}{3}$  of the original value. An unsaturated group in the alcohol of the ester has no effect on the rate of hydrolysis, whilst an unsaturated group in the acid considerably reduces the velocity. (See also J. Chem. Soc., Dec., 1918.)—J. F. S.

*Esterification in aqueous solution.* A. Purgotti. *Gaz. Chim. Ital.*, 1918, 48, II., 54—62.

ONLY slight esterification takes place between alcohol and acetic acid in aqueous solution, addition of sodium chloride appreciably increasing the proportion of ester formed. Acids catalyse the reaction in proportion to their dissociation, and a similar action is exerted by mono- and di-hydric phenols; gallic and tannic acids and trihydric phenols retard the esterification.—T. H. P.

#### PATENTS.

*Chloroform; Manufacture of* —. S. Utheim, Christiania, Norway. Eng. Pat. 116,094, 13.5.18. (Appl. 8026/18.) Int. Conv., 25.5.17.

A 10% AQUEOUS acetaldehyde solution is added gradually to a mixture of 100 grms. of bleaching powder and 1 litre of water, heated at 60°–80° C., and contained in a vessel provided with a stirrer. The aldehyde is instantaneously and quantitatively converted into chloroform which distils off and is collected. The temperature of the mixture may be maintained at 60°–80° C. by regulating the supply of aldehyde solution. Other hypochlorites may be used in place of bleaching powder and the solution should contain about 10% of active chlorine.—W. P. S.

*Tobacco; Process for treating* —. H. H. Albertsen, Roskilde, Denmark. Eng. Pat. 119,959, 2.1.18. (Appl. 138/18.)

INFERIOR tobaccos are soaked in hot water or in a steam bath and the sap containing the nicotine is removed by pressing or by other means. After subsequent drying the tobacco leaves are immersed in a decoction of stalks or dust derived from tobaccos of greater value; after the required quantity of "nicotine" has been absorbed, the tobacco is again dried. In this way it is possible to convert European tobaccos into the equivalent of the finer over-seas products.—D. F. T.

*p-[Hydr]oxyphenylarsinic acid; Manufacture of* —. W. W. Myddleton, Runcorn. Eng. Pat. 119,934, 10.1.18. (Appl. 573/18.)

INSTEAD of removing the *p*-hydroxyphenylarsinic acid from the crude reaction product obtained in any known manner, by extraction with acetone, the crude acid is treated in warm aqueous solution with a suitable neutral salt, such as sodium acetate, which is capable of being decomposed by the *p*-hydroxyphenylarsinic acid, the resulting sodium *p*-hydroxyphenylarsinate being easily obtained in a pure crystalline condition by cooling the solution after previous filtration and concentration; the salt may be purified further by recrystallisation.—D. F. T.

*Lime-tannin compound; Production of a* — slightly soluble in dilute acids. Knoll und Co., Ludwigshafen. Ger. Pat. 307,857, 14.4.16. Addition to 306,979 (this J., 1918, 672A).

A SOLUTION of tannic acid is heated with calcium hydroxide until a basic calcium tannate of a sufficiently low degree of solubility is produced.

—A. B. S.

## XXI. PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### PATENT.

*Colour photography.* W. V. D. Kelley, Brooklyn, N.Y., Assignor to Prizma, Inc. U.S. Pat. 1,278,461, 10.9.17. Appl., 7.2.16.

A TWO-COLOUR process is employed in which the first colour image is obtained by converting a

metallic, e.g. silver, image into a dye image through the medium of a metal-dye compound, and the second colour image is obtained by re-sensitising the film, e.g. with bichromate, and, after exposure under the second negative, treating with a solution of a dye which is absorbed in a degree varying with the hardness of the film.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Smokeless powder; Quantitative determination of acetone in* —. A. Pieroni. *Atti R. Accad. Lincei*, 1918, 27, II., 52—57.

THE discordant results obtained in the determination of acetone are due to the difficulty of converting acetone quantitatively into iodoform, to the ease with which the latter is attacked by excess of alkali, to the loss of iodine owing to secondary reactions, and to the volatility of iodoform. The following procedure gives satisfactory results with smokeless powders which contain between 3 and 0.25% of acetone. From 35 to 50 grms. of the powder in lumps is weighed to the nearest 0.01 gm. and ground in a special apparatus in which the powder is kept washed by a stream of distilled water so as to prevent evaporation of the solvent. The ground mass, together with the water, is either collected in a large beaker or passed directly through a funnel into a distillation flask of about 2 litres capacity; 100 c.c. of dilute sulphuric acid (1:1) is then added to prevent emulsification of the distillate. Steam is passed into the flask and the latter also heated with a burner. The distillate passes from the condenser into a bulb adapter reaching almost to the bottom of a test-tube which is full of water and is situated in a conical flask. From the latter unabsorbed gases pass through a washer, which retains any traces of acetone still unabsorbed. When 250–300 c.c. of distillate, i.e. rather more than one-half of the volume of liquid originally in the distilling flask, has been collected, the adapter is removed and a little fresh distillate collected in a test-tube and tested for acetone by means of potash and iodine. When acetone ceases to distil over, the whole of the liquid in the collecting flasks, together with the washings of these and of the adapter, is made up to 500 c.c. in a measuring flask. Exactly 100 c.c. of the mixed solution is pipetted into a cylinder of about 300 c.c. capacity with a ground stopper. From two burettes are then run in a solution containing 112 grms. of potassium hydroxide per litre and another containing 257 grms. of iodine and 230 grms. of potassium iodide per litre; the two solutions should drop at the same time and at the rate of about 20 drops per minute, the cylinder being kept shaken meanwhile. From time to time the addition is stopped for a time until the iodoform settles and the supernatant yellow liquid clarifies; if further addition of the potash and iodine solutions produces no turbidity, the transformation of the acetone is complete. After a rest of a few minutes, such excess of sodium chloride is added that 10 grms. or so remains undissolved, 50 c.c. of ether kept over sodium being then added, the cylinder tightly closed and the liquid shaken energetically three or four times. As soon as the two layers have separated completely, 25 c.c. of the ethereal iodoform solution is pipetted into 25 c.c. of saturated alcoholic caustic potash solution: the volume of alcoholic potash to be used varies with the volume of iodine used in the reaction with the acetone, the former being in general double the latter. The liquid is diluted with alcohol until clear, heated in a water-bath to evaporate the ether, and boiled for 15 minutes; the alcohol is then distilled off, the



liquid diluted with water, cooled, and acidified with dilute nitric acid, and the potassium iodide formed determined volumetrically by Volhard's method. If  $a$  is the number of c.c. of  $N/10$  silver nitrate solution used and  $b$  the weight in grams of the powder taken, the percentage of acetone in the latter will be  $2a/b$ .—T. H. P.

*Cap compositions; Analysis of* —. Marqueyrol and P. Lorient. Bull. Soc. Chim., 1918, 23, 401–403.

THE powder is first extracted with ether, to dissolve the organic components (trinitrotoluene, picric acid, etc.). The residue is filtered off, and the filtrate is evaporated at the ordinary temperature, the soluble material being finally dried at  $60^{\circ}\text{C}$ ., weighed, and then characterised. The residue is extracted with a few c.c. of cold water, filtered, and the insoluble matter washed with a very little water. The aqueous extract is evaporated to dryness, the residue weighed, and its composition (chlorates, nitrates, etc.) determined. The residue insoluble in ether and water is extracted with 10 c.c. of a 5% solution of potassium cyanide, being left in contact with the solution for two hours. The solution is filtered, the residue being washed, first with more of the solution and then with water, and the whole of the filtrate and washings is submitted to electrolysis. The mercury of any mercury fulminate in the original powder is deposited, washed, and weighed. The residue from this last extraction is mixed with a little water and 1 c.c. of glacial acetic acid and distilled into a 5% solution of silver nitrate. If the original powder contained any lead azide, a precipitate of silver azide is formed and is filtered off, washed with water, alcohol, and ether, and dried and weighed. By this process the approximate quantitative composition of the powder is obtained.—W. G.

*Trinitrotoluene and tetranitromethane; Fatal cases of poisoning by* —. Fischer. Zentralbl. Gewerbehyg., 1917, 205. Chem.-Zeit., 1918, 42, Rep., 165.

THE history of seven fatal cases of poisoning by trinitrotoluene is described, and the author expresses the opinion that the actual cause of the poisoning was the tetranitromethane present, in greater or smaller quantity, in the trinitrotoluene. Evidence in favour of this view is brought forward.

### XXIII.—ANALYSIS.

*Alkalimetry; Errors in* — due to the carbon dioxide content of distilled water. G. Bruhns. Z. anal. Chem., 1918, 57, 257–277.

DISTILLED water absorbs carbon dioxide from the atmosphere and this dissolved carbon dioxide has a marked influence on the results obtained in alkalimetric titrations if such water is used for the preparation of the standard solutions. The standard acid and alkali solutions themselves absorb carbon dioxide.—W. P. S.

*Iodotannic reagent [for determination of alkalinity]*. D. E. Tsakalotos and D. Dalmas. Bull. Soc. Chim., 1918, 23, 391–400.

THE reagent is a mixture of 1 c.c. of  $N/10$ -iodine solution with 1 c.c. of 1% tannin solution, and by means of it the alkalinity of very dilute alkaline solutions may be determined as follows. The alkaline solution is added from a burette to the 2 c.c. of reagent, with stirring, until a definite red

colour appears. From this point the alkaline solution is run in gradually, and after each addition a drop of the mixture is tested on starch paper. The end-point is reached when a blue colour is no longer formed on the test-paper. Knowing the volume of alkaline solution used, its alkalinity may be calculated by means of data given in a table in the original. By means of this reagent exact results may be obtained for alkaline solutions at a dilution of  $N/10,000$  to  $N/40,000$ .—W. G.

*Platinum; Replacement of* — in apparatus for electrolysis. P. Nicolardot and J. Boudet. Bull. Soc. Chim., 1918, 23, 387–391.

ELECTRODES made of alloys of gold and platinum containing from 12.5–25% Pt were found to vary in weight during electrolysis of certain solutions. The authors recommend the use of an alloy of gold, silver, and copper in the proportions of 920 : 50 : 30, the electrodes being coated with a thin layer of platinum (0.005 grm. per sq. cm.).—W. G.

*Magneto-chemistry; Applications of* — to analysis. II. A. Quartaroli. Gaz. Chim. Ital., 1918, 48, I., 65–78.

THE method previously described (this J., 1916, 278) for the determination of certain magnetic salts in solution may be advantageously replaced by the following procedure, which admits of the use of magnetic fields not of exceptionally great intensity and requires no special apparatus. In a magnetic field obtained by the use of horizontal poles of frusto-conical form with circular ends, is inserted a small tube about 8 mm. in internal diameter. This tube contains a standard dilute ferric chloride solution slightly acidified with hydrochloric acid, the surface of the liquid corresponding with the lower point where the poles begin to diverge. The solution of the ferric salt to be determined, best coloured with an ordinary indicator, is pipetted carefully on to the top of the standard solution so as not to mix with it. When the magnet is excited, the upper solution, if the more concentrated, bulges out into the lower and in some cases a globule becomes detached and remains suspended in the lower layer. The upper solution is then gradually diluted with successive diminishing quantities of water until a just perceptible bulge appears within about a minute, the concentrations of the two solutions then corresponding. Descriptions are given of the modes of applying this method to: the estimation of iron, either alone or in presence of manganese or chromium; the estimation of chromium, manganese, nickel, and cobalt; volumetric analysis with permanganate; volumetric analysis with bichromate and analysis of mixtures of chromic salts and chromates; the analysis of solutions containing ferrocyanides, ferricyanides, thiocyanates, and organic matter. (See also J. Chem. Soc., Dec., 1918.)—T. H. P.

*Potassium; Simplification of the method of estimation of* —. W. Hüttner. Kali, 1918, 12, 178–179. Z. angew. Chem., 1918, 31, Ref., 310.

A WEIGHED quantity of the potassium salt (in the form of sulphate) is dissolved in cold water in a graduated flask and sufficient barium bromide, in concentrated aqueous solution, is added, the whole being well shaken and the flask filled up to the mark. The precipitate is filtered off and 10 c.c. of the filtrate is precipitated with perchloric acid in a glass basin. The results obtained by this method are in agreement with those obtained when barium chloride is used. If it is necessary to use heat for the solution of the potassium salt, the

author recommends the use of hydrobromic rather than hydrochloric acid in order to avoid the formation of barium chloride, which is insoluble in alcohol and might cause an error in the result.

—W. G.

*Magnesium; Gravimetric determination of* —. L. W. Winkler. *Z. angew. Chem.*, 1918, **31**, 211–212.

Precipitation of magnesium as  $MgNH_4PO_4 \cdot 6H_2O$  and weighing it in this form is recommended: 100 c.c. of the solution, containing not more than 0.05 gram. of magnesium, is treated with 3 grams. of ammonium chloride, heated at about 90° C., 10 c.c. of 10% ammonia is added, and then 10 c.c. of 10% sodium phosphate solution is run in while the mixture is stirred. After 24 hrs., the precipitate is collected on a cotton-wool filter, washed with ammonia, then with methyl alcohol, dried for 24 hrs. over calcium chloride, and weighed. The filter is washed previously with methyl alcohol and dried over calcium chloride. Potassium and sodium chlorides if present in moderate quantity (not more than 5% in the solution) do not interfere, but if large quantities are present the magnesium must be determined by the ordinary method as pyrophosphate. The addition of a suitable quantity of ammonium chloride (at least 3 grams.) is necessary to ensure complete precipitation of the magnesium.

—W. P. S.

*Copper; Gravimetric determination of* — by means of sodium nitroprusside, and the separation of copper from mercury. E. Votoček and J. Pazourek. *Chem.-Zeit.*, 1918, **42**, 475–476.

A cupric salt is mixed with an excess of sodium nitroprusside solution acidified with dilute sulphuric acid, the mixture diluted to 170 c.c., shaken for 2 to 4 hours, the precipitated cupric nitroprusside then collected on a tared filter, washed with water, dried at 110° C., and weighed; it contains 22.75% Cu. The yield averages 99.2% of the amount of copper present. To determine copper in the presence of mercury, the copper is precipitated as cupric nitroprusside as described, but in this case a known excess of N/10 sodium chloride solution is also added to prevent precipitation of mercuric nitroprusside. The mercury in the filtrate from the copper precipitate is then precipitated as sulphide or determined by titrating the excess of sodium chloride with standard mercury solution. The presence of lead and bismuth does not interfere with the determination of copper, but tin salts should not be present.

—W. P. S.

*Copper; Determination of* — by means of potassium thiocyanate and potassium iodide. G. Bruhns. *Centralbl. Zuckerind.*, 1918, **26**, 354–356. *Z. angew. Chem.*, 1918, **31**, Ref., 310.

In the estimation of copper in Fehling's solution, by means of potassium thiocyanate and potassium iodide, by the method previously described (see this J., 1918, 445 A), considerable errors may occur if the acidified copper solution is allowed to stand for some time after the addition of potassium thiocyanate before being titrated with thio-sulphate solution. These errors are due to the gradual formation of insoluble cuprous thiocyanate, the copper thus precipitated being removed from the iodometric estimation.—W. G.

*Copper; Iodometric estimation of* —. I. M. Kolt-hoff. *Pharm. Weekblad*, 1918, **55**, 1328–1346.

A SUMMARY of the literature of the iodine method of estimating copper, and an account of the results obtained by varying the experimental conditions.

—A. J. W.

*Cadmium and nickel; Alkali iodides as reagents for* —. A. Agresti. *Gaz. Chim. Ital.*, 1918, **48**, 11., 30–34.

ADDITION of potassium iodide solution of 20–30% concentration to a distinctly ammoniacal solution of a cadmium salt produces a heavy white precipitate composed of microscopic regular octahedra having the formula,  $Cd(NH_4)_2I_2$ ; under similar conditions, no precipitate is obtained with ammoniacal copper solutions. Strongly ammoniacal solutions of nickel salts, however, yield a precipitate of  $Ni(NH_4)_2I_2$ , which consists of bluish-violet, microscopic octahedra; if the concentration of the nickel is low, the precipitate is formed only after shaking the liquid and scratching the side of the vessel with a glass rod. Both these precipitates undergo hydrolysis. Since cobalt also gives a precipitate with this reagent, it should be removed by means of Vogel's reaction before the solution is tested for nickel. To this end the solution is treated with concentrated ammonium thiocyanate solution and the whole shaken with a mixture of 1 vol. of amyl alcohol and 10 vols. of ether. By means of a separating funnel the aqueous liquid is separated from the supernatant blue alcohol-ether solution of ammonium cobalto-thiocyanate and further extracted with two fresh portions of the alcohol-ether mixture. The residual greenish aqueous liquid is treated with ammonia and potassium iodide solution as before. Potassium iodide is not so sensitive a reagent for nickel as dimethylglyoxime, but the latter is costly and at present not easily procurable.—T. II. P.

*Electric heater.* Dean. See IIA.

*Drilling oils, etc.* Marcusson. See IIA.

*Tars and pitches.* Weiss. See III.

*Oxygen-yielding bleaching materials.* Grün and Jungmann. See VI.

*Hydrochloric, bromic, and iodic acids.* Purgotti. See VII.

*Sulphur in pyrites.* Binder. See VII.

*Selenious and selenic acids.* Moser and Prinz. See VII.

*Sulphites, thiosulphates, etc.* Sander. See VII.

*Nitrites and hydrogen peroxide.* Quartaroli. See VII.

*Tungsten compounds.* Jannasch and Leiste. See VII.

*Molybdenum compounds.* Jannasch and Laubl. See VII.

*Detecting strain in iron.* Whiteley and Hallimond. See X.

*Determining lead in tin-plate.* Deininger. See X.

*Composition of alloys.* Von Schwarz. See X.

*Gallium.* Dennis and Bridgman. See X.

*Saponifiable and total fat.* Prescher. See XII.

*Adhesiveness of glue.* Rudeloff. See XV.

*Determining aldoses.* Colin and Liévin. See XVII.

*Watering of wine.* Pratolongo. See XVIII.

*Distinguishing whey proteins and curd.* Lüning and Tönies. See XIX.



*Determining water in margarine.* Prescher.  
See XIXA.

*Wheat, rye, and potato starches.* Unna. See XIXA.

*B. botulinus in foods.* Bornand. See XIXA.

*Phosphorus in mouse poisons.* Mach and Lederle.  
See XIXE.

*Uric acid in blood.* Curtman and Lehrman.  
See XX.

*Thiophen test for lactic acid.* Fearon. See XX.

*Colour test for aldehydes.* Fearon. See XX.

*Acetone in smokeless powder.* Pieroni. See XXII.

*Cap compositions.* Marquayrol and Lorette.  
See XXII.

#### PATENT.

*Burettes and the like.* J. W. Towers, Widnes,  
and J. Harger, Gateacre, Lanes. Eng. Pat.  
119,580, 11.12.17. (Appl. 18,331/17.)

A DEVICE for use in place of taps on burettes, etc., is described. The bore of the exit tube of the burette is blocked and above and below the block the bore is extended laterally through the wall of the tube. A rubber tube is slipped over the tube to close the two openings thus formed. By pinching the rubber tube the openings may be uncovered and a channel made for the liquid in the burette to flow from the upper part of the bore to the lower and so be discharged. If desired, a piece of glass rod may be placed in the rubber tube; if this rod is moved over the two holes by pressing it with the finger, a channel is formed for the liquid. When the rod is moved off the holes, the latter are closed by the rubber tube pressing on them.

—W. P. S.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately, and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Apeldoornsche Machinefabriek. Refrigerating, and refrigerating apparatus. 19,084 and 19,106. Nov. 20 and 21. (Holland, 20.11.17 and 8.4.18.)

Archer. Kilns. 18,610. Nov. 13.

Brettell. Heat-insulating coverings. 19,204. Nov. 22.

Calvert. Apparatus for catalytic reactions. 18,632. Nov. 13.

Candy. 19,096. See XIX.

Chandler, and South Metropolitan Gas Co. Gas-heated furnaces. 18,983. Nov. 19.

Commin. Mechanical agitators. 18,402. Nov. 11.

Cuming. Utilising vacuum principle for insulation of high and low temperatures. 18,948. Nov. 19.

Cuming. Conversion of wood waste, vegetable and like substances and their utilisation as insulating material. 18,949. Nov. 19.

Drake, and Drakes, Ltd. Regenerative furnaces. 18,405. Nov. 11.

Dufton. Still heads. 18,415. Nov. 11.

Flechtner. Filtering apparatus for gases etc. 18,637. Nov. 14.

Fox and Martin. Furnace. 18,717. Nov. 14.

Govers. Apparatus and method for heat treatment of chemical compounds. 19,271. Nov. 22. (U.S., July 18.)

Hopper. Crushing and drying machine. 19,253. Nov. 22. (Fr., 25.7.17.)

Instone. Ice-making machinery. 18,887. Nov. 13.

Instone. Freezing media. 19,244. Nov. 22.

Kirke. Gas-fired furnaces. 18,718. Nov. 14.

McCaskell. Continuous pressure filtering process and apparatus. 19,133. Nov. 21.

Poore. Scrubbers, saturators, etc. 19,060. Nov. 20.

Renshaw. 18,654. See II.

Sturgeon. Centrifugal separators. 19,233. Nov. 22.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,383 (1917). Hadfield and Bawtree. See VIII.

14,691 (1917). Candy. See XIX.

16,444 (1917). Morison. Apparatus for filtering liquids. (120,442.) Nov. 20.

16,587 (1917). Thorncroft. Extraction of juice from vegetable substances. (120,448.) Nov. 20.

16,741 (1917). Henshaw. Leaching flue-dust. (111,845.) Nov. 20.

16,965 (1917). Hutchins. Muffle furnaces. (120,633.) Nov. 27.

17,190 (1917). Abbott. Drying-chambers. (120,635.) Nov. 27.

1646 (1918). Lund. Rod-mills. (113,097.) Nov. 20.

3144 (1918). Mangelsdorff. Apparatus for dehydrating. (120,501.) Nov. 20.

7473 (1918). Aubert. Furnaces. (115,840.) Nov. 27.

8285 (1918). Batchelor. Furnaces. (120,522.) Nov. 20.

9534 (1918). Trefileries et Laminaires du Havre. Furnaces. (120,031.) Nov. 20.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Bateman. Deodorising and purifying oils, liquids, etc. 18,691. Nov. 14.

Bowen. Apparatus for producing artificial fuel. 18,432. Nov. 11.

Burney. Apparatus for distilling solid carbonaceous material. 19,177. Nov. 21.

Chandler, and South Metropolitan Gas Co. 18,983. See I.

Cutler. Dust interceptors for water-gas plants. 18,662. Nov. 14.

Dobson and Pickard. Gas producers. 18,430. Nov. 11.

Duchemin. 18,452. See VII.

Harger, and Lever Bros. 18,767. See VII.

Joseph (Rae and Symores). Rendering alcohol unfit for human consumption. 19,095. Nov. 21.

Masters. Manufacture of blue and carburetted water gas. 18,593. Nov. 13.

Merz and McLellan, Michie, and Weeks. Low-temperature distillation of fuel. 19,083. Nov. 20.

Mogford. Distillation of solid fuel. 19,199. Nov. 22.

Poore. 19,060. See I.

Rappaport. Manufacture of acetone by dry distillation of pyrolusite of lime, etc. 18,984. Nov. 19.

Renshaw. Utilising waste gases of furnaces, kilns, and ovens. 18,654. Nov. 14.

Soc. le Coke Industriel. Apparatus for separating coke or carbon from slag etc. 19,322. Nov. 23. (Fr., 23.11.17.)

Wade (Le Petit). Production of fuels. 19,134. Nov. 21.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,089 (1917). Smith. Gas-producers. (120,599.) Nov. 27.

17,103 (1917) and 7586 (1918). Glover, West, and Wild. Retorts for the continuous carbonisation of coal and the like. (120,458.) Nov. 20.

19,307 (1917). Marks (General Engineering Co.). Initiating the combustion of hydrocarbon fuels. (120,663.) Nov. 27.

731 (1918). Sivyer. Apparatus for the manufacture of coke. (113,091.) Nov. 27.

2832 (1918). Smith. Scrubbers, cleansers, and coolers for gas. (120,681.) Nov. 27.

### III.—TAR AND TAR PRODUCTS.

#### APPLICATIONS.

Barnett Co. Catalytic oxidation of benzene. 18,550. Nov. 12. (U.S., Jan. 5.)

#### COMPLETE SPECIFICATION ACCEPTED.

12,839 (1917). Southcombe and Downie. *See* XX.

### IV.—COLOURING MATTERS AND DYES.

#### COMPLETE SPECIFICATION ACCEPTED.

11,625 (1917). Hart. Production of dyes. (120,588.) Nov. 27.

### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### APPLICATIONS.

Aitken. Paper-making machines etc. 18,728. Nov. 15.

Bleachers' Assoc., Ltd., and Higgins. Scouring cotton yarns and fabrics etc. 19,220. Nov. 20.

Coleman and Jones. Production of pulp for manufacture of paper etc. 18,785. Nov. 15.

Coleman and Jones. Manufacture of paper etc. 18,786. Nov. 15.

Cross, Cunningham, and Fine Cotton Spinners' and Doublers' Assoc. Treatment of cellulose textiles. 19,141. Nov. 21.

Cuning. 18,949. *See* I.

Fenton. Treatment of rags of woven, knitted, etc. fabrics. 18,551. Nov. 13.

Konagai. Waterproof paper. 19,203. Nov. 22.

Paramor, and Watford Engineering Works. Machines for straining paper pulp. 18,587. Nov. 13.

#### COMPLETE SPECIFICATIONS ACCEPTED.

16,190 (1917). Walton. Fireproofing processes and compositions. (120,421.) Nov. 20.

8158 (1918). McKee. *See* XVIII.

### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

#### APPLICATIONS.

Calico Printers' Assoc., and Roberts. Machines for washing, bleaching, dyeing, etc. textile fabrics and yarns in rope form. 19,298 and 19,299. Nov. 22.

Drey. Producing discharge and resist effects on pile fabrics. 18,480. Nov. 12.

Farrell. Dye jigs. 19,192 and 19,280. Nov. 22 and 23.

### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

#### APPLICATIONS.

Calvert. Production of cyanide. 19,135. Nov. 21.

Duchemin. Treatment of pyroligneous acid. 18,452. Nov. 11.

Espenhahn. Treatment of gases containing sulphur dioxide. 18,772. Nov. 15. (Australia, 15,11,17.)

Field, and Metals Extraction Corporation. Purification of zinc solutions. 18,832. Nov. 16.

Griffiths. Production of metallic sulphides. 18,399. Nov. 11.

Harger, and Lever Bros. Manufacture of hydrogen, nitrogen, and carbon dioxide. 18,767. Nov. 15.

Jørgensen. Treatment of pyrolusite. 19,236. Nov. 22.

Mond (Internat. Precipitation Co.). 18,554. *See* IX.

Norsk Alkali Aktieselskabet. Apparatus with horizontal diaphragms for electrolysis of alkaline chlorides. 18,702. Nov. 14. (Norway, Apr. 4.)

Pierce. Production of barium oxide. 18,993. Nov. 19.

Wilson. Electro-thermal arsenic-refining and manufacturing furnace. 18,908. Nov. 18.

Wilson. Rotary regenerative gas arsenic roasting furnace. 18,909. Nov. 18.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,745 (1917). Wilton. Distillation and recovery of ammonia from ammoniacal liquor. (120,596.) Nov. 27.

16,741 (1917). Henshaw. *See* I.

### VIII.—GLASS; CERAMICS.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,383 (1917). Hadfield and Bawtree. Means for rendering porous vessels impervious to fluid. (120,410.) Nov. 20.

17,454 (1917). Kennedy. Refractory material. (111,853.) Nov. 27.

17,893 (1917). Marsh. Manufacture of refractory substances, artificial slate, etc. (120,471.) Nov. 20.

1284 (1918). Miller. Controlling the outflow of glass from melting-tanks. (112,947.) Nov. 20.

### IX.—BUILDING MATERIALS.

#### APPLICATIONS.

Atkins and Colquhoun. Manufacture of bricks, tiles, clinkers, road ballast or metal, etc. 18,843 and 18,844. Nov. 16.

Atkins and Colquhoun. Crude-oil burners for firing brick kilns etc. 18,845. Nov. 16.

Davidson. Seasoning sawn timber. 19,091. Nov. 21.

Horn. Making concrete resistant to moisture. 18,960. Nov. 19. (U.S., 20,10,17.)

Mond (International Precipitation Co.). Recovery of potassium compound in connection with cement manufacture. 18,554. Nov. 13.

### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

#### APPLICATIONS.

Allen and Ryding. Manufacture of open-hearth steel. 19,168. Nov. 21.

Angel. Treatment and reduction of sulphides, refractory etc. ores. 19,230. Nov. 22.

Ballantine and Sulman. Manufacture of alloys. 19,318. Nov. 23.

Coles. Manufacture of finely-divided metals. 18,486. Nov. 12.

Davis. Crucible furnaces. 18,621. Nov. 13.

Falvet. Manufacture of steel. 18,518. Nov. 12.

Falvet. Manufacture of iron or steel or their alloys. 18,519. Nov. 12.

Gregory. Recovery of tin from tinned metals. 18,818. Nov. 16.

Hadfield. Manufacture of alloy steel. 18,684. Nov. 14.

Hadfield. Refining steel. 18,685. Nov. 14.

Haglund. Separating and refining metals. 18,495. Nov. 12. (Norway, 19,12,17.)

Harvey. Metal-melting furnace. 18,529. Nov. 12.



Kuehrich. High-speed tool steel. 18,413. Nov. 11.  
 McGahan. Alloying furnaces. 18,616. Nov. 13.  
 Marks (American Manganese Steel Co.). Recovering and making manganese steel. 18,525 and 18,526. Nov. 12.  
 Mate and Rourke. Furnaces for rendering metal articles non-oxidisable. 18,810. Nov. 16.  
 Paton. Crucibles for molten metals. 18,606. Nov. 13.  
 Riley. Coating steel with white metal. 18,982. Nov. 19.  
 Seward. Electro-depositing magnesium. 18,467. Nov. 11. (U.S., 19,11,17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,632 (1916). Withers (Charles, Broderick, and Kirby). Puddling-furnaces and processes for making wrought iron. (120,400.) Nov. 20.  
 16,687 (1917). Mate. Anti-rust treatment of metals. (120,619.) Nov. 27.  
 17,892 (1917). Cortese. Furnaces for roasting ores and the like. (120,654.) Nov. 27.  
 10,957 (1918). Carlisle and Harbord. Tempering ovens or furnaces. (120,530.) Nov. 20.

#### XI.—ELECTRO-CHEMISTRY.

##### APPLICATIONS.

Norsk Alkali Aktieselskabet. 18,702. *See* VII.  
 Perry, and Siemens Bros. and Co. Galvanic cells. 18,507. Nov. 12.  
 Seward. 18,467. *See* X.

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,076 (1917). Ablett, Quincey, and Adams. Electrodes for secondary batteries. (120,598.) Nov. 27.  
 16,394 (1917). Oldham and Oldham. Galvanic batteries. (120,439.) Nov. 20.  
 16,507 (1917). Baur. Electric furnaces. (120,444.) Nov. 20.  
 16,645 (1917). Levin. Electrolytic gas-generators. (111,483.) Nov. 27.  
 2384 (1918). Fery. Electric cells. (120,681.) Nov. 27.  
 3840 (1918). Baxter. Electric cells and batteries. (120,689.) Nov. 27.  
 3901 (1918). Illemaun and Montgoimerie. Non-conducting material. (120,691.) Nov. 27.

#### XII.—FATS; OILS; WAXES.

##### APPLICATIONS.

Bateman. 18,691. *See* II.  
 Calvert. Treating oils and fats. 19,126. Nov. 21.  
 Sheppard. 18,977. *See* XIII.  
 Townsend. Detergent. 19,038. Nov. 20.

#### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

##### APPLICATIONS.

Howse. Synthetic production of resin and varnish. 19,023. Nov. 20.  
 Ivanson and Roberts. Composition impervious to oils, spirits, etc. 18,959. Nov. 19.  
 Sheppard. Softening agent for oxidised or polymerised oils and fats, gums, and resins. 18,977. Nov. 19.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

##### APPLICATIONS.

Levinson. Rubber compound. 18,671. Nov. 14.  
 Macdonald. Rubber substitute. 19,128. Nov. 21.

#### XV.—LEATHER; BONE; HORN; GLUE.

##### APPLICATIONS.

Bailly and Peyrache. Method of preparing bates for hides. 19,236. Nov. 22. (Fr., 22,11,17.)  
 Randall. Liming and tanning hides. 19,120 and 19,121. Nov. 21.

#### XVII.—SUGARS; STARCHES; GUMS.

##### APPLICATIONS.

Higgins. Production of sugar and saccharin from seaweed. 18,497. Nov. 12.  
 Sheppard. 18,977. *See* XIII.

#### XVIII.—FERMENTATION INDUSTRIES.

##### APPLICATION.

Joseph (Rae and Symores). *See* II.

#### COMPLETE SPECIFICATION ACCEPTED.

8158 (1918). McKee. Process of obtaining alcohol from sulphite liquors. (120,520.) Nov. 20.

#### XIX.—FOODS; WATER PURIFICATION; SANITATION.

##### APPLICATIONS.

Barber. Production of pectous substances. 18,668. Nov. 14.  
 Bateman. 18,691. *See* II.  
 Candy. Filtration of water. 19,096. Nov. 21.  
 Coleman and Jones. Treatment of fodder or forage. 18,787. Nov. 15.  
 Newbould. Butter substitutes. 19,209. Nov. 22.  
 Oxley. Alimentary product. 18,924. Nov. 19.  
 Turner. Heating, drying, and cooling grain and hydrolysing wheat etc. 18,721. Nov. 15.

#### COMPLETE SPECIFICATIONS ACCEPTED.

14,691 (1917). Candy. Filtration of water. (120,595.) Nov. 27.  
 16,587 (1917). Thorneycroft. Extraction of juice from vegetable substances. (120,448.) Nov. 20.  
 9207 (1918). Abrahamsen. Antiseptics, disinfectants, and the like. (117,072.) Nov. 20.

#### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

##### APPLICATIONS.

Fryer and Fryer. Method of obtaining nicotine. 18,748. Nov. 15.  
 Pope and Turner. Production of organic compounds. 18,899. Nov. 18.

#### COMPLETE SPECIFICATION ACCEPTED.

12,829 (1917). Southcombe and Downie. Separation and purification of sulphonic acids. (120,405.) Nov. 20.

#### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### COMPLETE SPECIFICATION ACCEPTED.

16,572 (1917). Hess-Ives Corporation. Colour photography. (112,769.) Nov. 27.

#### XXIII.—ANALYSIS.

##### APPLICATION.

Aktiebolaget Ingeniörsfirma F. Egnell. Gas-analysing apparatus. 19,066. Nov. 20. (Ger., 20,11,17.)

**I.—GENERAL ; PLANT ; MACHINERY.**

*Boiler settings; Measuring temperature of gases in —.* H. Krelsinger and J. F. Barkley. U.S. Bureau of Mines, Bull. 145, 1918. 72 pages.

THE errors in measuring the temperature of hot gases are chiefly due to radiation from the instrument, variation of the temperature at different points in a cross-section of the path of the gases, and fluctuation of the temperature at the point of measurement. The radiation error is the most significant and in some cases may amount to several hundred degrees Fahrenheit. A thermocouple having the hot junction inclosed in a protecting tube reads lower than an unprotected couple, but of course the latter can only be used where it will not be subject to contamination. The smaller the wires at the hot junction of the couple the more accurate is the determined temperature. The true temperature can be obtained approximately by using three or more couples of various sizes and determining from these readings, by extrapolation, the theoretical indication of a couple of zero diameter. The nature and extent of the radiation error was determined in this manner for the burning gases in settings of Babcock and Wilcox, Helme, and Stirling water-tube boilers. By screening the thermo-couple so that hot gases pass on both sides of the screen, the radiation error can be considerably reduced and results can be obtained which are only about 20° F. (11° C.) too low. For temperatures of flue gases the copper-constantan couple can be more advantageously used than the platinum-platinum rhodium couple because its E.M.F. is about four times that of the latter. Although easily contaminated, the relative cheapness of copper-constantan couples enables them to be readily replaced. Full instructions are given in the report for making, standardising, and using this couple in conjunction with a portable potentiometer. The theoretical aspect of the radiation error is treated in much detail, due attention being paid to emissivity, size of couple, and the effect of the temperature and velocity of the gas stream.—T. F. E. R.

**PATENTS.**

*Drying liquids; Machines for —.* T. Blom and A. Gledle, Wärmland, Sweden. Eng. Pat. 110,753, 22.10.17. (Appl. 15,347/17.) Int. Conv., 23.10.16.

THE liquid to be dried is fed from a hopper, which may be in the form of a closed chamber subjected to a vacuum and serving for the preliminary concentration of the liquid, on to the surface of an annular horizontal plate which forms the upper side of an annular chamber rotating within a casing about a hollow vertical axis through which a suitable heating medium passes into and out of the chamber. Heated air from the lower part of the casing is caused to circulate over the surface of the plate in a direction opposite to that in which it is rotated. A scraper removes the dried material from the plate at a point just before it passes beneath the feed hopper.—W. H. C.

*Drying fruit, vegetables and other materials; Apparatus for use in —.* H. A. Price and J. Sugg, London. Eng. Pat. 120,283, 20.11.17. (Appl. 17,118/17.)

THE fruit or other material is supported on perforated trays spaced apart one above the other in a drying chamber. Air is heated by a burner mounted on a swing door in a chamber placed below the drying chamber, and separated from it by an inclined baffle-plate. Inclined adjustable

baffles are arranged between the trays so that the heated air passes under, over, and through the material on the trays.—W. H. C.

*Dryer.* F. G. Sargent, Westford, Mass., Assignor to C. G. Sargent's Sons Corporation, Graniteville, Mass. U.S. Pat. 1,279,165, 17.9.18. Appl. 20.12.17.

SUPERPOSED reciprocating conveyors mounted in a frame are arranged in a casing so as to leave a considerable space between the walls of the casing and the top, bottom, and one side of the series. The opposite edges of the conveyors are slightly spaced from the adjacent side wall of the casing. At each side of each conveyor a vertical partition extends upwards, a space being left between it and the bottom of the next partition for the circulation of air. A fan is arranged in the bottom of the casing to circulate air upwards through the conveyors and downwards through the wider side space back to the fan. If a conveyor is clogged, the air circulates round it through the spaces at the sides.—W. F. F.

*Gas dryer.* J. Kennedy, Pittsburg, Pa. U.S. Pat. 1,280,043, 24.9.18. Appl., 16.4.18.

THE apparatus comprises a vertical casing with a gas inlet at the top and a hopper-shaped lower portion provided with an outlet for dirt and water. A helical baffle extends through the casing and at its lower end terminates at a point above the hopper. A gas outlet pipe has its open end just above the hopper, and extends obliquely upwards through one side of the casing at a point between the lower end of the baffle and the hopper.—J. H. P.

*Evaporator; Multiple effect —.* W. S. Herriot, Glasgow. From N. Deerr, Brooklyn, N.Y., U.S.A. Eng. Pat. 120,276, 12.11.17. (Appl. 16,544/17.)

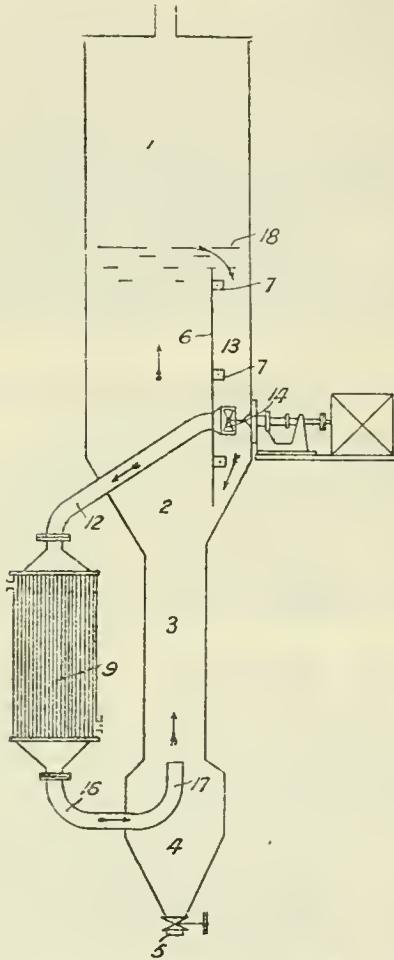
IN a multiple-effect evaporator with vertical tubes and rectangular tube-plates, the calandria is designed so that the outlets for the uncondensable gases and condensed water respectively are at the side of the vapour chamber opposite to that at which the vapour enters. The chamber is divided into two parts by a downtake of rectangular cross-section in the middle, extending almost the whole length of the chamber. Uncondensed gases escape through vertical perforated tubes connected to a manifold, and condensed water flows through other vertical tubes connected to another manifold. The liquid to be evaporated is introduced and withdrawn through perforated horizontal tubes disposed, one on each side of the downtake, beneath the lower tube-plate. Circulation of the liquid up through the tubes and down through the downtake is promoted by curved deflecting plates beneath the lower tube-plate.—J. H. P.

*Evaporating and crystallising apparatus especially adapted for obtaining large crystals.* Soc. d'Exploit. de Proc. Evaporatoires, Système Prache et Bouillon, Paris. Eng. Pat. 112,943, 21.1.18. (Appl. 1171/18.) Int. Conv., 23.1.17.

THE solution to be evaporated is charged into the vessel, 1, 2, 3, 4, up to the level, 18, and is maintained at this level by the addition of more solution as the solvent evaporates. The propelling screw, 14, situated in the end of the pipe, 12, forces the liquid from the space, 13, between the wall of the upper part of the vessel and the partition, 6, downwards through the tubes of the heater, 9. The liquid is here heated to the temperature at which it would boil under atmospheric pressure, but



ebullition is prevented by the pressure due to the propelling screw and the head of liquid. The heated liquid passes back by the pipe, 16, 17, into the narrow cylindrical portion, 3, of the vessel, and passes upwards, circulating in the direction shown by the arrows. When the rising liquid reaches the level of the conical portion, 2, it commences to boil owing to the diminution of pressure

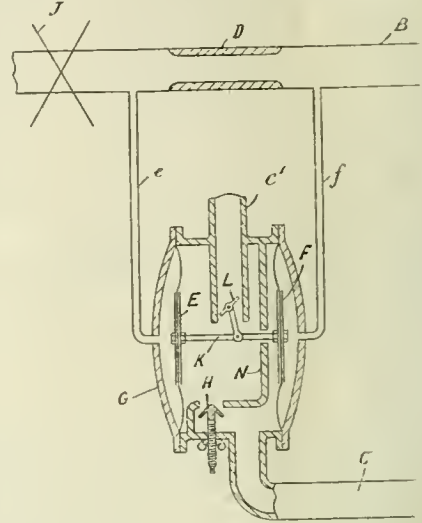


and deposits fine crystals. The vapour escapes from the top of the chamber, 1, and the fine crystals are maintained in suspension by the upward current of liquid until they have grown sufficiently large for their weight to overcome the upward thrust. They then fall through the liquid into the collecting chamber, 4, and are removed from time to time through the valve, 5.—W. H. C.

*Furnaces; Apparatus for maintaining proportional delivery of gas and air to gas-fired* — J. and G. Keith, London. Eng. Pat. 120,076, 20.10.17. (Appl. 15,253/17.)

The air supply to a gas-fired furnace passes through the control valve, J, and constricted member, D, to the pipe, B. The diaphragms, E, F, are connected by a rod, K, and operate a butterfly-valve, L, in the gas inlet, C'. A partition, N, provided with a manually operated valve, H, divides the chamber, G, into two compartments each containing a diaphragm. Tubes, e, f, connect the spaces at the back of the diaphragms, E, F, to the high and low pressure sides of the constriction, D. Any modification in the rate of flow of the air in the

pipe, B, varies the relative pressures on the backs of the diaphragms and moves the rod, K, to operate the valve, L, so that the proportion of gas passing through to the pipe, C, and air passing through the pipe, B, remains constant. To avoid any excessive pressure difference on the two sides of either diaphragm, a modification is described in which the two sides of one diaphragm are exposed to the two air pressures only, and the two sides of the



other diaphragm to gas pressures only. Improved sensitiveness may be obtained by keeping the stroke of the diaphragms as short as possible by the provision of a relay valve for the gas supply, operated by the movement of the diaphragms.

—W. F. F.

*Recuperator furnace.* W. H. Stubblebine, Allentown, Pa. U.S. Pat. 1,279,932, 24.9.18. Appl. 19.10.17.

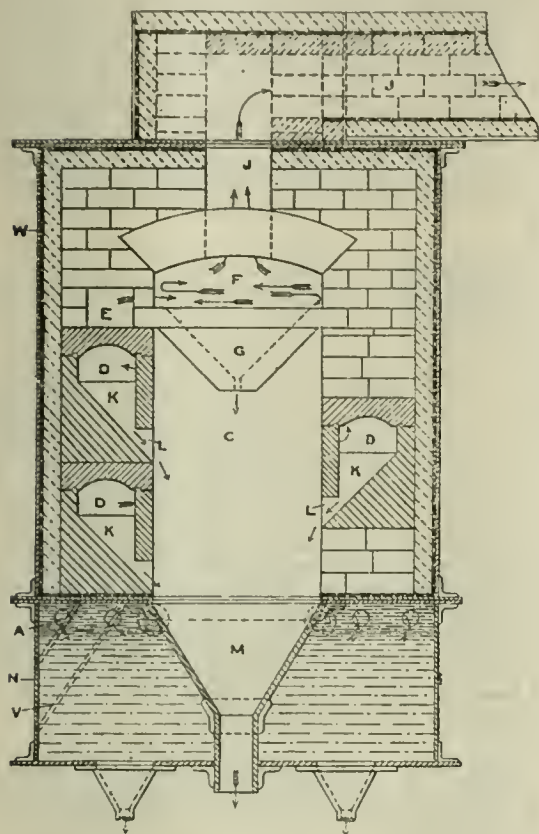
THE furnace comprises a fire chamber, a working chamber, a preheating chamber, and an auxiliary recuperator chamber. Flues connect the preheating chamber with the fire chamber, and a passageway leads from the preheating chamber and communicates with flues in the roof of the fire chamber. Coils connected with an air supply pipe are mounted in the preheating chamber and have nozzles opening into the latter. A blast pipe communicates with the fire chamber. The auxiliary recuperating chamber is provided with ports communicating with the working chamber. Air-heating coils within the auxiliary recuperating chamber have nozzles leading into passageways in the walls of the working chamber, which are provided with ports opening into the working chamber.

—J. H. P.

*Furnaces for generating heat for steam raising or other suitable purposes.* F. E. Whitham, Sowerby Bridge, Yorks. Eng. Pat. 120,274, 9.11.17. (Appl. 16,409/17.)

A MIXTURE of powdered fuel and air is burnt in a combustion chamber, not shown, provided with grate-bars leading to a shoot, V, to receive any solid residue. The combustion chamber discharges into the bottom of a helical flue, D, surrounding a central chamber, C, and leading through a tangential passage, E, to a circular chamber, F, and thence by the passage, J, to a boiler or the like. The flue, D, is constructed of blocks having a number of inverted V-shaped bridges, K, forming

pockets between them, which are provided with outlets, L, into the chamber, C. Incombustible material in the gases passing through the flue, D, falls into the pockets and is discharged into the chamber, C. The chamber, F, is provided with



peripheral pockets which receive solid material from the rotating gases and discharge it into the hopper, G, and thence into the chamber, C. The furnace is mounted on a feed-water tank, N, which is divided into compartments through which the water is circulated. The water is heated by the hot incombustible material passing through the hopper, M, and shoot, V.—W. F. F.

*Gases and liquids; Apparatus for bringing — into intimate contact.* R. S. Hilton and J. H. Willis, Birmingham. Eng. Pat. 120,304, 4.1.18. (Appl. 218/18.)

SEVERAL superposed chambers, separated by conical partitions, each have a central gas passage covered by a hood provided with louvres through which the gas passes upwards to the next chamber. The liquid which collects in the annular space between each cone and the side wall of the chamber is circulated by a pump or injector to a sprayer in the gas passage of the partition next above, and just below the hood, so that it returns to the same chamber and is thus concentrated by continuous absorption of gas. All the circulating pumps may be mounted on the same vertical driving shaft, and the sprayers may be mounted on horizontal guide rods and connected to valve-boxes, so that any one may be withdrawn without affecting the working of the others. A perforated grid carrying scrubbing material may be arranged just over the gas passage in each partition.—W. F. F.

*Gases and vapours; Purification of —.* II. Zschocke, Kaiserslautern. Ger. Pat. 307,890, 22.2.16.

THE invention comprises a series of centrifugal machines with gas passages from one to the other. Each centrifugal machine contains two sets of rotating vanes and one set of stationary vanes. Prior to entering the first centrifugal, the gas is subjected to a fine spray of water, whereby the heavier impurities are removed. The gas is likewise subjected to a water spray in the centrifugal. After passage through a centrifugal, the gas expands into a free space prior to passage into the next centrifugal, which is of larger diameter and in which the vanes rotate at a higher speed. In a modification in which the centrifugal machines are all of the same diameter, successive passage-ways for the gas from one centrifugal to the next are of smaller cross-section than the preceding passage-way, so that the velocity of the gas is augmented as the gas passes through the apparatus.

—J. S. G. T.

*Filter.* J. M. Witmer, Lancaster, Pa. U.S. Pat. 1,279,063, 17.9.18. Appl., 5.5.16.

AN endless conveyor belt of filtering material passes over a series of supporting rollers having a larger diameter at the middle, so that the belt is arched in passing over the rollers and liquid is drained towards the edges.—W. F. F.

*Filter cakes; Method of washing —.* A. E. Genter, Assignor to United Filters Corporation, Salt Lake City, Utah. U.S. Pat. 1,280,439, 1.10.18. Appl., 15.9.17.

IN a method of washing a filter-cake *in situ* by means of a current of atomised fluid, there is interposed between the source of fluid supply and the filter-cake an agent to retard the impinging velocity of the current against the cake.—J. H. P.

*Replacing a liquid containing suspended solids by another liquid; Process of —.* G. Moore, Joplin, Mo. U.S. Pat. 1,279,573, 24.9.18. Appl., 12.6.13.

SUSPENDED solids present in a strong solution are allowed to settle on to the surface of a travelling band or the like, which is then removed to a weaker solution. The settled solids are stirred up and again allowed to settle on to the supporting surface, which is then removed to a still weaker solution or to water and the process repeated.—W. H. C.

*Refrigerating-machine.* D. I. Davis, Chicago, Ill. U.S. Pat. 1,280,101, 24.9.18. Appl., 15.6.16. Renewed 8.4.18.

THE gaseous refrigerant after receiving an initial compression in a low-stage compressor is mixed in the connecting main with some of the condensed liquid from the condenser and then passes to a high-stage compressor where the final compression is effected.—W. H. C.

*Centrifugal process.* T. A. Bryson, Troy, N.J., Assignor to Tolhurst Machine Works. U.S. Pat. 1,280,153, 1.10.18. Appl., 21.3.18.

FINELY divided impervious material in suspension in a liquid is separated by centrifugal action so as to form a gradually thickening outer layer of the material and simultaneously the separated liquid is drained off from just within the impervious layer, *e.g.*, in the apparatus described in U.S. Pat. 1,273,817 (this J., 1918, 614A).—J. H. P.



*Catalyst and method of making same.* A. Schwarmann, Assignor to Kellogg Products, Inc., Buffalo, N.Y. U.S. Pat. 1,280,314, 1.10.18. Appl., 13.8.17.

A NON-SLIMING decolorising catalyst is obtained by impregnating a porous powder having high adsorbing power for colouring matters, e.g. fullers' earth, with a nickel salt, precipitating the nickel in the pores of the material with a solution containing a dissolved nitrogenous organic colloid, e.g. dissolved wool, drying, and reducing the nickel to the colloidal metallic state. The material should be free from alkali-soluble substances.—A. de W.

*Gases and vapours: Method of and apparatus for treating — electrically.* W. T. Hoofnagle, Glen Ridge, N.J., Assignor to Electro-Chemical Products Co., New York. U.S. Pat. 1,280,471, 1.10.18. Appl., 23.11.17.

THE reaction chamber is divided by a horizontal diaphragm provided with openings, over which tubular electrodes with internal heat-radiating ribs are mounted. The opposing electrodes project upwards into the tubes and are also provided with heat-radiating ribs. An electrical condenser is connected across the terminals of the source of current, and the electrodes are all connected in series, with a spark-gap between one condenser terminal and one of the electrodes. The gas to be treated passes at a pressure substantially below atmospheric from one compartment to the other through the tubular electrodes, thus passing in parallel between the electrodes. The discharges between the electrodes are of short duration and high current density, and the gas is passed through at a rate proportional to the frequency of the discharge.—W. F. F.

*Steam; Generation of — and heating of liquids.* P. St. G. Kirke, London. U.S. Pat. 1,279,710, 24.9.18. Appl., 20.11.16.

SEE Eng. Pat. 17,842 of 1915; this J., 1916, 1252.

*Drying wet or watery materials; Apparatus for —.* J. R. Van Musschenbroek, The Hague, Netherlands. U.S. Pat. 1,281,234, 8.10.18. Appl., 4.2.18.

SEE Eng. Pat. 114,442 of 1917; this J., 1918, 320 A.

*Pulverising machine.* Eng. Pat. 120,309. See VII.

*Evaporating furnaces.* Ger. Pat. 307,564. See VII.

*Centrifugal separators.* Eng. Pat. 120,319. See VIII.

## IIa.—FUEL; GAS; MINERAL OILS AND WAXES.

*Combustion in fuel beds of hand-fired furnaces; Low-rate —.* H. Kreisinger, C. E. Augustine, and S. H. Katz. U.S. Bureau of Mines. Tech. Paper 139, 1918. 52 pages.

TESTS have been made with the object of studying the processes of combustion in the fuel bed of hand-fired furnaces such as the American stove. These stoves burn coal at rates below 20 lb. per sq. ft. per hour. Furnaces burning coal at much higher rates, such as those of locomotive boilers, have been reported upon in Tech. Paper 137 (see this J., 1917, 919). The experimental furnace consisted of a steel cylinder, 30 in. high, lined with  $4\frac{1}{2}$ " fire-bricks, and having a grate area of 1 sq. ft. The

air spaces in the grate were  $\frac{1}{4}$ " across and constituted about 60% of the total grate area. Arrangements were made for sampling the gases at different heights in the fuel bed, the latter being either 6 or 12 in. deep. The fuel was charged in at the top and the products escaped by the same opening. The fuels tested were Pittsburgh coal (bituminous), anthracite, metallurgical coke, and semi-coke, broken into pieces which would pass through a screen with  $1\frac{1}{2}$ " holes and remain on one with 1" holes. The gases arising from the fuel bed contained 16 to 30% of combustible gases. The following gas analyses and temperatures are typical of those tests in which bituminous coal was used. The fuel bed was 12" deep and was replenished with small quantities at short intervals.

Height above grate at which sample was taken	CO <sub>2</sub>	Unsat. hydrocarbons	O <sub>2</sub>	CO	CH <sub>4</sub>	H <sub>2</sub>	N <sub>2</sub>	Temp °F
Inches	%		%	%	%	%	%	
1-5	15.2	—	4.2	1.5	0.0	0.1	79.0	1237
3-0	13.7	—	5.1	3.0	0.0	0.1	78.1	1302
4-5	17.8	0.0	0.8	2.8	0.0	0.2	78.4	1296
6-0	14.2	0.0	0.7	9.4	0.0	0.5	75.2	1348
7-5	13.8	0.0	0.8	10.1	0.0	0.5	74.8	1068
9-0	12.4	0.1	0.0	13.4	0.4	0.0	72.8	999
10-5	7.7	0.4	0.6	18.6	1.6	2.0	69.1	—
12-0	6.8	0.1	11.3	2.3	0.3	0.5	78.4	—
13-5	7.0	0.0	11.6	1.2	0.1	0.2	79.9	—

7 lb. of air was necessary to gasify 1 lb. of coal, this ratio remaining constant over a wide range. An additional 7 lb. of air was required to burn the combustible products and was supplied above the fuel bed; the quantity required temporarily increased during the evolution of coal distillation products. It was found practically impossible to keep the fuel bed burning when the air supply to the grate was cut off.—T. F. E. R

*Calorimeter installation; A convenient multiple-unit —.* J. D. Davis and E. L. Wallace. U.S. Bureau of Mines, Tech. Paper 91, 1918. 48 pages.

AN installation of bomb calorimeters intended primarily for the determination of the calorific values of samples of coal is described. The objects aimed at in the installation are facility of operation, compactness, durability, and higher accuracy than that of the usual commercial calorimeter. The bomb consists of a gold-plated cylindrical Monel-metal shell of about 600 c.c. capacity, and is sealed to its base by a one-eighth turn with a wrench. The sealing device comprises a steel receiving nut and lock and a gasket of electricians' solder, and is an adaptation of the principle used in breech locks of cannon. Six calorimeter units are mounted in a bath, the temperature of which is maintained constant to  $\pm 0.02^\circ\text{C}$ . by means of a regulator operating one or other of two relays. The water supplied to the calorimeter is stored in a large tank and is kept constant to  $0.1^\circ\text{C}$ . by means of ice water, the flow of which to the tank is controlled by a thermo-regulator. A measured volume of this water is supplied to the calorimeter by means of a pipette immersed in the tank at constant temperature. Platinum thermometers of fundamental interval about 10 ohms are employed in the calorimeter and by means of a special switch, one bridge and galvanometer can be used with three calorimeter outfits. The resistance bridges are of special construction and read to 0.00005 ohm. The thermometers are calibrated at the ice and steam points, and at the transition point of Na<sub>2</sub>SO<sub>4</sub> (32.384°C.). It is recommended that the water equivalent of the calorimeter be determined by standardisation with pure substances (such as

sucrose) supplied by the Bureau of Standards. A comparison of results obtained by this method with those obtained electrically showed agreement to 1 grm. in the value of the water equivalent. The radiation correction is obtained from the results of combustion of different weights of standard substance, and a radiation correction curve is plotted from such results. The time necessary for an observation is about 8 minutes, and 35 determinations of calorific value per day, with a possible error of about 3 B.Th.U., can be made.—J. S. G. T.

*Coal gas; Liquid purification of, and simultaneous recovery of sulphur and ammonia from —.* P. Parrish. Southern District Assoc. Gas Eng. and Managers, Nov. 14, 1918. Gas J., 1918, **144**, 413—420.

Liquid purification is a continuous process which aims chiefly at the removal of carbon dioxide and hydrogen sulphide from crude coal gas (freed from tar) by the introduction of sufficient ammonia to combine with them, and the subsequent removal by washing with water of the ammonium salts and excess ammonia. The cycle of operations involves purification of the crude gas, dissociation of the crude liquor containing the ammonium salts with removal of the waste gases ( $H_2S$ ,  $CO_2$ , etc.), recovery of the ammonia from the purified liquor for use over again, and use of the hydrogen sulphide in the waste gases for sulphuric acid manufacture or the production of sulphur in a Claus kln. After leaving the tar scrubbers a typical coal gas contains (per ton of coal carbonised) 10.21 to 13 lb.  $H_2S$ ; 0.56 to 0.74 lb.  $CS_2$ ; approx. 1.67 lb. HCN, and approx. 30% lb.  $CO_2$ . The ammonia necessary to combine with these impurities is theoretically 38.08 lb., i.e. about 5 times the quantity normally present in the gas. The author advises the use of seven times that normally present. Every means must be taken to cut down ammonia loss, which is the *bête noire* of the process. This can be done by avoiding excessive pumping and fixing ammonia traps for the displaced air in storage tanks, also by covering stored liquor with oil. The gas washing is preferably effected in washers in which splashes are fixed, arranged on the cascade principle; pumping operations are thereby reduced. The crude liquor from the washers is decomposed with steam in a dissociator still and the hydrogen sulphide, carbon dioxide, etc., with about one-fifth of the ammonia, are driven off. About 65% of the ammonia in these gases is recovered for sale and the remainder removed and added to the crude liquor. The ammonia for sale represents that which would be normally extracted from the gas. The ammonia-free mixture of hydrogen sulphide, carbon dioxide, etc. contains not more than 20%  $H_2S$  and is burnt in a special Claus kln, sulphur being recovered. The purified ammonia liquor leaving the dissociator still passes into an ordinary ammonia-recovery still. From data it is deduced that the equivalent of 80,417 gallons of 1.39% ammonia (8 oz. liquor) would have to be distilled daily for a works carbonising 250 tons of coal per day and using the liquid purification process. By working the dissociator under a pressure up to 3 or more atm., greater concentrations of ammonia can be used (up to 6%  $NH_3$ ) and consequently the liquor to be distilled could be reduced to something like 19,000 gallons in the above case. Under correct conditions the process can be conducted with an overall efficiency of the complete purification plant of 93% of the theoretical. Many diagrams and recommendations concerning size and design of suitable stills, etc. are given, and the paper concludes with a brief criticism of the Burkheiser process, which aims at the production of ammonium sulphate from coal gas without using sulphuric acid.

—T. F. E. R.

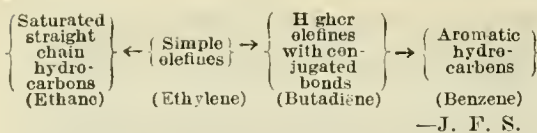
*Gas-meters; Report on the corrosion of dry —.* J. G. Tappay. Life of Gas-Meters Research Committee of Inst. of Gas Eng., 1918. [Advance proof.]

Liquor removed from a dry gas meter was examined and found to consist of three fractions: (1) An oily fraction composed of 94.5% of unsaturated hydrocarbons with resins and naphthalene, 5% of pyridine bases, and 0.5% of paraffins. (2) An aqueous part containing pyridine bases, ferrous and ferric sulphate, ferric thiocyanate, ammonium thiocyanate, and leather substance in a dissolved state (compare Lessing, this J., 1916, 824; Lamb, this J., 1916, 989). (3) A solid portion consisting principally of leather substance (91%) together with smaller amounts of iron oxide, sulphates of iron, tin, and zinc, and some sulphur. The effect of each of these substances and of various mixtures of them upon tinned sheet iron, and upon dressed and undressed leather was ascertained, the materials being subjected to the influence of solutions of the various substances for a period of 4 months. In like manner the effects of sulphur dioxide, carbon bisulphide, ammonia, and a mixture of carbon bisulphide and ammonia were ascertained. It was found that practically all the substances exercised a deleterious effect upon some or all of the materials employed. The action of the various salts is given in detail. Benzol was found to retard the action of a moist mixture of carbon bisulphide and ammonia upon meter materials. In many meters which failed to register correctly the fault was attributed to the presence of a resinous material on the valve. The error of registration of the meters was considerably reduced by spraying the insides of the meters with "solar oil" distillate. A further advantage is achieved by such spraying in that corrosion of the metal parts of the meter is prevented. Suggestions are made as to the possible origin of some of the substances found in the liquor abstracted from the meters.—J. S. G. T.

*Aromatic hydrocarbons; Formation of — from natural gas condensate.* J. G. Davidson. J. Ind. Eng. Chem., 1918, **10**, 901—910.

NATURAL gas condensate containing chiefly ethane and propane with small quantities of butane and pentane has been subjected to "cracking" at various temperatures and pressures in the presence of metals. The products of the reaction are both gaseous and liquid, the latter being of a tarry nature and containing aromatic hydrocarbons. The experiments show that most metals are without effect on the conversion of paraffin hydrocarbons into aromatic hydrocarbons. Nickel, iron, and cobalt are negative catalysts for this reaction, but they accelerate the decomposition of paraffin hydrocarbons into carbon and hydrogen. A temperature of 550° C. is the most favourable for the production of liquid tar, and the formation of complex aromatic substances increases with the temperature. Increase of pressure inhibits the formation of tar, whilst diminished pressure increases the yield of unsaturated substances but also decreases the actual yield of tar. Butadiene has been isolated in fairly large amounts from the unsaturated compounds produced in the thermal decomposition of the natural gas condensate. Acetylene added to the natural gas condensate is without action in the formation of aromatic hydrocarbons. Tar containing aromatic substances has been produced from the "cracking" of a mixture of butadiene and ethylene. The most probable reaction for the formation of aromatic substances from natural gas condensates is:





from bituminous coal. Katz. See III.

Oxidation of carbon monoxide. Hofmann. See VII.

Hydrogen phosphide in hydrogen. Soyer. See VII.

#### PATENTS.

Coke ovens or analogous devices; Multiple regenerators for —. D. Bagley, London. Eng. Pat. 120,079, 22.10.17. (Appl. 15,346/17.)

To prevent short-circuiting of the gases in coke-oven regenerators, the latter are divided into several vertical compartments. The waste gases travel downwards through these compartments into a collecting flue connected at one end with the main waste gas flue. In order that the chimney shall exert an equal pull on each compartment, the opening into the collecting flue is larger the further the compartment is from the waste gas flue. On reversing the gas streams the secondary air is admitted through an intake flue at the end of the regenerator—this flue thus acting as a heat insulator—and enters a horizontal chamber which feeds the regenerator compartments. This chamber gradually tapers towards the other end to ensure uniform distribution of the air.—T. F. E. R.

Coking apparatus. F. Pribyl, Wilmette, Ill. U.S. Pat. 1,279,757, 24.9.18. Appl., 4.11.13. Renewed 17.12.17.

A CLOSED chamber is provided with two parallel vertical partitions extending upwards from the floor about two-thirds of the height of the chamber, and dividing the lower part into three similar compartments. The partitions are provided with openings near their upper edges. Fuel is delivered into the upper portion of the chamber so as to fall into all the compartments, and the central compartment is provided with a grate just below the openings in the partitions, to which air is admitted to burn the fuel and generate producer gas. The hot gas passes through the openings in the partitions, and then through the fuel in the side compartments to distil this fuel. The gas and distillate pass out through openings in the walls below the level of the partition openings, and the coked material is continuously removed at any desired rate from the bottom of these compartments without admission of air or escape of gas.—W. F. F.

Chamber ovens; Continuously operated — for the manufacture of coke and gas. J. Lütz, Essen-Bredene. Ger. Pat. 307,987, 22.6.16.

The invention comprises a coking oven of annular cross-section, in the interior and around the circumference of which chambers are arranged for the collection and passage of gas. These gas chambers are kept cool by means of water tubes. The gas-collecting chambers are provided with outlets for high-grade gas at their upper part and for low-grade gas at their lower part.—J. S. G. T.

Combustion; Process of —. B. Valjean, Santa Ana, Cal. U.S. Pat. 1,280,596, 1.10.18. Appl. 12.7.16.

A NON-EXPLOSIVE poor mixture of gas and air is mixed with a non-explosive rich mixture of gas and air in such regulated proportions that the resulting combustible mixture burns with any desired type of flame from luminous to non-luminous.—W. F. F.

Power gas; Manufacture of — and the recovery of hydrocarbon oils. F. W. Chamier, H. J. Craig, and J. F. L. Moeller, London. Eng. Pat. 120,051, 21.12.16. (Appl. 18,335/16.)

A GENERATOR for the production of power gas is divided by a horizontal partition into two chambers. Coal is fed through a hopper into the upper chamber and is heated to 450°–650° C. by means of a mixture of steam and a portion of the gas generated in the lower chamber, introduced either through a perforated grate or through pipes. The coke formed is dropped intermittently into the lower chamber, into the bottom of which steam is admitted. The gas generated in each chamber is drawn off at the top, that from the upper chamber being treated for the removal of hydrocarbons, tar, etc., and that from the lower chamber, after a portion has been drawn off for injection into the upper chamber, passing through an apparatus in which it is utilised to produce the necessary steam and then into the final scrubbing tower together with the first portion of gas. The horizontal partition may be dispensed with, a portion of the gas from the lower chamber being allowed to rise directly into the upper cooler chamber, into which steam is injected. —L. A. C.

Hydrocarbons; Formation of — having low boiling points from hydrocarbons having high boiling points. D. T. Day, Washington, D.C., F. MacDonald, Casper, Wyo., and R. B. Day, Swarthmore, Pa., U.S.A. Eng. Pat. 113,264, 16.11.17. (Appl. 16,898/17.) Int. Conv., 2.2.17.

A HORIZONTAL combustion chamber of metal lined with firebrick is connected with a vertical cracking chamber similar in construction to the combustion chamber. The oil under treatment is injected, together with steam, into the lower end of the cracking chamber, in which it comes in contact with the products of combustion generated in the first chamber, and the vapours are led off at the top to a condenser. Carbon deposited in the cracking chamber may be removed without interruption of the process by admitting an excess of air into the combustion chamber. The temperature employed in the chamber varies from 700° F. (about 370° C.) to 3000° F. (about 1650° C.), and pressures up to 600 lb. per sq. in. may be used.—L. A. C.

Hydrocarbon materials; Treatment of —. R. B. Day, Swarthmore, Pa., and D. T. Day, Washington, D.C. U.S. Pat. 1,280,179, 1.10.18. Appl. 28.9.17.

THE vapours generated in the cracking apparatus described in Eng. Pat. 113,264 of 1917 (preceding) are bubbled through a body of oil in a suitable container, thus preheating the oil and vaporising the lighter portions. The combined vapours are washed in a scrubbing tower with oil, which is afterwards passed to the preheater and cracking chamber. —L. A. C.

Hydrocarbon materials [gasoline]; Treatment and refining of —. D. T. Day, Washington, D.C., and R. B. Day, Swarthmore, Pa., U.S.A. Eng. Pat. 119,441, 16.11.17. (Appl. 16,900/17.) Int. Conv., 28.9.17.

IN order to purify gasoline prepared as described in Eng. Pat. 113,264 and U.S. Pat. 1,280,179 (preceding), the temperature at the outlet end of the scrubbing tower is maintained at 190° C., all material condensing above that temperature being returned for further treatment. The vapour leaving the top of the tower passes through a condenser and the condensed gasoline is washed successively with sulphuric acid, water, and caustic soda solution by passing it upwards through tanks containing these reagents.—L. A. C.

*Hydrocarbons [of low boiling point]: Apparatus for producing — [from hydrocarbons of high boiling point].* R. B. Day, Swarthmore, Pa., U.S.A. Eng. Pat. 120,230, 27.9.17. (Appl. 12,954/17.)

PREHEATED oil and water are pumped together into the upper end of a cracking tube provided with a number of coils situated in a chamber heated from 600° F. (about 320° C.) to 2000° F. (about 1100° C.). At the lower and hotter end of the tube there is a catalyst of nickel-chromium steel in the form of a woven net. The cracked vapours are led off through two condensers, in the first of which the cooling medium is the oil under treatment, which is thus preheated, while in the second water is used. The cracking tubes are arranged in a battery of six, each being supplied by means of a separate pump. An air pump is also connected with the top of each cracking tube in order to blow out the tube when necessary and remove deposits of carbon. A temperature of 1100° F. (about 590° C.) and a pressure of 150 lb. per sq. in. in the cracking tubes are stated to give excellent results.—L. A. C.

*Hydrocarbon products; Extraction of — from shales and coal.* D. T. Day, Washington, D.C., Assignor to V. L. Reed, Denver, Colo. U.S. Pat. 1,280,178, 1.10.18. Appl., 28.9.17.

SHALE or coal is subjected before distillation to the solvent action of an acid together with the vapours obtained by the destructive distillation of previously treated material.—L. A. C.

*[Mineral] oil refining [by distillation].* S. Brickman, Oklahoma, Okla. U.S. Pat. 1,279,506, 24.9.18. Appl., 3.9.17.

A CYLINDRICAL casing, spaced from the sides and top of a fireproof chamber, is provided with a number of partitions slightly inclined to the vertical, which are joined at their upper and lower edges so as to form a number of oil receptacles, the space above the receptacles forming a vapour-blending chamber. Oil is supplied to the receptacles, which are heated by means arranged between the receptacles and the bottom of the casing, an additional heating means being provided on the exterior of the casing. A flat superheater is disposed horizontally between the top of the casing and the top of the outer chamber, and an inlet from the vapour-blending space to the superheater and an outlet from the latter are also provided.—B. N.

*Oils; Process for cleaning waste [lubricating] —.* Patentierte Oelreinigungs-Fabrik A.-G., Budapest. Ger. Pat. 305,942, 11.10.16. Int. Conv., 2.5.16.

THE dirty oil is mixed with solid absorbent substances which are unaffected by oil and by water, added preferably in the form of a powder, so as to produce a pasty mass which is then subjected to compression. Suitable substances are sawdust, shavings, and the like, also cotton and tow.

—J. F. B.

*Asphalts; Process for the manufacture of artificial —.* R. Blum, Berlin-Grünewald. Ger. Pat. 307,961, 19.1.18.

INSTEAD of the usual asphalt materials, bituminous minerals are employed, particularly alum ore (alum earth), in combination with other known materials. The composition is used instead of tars or pitches, together with limestone for stamped asphalt, and on account of its high content of sulphur it shows some similarity to natural Trinidad asphalt.

—J. F. B.

*Petroleum and other hydrocarbons; Method for treating — to produce hydrocarbons having lower boiling points.* C. P. Dubbs, Wilmette, Ill., U.S.A. Eng. Pat. 112,010, 31.10.17. (Appl. 15,868/17.) Int. Conv., 27.11.16.

SEE U.S. Pat. 1,231,509 of 1917; this J., 1917, 921.

*Hydrocarbons; Vaporising — and producing hydrocarbons having low boiling points from hydrocarbons having high boiling points.* D. T. Day, Washington, D.C., and R. B. Day, Swarthmore, Pa., U.S.A. Eng. Pat. 119,440, 16.11.17. (Appl. 16,899/17.) Int. Conv., 28.9.17.

SEE U.S. Pat. 1,280,179 of 1918; preceding.

*Gas-fired furnaces.* Eng. Pat. 120,076. See 1.

*Bringing gases and liquids into contact.* Eng. Pat. 120,304. See 1.

*Cooling distillation residues.* Eng. Pat. 120,329. See 11b.

*Ammonium chloride, etc.* Eng. Pat. 119,971. See VII.

## IIb.—DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

*Decolorising carbon.* Zerban and Freeland. See XVII.

### PATENTS.

*Distillation or carbonisation of coal and cannel; Apparatus for the —.* J. T. Key, Ashbourne. Eng. Pat. 120,139, 14.12.17. (Appl. 18,578/17.)

A NUMBER of retorts in which coal or cannel is carbonised are connected with a mixing chamber provided with a series of perforated grids. When the process of carbonisation of the coal in the retort is completed, steam is introduced by a series of jets into the incandescent retort and passes through the coke into the mixing chamber. The gas from contiguous retorts entering the mixing chamber is thus subjected to the influence of this steam and a separation of the heavier impurities in the gas is effected.—J. S. G. T.

*Distillation of carbonaceous substances; Destructive —.* S. N. Wellington, London. Eng. Pat. 120,223, 30.7.17. (Appl. 10,973/17.)

THE gas and vapour produced by the low temperature distillation of carbonaceous substances is cooled and passed through a tar extractor to remove tar, and is then passed through a heat exchanger in which it is heated by means of producer gas. A form of heat exchanger consisting of a series of vertical tubes enclosed in a casing is more particularly described. The gas passes downwards through the tubes in one half of the casing and upwards in the remaining tubes. The heating gases circulate in the casing in counter-current to the gas to be heated. The gas thus reheated is passed direct to a saturator to recover its ammonia.

—J. S. G. T.

*Distillation of carbonaceous substances.* E. R. Sutcliffe, Leigh, Assignor to Pure Coal Briquettes, Ltd., Cardiff. U.S. Pat. 1,279,172, 17.9.18. Appl., 12.4.15.

A CONTINUOUS process of carbonisation is carried out in a series of ovens so connected that the heat



necessary for the distillation of the charge in an oven is obtained by the combustion of gas which has been preheated by passage through another oven of the series. The charges in the several ovens are distilled in succession, and the hot gases pass through other ovens of the series, thereby heating the charge in such ovens.—J. S. G. T.

*Cooling of hot residues from distillation processes.* B. E. D. Kilburn, London. From Sulzer Frères, Soc. Anon., Winterthur, Switzerland. Eng. Pat. 120,329, 7.3.18. (Appl. 3997/18.)

The residual material from the distillation process, e.g., coke, is discharged into a series of water-jacketed receptacles. The water jackets are all connected with a suitable steam generator, and are surrounded with air chambers furnished with baffle-plates. Air is circulated by means of a blower through the closed circuit consisting of the receptacle containing the coke and the air chambers, whereby the heat of the coke is transferred to the water in the jackets. The rate of circulation of the air can be varied and the direction of the circulation reversed.—J. S. G. T.

*Electric [incandescence] lamp.* J. A. Orange, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,279,415, 17.9.18. Appl., 12.9.14.

An electric lamp comprises an envelope filled with an inert gas, such as argon, at a pressure of about 150 to 200 mm. of mercury. Tungsten electrodes, of about 40 "mils" diameter and separated by a space of about 2 to 10 "mils," are so proportioned in shape and mass as to be adapted for heating to incandescence by an arc operating between the electrodes, "at a lighting efficiency materially higher than one watt per candle power." The current-carrying conductors for the electrodes have a diameter of about 20 "mils," but adjacent to the electrodes have a cross section equal to a diameter of 4 to 8 "mils."—B. N.

*Ionic discharges [for light projection apparatus]: Method of controlling and apparatus for producing* — D. F. Comstock, Brookline, Assignor to Kalmus, Comstock, and Wescott, Inc., Boston, Mass. U.S. Pat. 1,279,990, 24.9.18. Appl., 14.11.16.

SEE Eng. Pat. 110,982 of 1916; this J., 1918, 4A.

### III.—TAR AND TAR PRODUCTS.

*Tars distilled from bituminous coal in hand-fired furnaces.* S. H. Katz. U.S. Bureau of Mines, Tech. Paper 195, 1918, 20 pages.

The quantity and composition of the tar produced in two types of hand-fired furnaces were determined. The one furnace had a grate area of about 1 sq. ft., the other a grate area of about 30 sq. ft. A fuel bed of 6 in. depth was maintained. The air supply was measured. Various rates of firing were employed. Samples of gas were withdrawn by water-cooled sampling tubes inserted radially at various points below, above, and at the surface of the fuel bed. The tar and soot contained in the samples of gas were retained by an alundum thimble containing asbestos, and were extracted with benzol in a Soxhlet apparatus. The tar was collected over a period of months before sufficient was obtained for experimental purposes. The main conclusions are as follows: Qualitatively, the tars obtained were much the same as Jones and Wheeler's "primary volatile matter" (this J., 1914, 192). When coal is added to fires in uniform quantities and at short regular intervals, the

greatest quantity of tar in the gases is at the surface of the bed. At a point 1 ft. above the bed of coal burning in a large hot furnace at moderately rapid rates, practically all tar had disappeared. Naphthalene and anthracene were absent from the tars collected. The tar that escapes burning is decomposed to soot and fixed gases, and such decomposition occurs in less than 0.1 sec.—J. S. G. T.

*s-Chlorobenzenedisulphonic acid and some of its derivatives.* S. C. J. Olivier. Rec. Trav. Chlm. Pays-Bas, 1918, 37, 307—314.

1-Chlorobenzene-3,5-disulphonic acid, decomposing at 100° C., is obtained by heating chlorobenzene with five times its volume of fuming sulphuric acid containing 20% of sulphur trioxide at 300° C. for six hours. It yields a dichloride, m.pt., 105.5°—106° C., which when heated in a sealed tube with phosphorus pentachloride for four hours at 200°—210° C. yields s-trichlorobenzene. 1-Chlorobenzene-2,4-disulphonic acid is obtained by diazotising 2,4-disulphanilic acid in hydrochloric acid solution and decomposing the product with copper.—W. G.

*Diphenylamine; Nitro-derivatives of* — H. Ryan and T. Glover. Proc. Roy. Irish Acad., 1918, 34 [B], 97—105.

CONSIDERABLE discrepancies appear in the literature of the nitrodiphenylamines. With the object of removing these, the authors have prepared a series of substances by synthetic methods, that due to Goldberg (Ger. Pat. 185,663), in which aromatic amines are coupled with the halogen derivatives of aromatic nitro-compounds in nitrobenzene solution in the presence of potassium carbonate and cuprous iodide, being chiefly used. The following compounds are described: —p-nitrodiphenylamine, m.pt. 133°—134° C., which, contrary to Goldberg's statement (*loc. cit.*), yields a colourless solution in concentrated sulphuric acid; m-nitrodiphenylaminosamine, colourless, acicular crystals, m.pt. 89°—90° C.; 2,4-dinitrodiphenylaminosamine, pale yellow prisms, m.pt. 149°—151° C. (by the action of isoamyl nitrite on a cold solution of 2,4-dinitrodiphenylamine in glacial acetic acid; at a slightly higher temperature, 2,4,2',4'-tetranitrodiphenylamine slowly separates); 3,4'-dinitrodiphenylamine, pale yellow crystals, m.pt. 210°—212° C. after softening at 205° C.; 2,4,6-trinitrodiphenylamine, scarlet red prisms, m.pt. 178° C.; 2,4,3'-trinitrodiphenylamine, brown platy crystals, m.pt. 193°—194° C.; 2,4,3'-trinitro-5-methyldiphenylamine, dark yellow prisms, m.pt. 199° C. (slight decomp.); 2,4,4'-trinitro-5-methyldiphenylamine, straw-coloured, prismatic needles, m.pt. 210° C. (slight decomp.); 2,6,3'-trinitro-3-methyldiphenylamine (?), prismatic needles, m.pt. 199° C. (decomp.); 2,4,2',4'-tetranitrodiphenylamine, brownish prisms, m.pt. 199°—200° C.; 2,4,6,3'-tetranitrodiphenylamine, short yellow prisms, m.pt. 210° C. (corr.); 2,4,6,4'-tetranitrodiphenylamine, golden yellow prisms, m.pt. 222° C. m-Nitrodiphenylaminosamine is converted by nitric acid in glacial acetic acid solution into trinitrodiphenylaminosamine, yellow prismatic needles, m.pt. 184°—185° C. (decomp.) after softening at about 179° C. 2,4,3'-Trinitrodiphenylamine yields tetranitrodiphenylamine, yellow crystals, m.pt. 190° C., when treated with isoamyl nitrite. Under similar circumstances, perylaniline gives two compounds one of which, m.pt. 236° C., is probably 2,4,6,2',4',6'-hexanitrodiphenylamine, whilst the other, m.pt. 193°—194° C., appears to be 2,4,6,2',4'-pentanitrodiphenylamine. —H. W.

*Aromatic hydrocarbons.* Davidson. See 11A.

## PATENTS.

*Sulphonation of [volatile] hydrocarbons and hydrocarbon derivatives.* C. R. Downs, Cliffside, N.J., Assignor to The Barrett Co. U.S. Pats. (A) 1,279,295 and (B) 1,279,296, 17.9.18. Appl., 5.1.18.

(A) BENZENE, or other volatile hydrocarbon or hydrocarbon derivative, is treated with sufficient sulphuric acid to form the disulphonic acid, first at a low temperature and atmospheric pressure, whereby the non-volatile benzenemonosulphonic acid is formed, and then at the temperature necessary for the formation of the disulphonic acid (below 260° C.) and under a pressure sufficiently reduced to remove the water of reaction and maintain the acid at the strength required for the completion of the reaction.

(B) Benzene is introduced progressively into a body of sulphonating acid maintained at the sulphonating temperature, i.e., above the boiling point of benzene, thereby effecting intimate contact of benzene vapour with the acid. The reaction mixture, as in (A), is subjected to a reduced pressure.

—L. A. C.

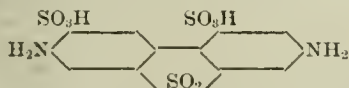
*Toluene; Process of chlorinating* — C. C. Loomis, Syracuse, N.Y., Assignor to Smet-Solvay Co., Solvay, N.Y. U.S. Pat. 1,280,612, 1.10.18. Appl., 18.10.17.

TOLUENE is heated with dry bleaching powder without the addition of an acid or other substance for decomposing the bleaching powder.—L. A. C.

## IV.—COLOURING MATTERS AND DYES.

*[Azo] dyes derived from benzidine-sulphone; Synthesis of some new substantive* — H. Ryan, J. Algar, and P. O'Connell. Proc. Roy. Irish Acad., 1918, 34 [B], 85–96.

A SERIES of dyes of the benzidine type has been prepared by coupling hydroxy- and amino-compounds with the tetrazo-derivative of benzidine-sulphone-disulphonic acid,



The dyes have been isolated in the form of pure sodium salts; they act as direct dyes towards cotton and the colours are unaffected by washing. Products have been obtained with the following substances, the shade obtained on cotton being placed within brackets: naphthionic acid, dark-blue, amorphous powder (purple);  $\beta$ -naphthylamine, reddish amorphous powder (violet-red);  $\alpha$ -naphthylamine, dark-red powder (navy blue); salicylic acid, reddish-brown powder (orange); "R" acid, reddish-blue powder (violet-red); "G" acid, reddish amorphous powder (pink); "H" acid, dark-blue powder (light blue);  $\beta$ -naphthol-6-monosulphonic acid, dark-bluish amorphous powder (light purple); catechol, dark-bluish powder (light brown); resorcinol, dark-bluish powder (maroon); quinol, brownish powder (buff); pyrogallol, chocolate-brown powder (buff); gallic acid, dark-brown powder (light brown); sulphanilic acid, orange-red amorphous powder (canary yellow); dimethylaniline, dark-bluish powder (deep purple).—H. W.

*Alizarin; Action of potassium ferrieyanide on — in alkaline solution and the constitution of salts of hydroxyanthraquinones.* R. Scholl and A. Zinke. Ber., 1918, 51, 1419–1435.

ALIZARIN is oxidised to 2-hydroxy-1,4-naphthoquinone-3-vinylglyoxylic acid,  $\text{HO.C}_6\text{H}_4\text{O}_2\text{CH}:\text{CH.CO.CO}_2\text{H}$ , yellow leaflets, m.pt. about 230° C. (decomp., darkening above 150° C.), by an alkaline solution of potassium ferrieyanide at the ordinary temperature. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Anthraquinone-fluorenone series; Rearrangement reactions in the* — A. Schaarschmidt and J. Herzenberg. Ber., 1918, 51, 1230–1237.

ANTHRAQUINONE-2,1-fluorenone is converted into anthraquinone-2,3-fluorenone by fusion with potassium hydroxide at 220°–230° C. and treatment of the product with concentrated sulphuric acid. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Curcumin; Synthesis of* — V. Lampe. Ber., 1918, 51, 1347–1355.

THE condensation of ethyl sodioacetoacetate and carbomethoxyferuloyl chloride yields ethyl  $\alpha$ -carbomethoxyferuloylacetoacetate,  $\text{CO}_2\text{CH}_3\text{O.C}_6\text{H}_4\text{CH}(\text{OCH}_3)\text{CH}:\text{CH.CO.CH}(\text{CO.CH}_3)\text{CO}_2\text{C}_2\text{H}_5$ , from which, by the successive processes of hydrolysis, condensation with carbomethoxyferuloyl chloride, and hydrolysis, are successively obtained carbomethoxyferuloylacetone, dicarbomethoxydiferuloylacetone (not isolated), dicarbomethoxydiferuloylmethane, and diferuloylmethane, the last-named substance being identical with natural curcumin, which thus has the constitution  $\text{CH}_2(\text{CO.CH}:\text{CH.C}_6\text{H}_4[\text{OH}]\text{OCH}_3)_2$  suggested by Milobedzka, Kostanecki, and Lampe in 1910 (this J., 1910, 1002; see also J. Chem. Soc., Jan., 1919).

—C. S.

*pp'-Dihydroxy- and p-hydroxy-dicinnamoylmethane; Syntheses of* — V. Lampe and M. Godlewska. Ber., 1918, 51, 1355–1360.

THE syntheses follow the same course as that of curcumin (see preceding abstract). Ethyl  $\alpha$ -p-carbomethoxycinnamoylacetoacetate,  $\text{CO}_2\text{CH}_3\text{O.C}_6\text{H}_4\text{CH}:\text{CH.CO.CH}(\text{CO.CH}_3)\text{CO}_2\text{C}_2\text{H}_5$ , p-carbomethoxycinnamoylacetone, p-hydroxycinnamoylacetone, pp'-dicarbomethoxydicinnamoylmethane, pp'-dihydroxydicinnamoylmethane, and p-hydroxydicinnamoylmethane are described. The last two compounds are compared with curcumin with respect to their dyeing action on mordanted cotton and to the change of colour produced by boric acid. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Isobrazilein and certain related anhydropyranol salts; Synthesis of* — I. H. G. Crabtree and R. Robinson. Chem. Soc. Trans., 1918, 113, 859–880.

DIBYDROBUTEIN trimethyl ether (2-hydroxy-4-methoxyphenyl  $\beta$ -3,4-dimethoxyphenylethyl ketone), colourless prisms, m.pt. 84° C., obtained by reducing a solution of veratrylidene-paeanol in acetic acid at 70°–80° C. by hydrogen and palladium chloride, was converted into isobrazilein ferriehloride trimethyl ether by boiling with zinc chloride and anhydrous formic acid and treating the cooled solution with dilute hydrochloric acid and an excess of concentrated ferrie chloride solution.

Appendix I. [R. Robinson and M. R. Turner]. The preparation of 7-methoxy-2-phenyl-1,4-benzopyranol anhydrochloride, orange needles, m.pt. 102°–103° C. (decomp.), and of 7-methoxy-2,4-diphenyl-1,4-benzopyranol, m.pt. 55°–57° C., is described.



*Appendix II.* [J. A. Prescott]. The preparation is described of 7-hydroxy-4,3-indeno-1,2-benzopyrone, colourless needles, darkening at about 230° C. and becoming blue at 280° C. This is the first compound to be synthesised the molecule of which contains the brazilin and hæmatoxylin skeleton.—C. S.

#### PATENTS.

*Dye [of the Sulphur Black type] and process of making same.* C. Ellis, Montclair, N.J., Assignor to Ellis-Foster Co. U.S. Pat. 1,279,307, 17.9.18. Appl., 26.6.16.

DIESTUFFS of the Sulphur Black type, free from sulphur, and capable of being dried at atmospheric pressure without decomposition, are prepared by heating a nitrated aromatic phenol (dinitrophenol) with an equivalent quantity of an aqueous alkaline sulphide under a pressure equal to or greater than that of the atmosphere until complete reduction of the nitro groups is effected.—L. A. C.

*Soluble dye in sheet form.* F. Fear, Brooklyn, N.Y. U.S. Pat. 1,279,679, 24.9.18. Appl., 15.2.18.

A FIBROUS web is produced by beating unbleached paper stock composed of cotton fibres in a solution of sulphuric acid. The web is dried, led through a dye-bath, and again dried so as to crystallise the dyestuff upon the surface of the web.—L. A. C.

*Anthracene derivative [pyranthridone]; Production of an —.* R. Scholl, Dresden. Ger. Pat. 307,399, 11.11.16.

A NEW compound, pyranthridone (this J., 1918, 364 A), applicable in the dye industry, is prepared by the action of dehydrating agents on 5-methyl-3,4-phthaloyl-S-(CO)-9-benzoylenephenaanthridine.

—J. H. L.

*Anthracene dyes and process of making same.* R. Bohn, Mannheim, Assignor to Badische Anilin und Soda Fabr., Ludwigshafen, Germany. U.S. Pat. 1,280,648, 8.10.18. Appl., 8.5.15.

SEE Eng. Pats. 100,580 and 100,581 of 1915; this J., 1916, 831.

*Azo dye and process of making.* E. F. and H. W. Ehrhardt, Birmingham. U.S. Pat. 1,281,243, 8.10.18. Appl., 4.1.18.

SEE Eng. Pat. 113,181 of 1917; this J., 1918, 202 A.

## V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

#### PATENTS.

*Viscose; Manufacture of threads from —.* T. H. Verhave, The Hague, Netherlands. U.S. Pat. 1,280,338, 1.10.18. Appl., 23.2.15.

SEE Eng. Pat. 2485 of 1915; this J., 1916, 250. It is specified that the spinning bath should contain at least 3 mols. SO<sub>4</sub> ions to each 2 mols. H-ions."

*Composition or pulp [for paper-making].* U. Okazaki, Kitaguchi, Assignor to H. E. Ridings, Yokohama, Japan. U.S. Pat. 1,278,199, 10.9.18. Appl., 1.11.17.

A FIBROUS composition or paper-making pulp is prepared by treating sugar-cane leaves with water, caustic soda, lime, bleaching powder, and sulphuric acid.—J. F. B.

*Paper; Means for imparting surface finish to —.* H. P. Howe, Steep Falls, Assignor to Eastern Manufacturing Co., South Brewer, Me. U.S. Pat. 1,278,659, 10.9.18. Appl., 2.2.18.

THE machine comprises a pair of curved finishing plates covered with linen fabric which is maintained under tension on the convex surface of each plate. The curved plates are oscillated in tangential contact with each other between a pair of calender rolls. Means are provided for feeding sheets of paper between the two oscillating plates and for stacking them in a receiving box after passing between the rolls. A jet of air is directed under the finished sheets to lift them from the finishing plates for delivery to the stack. Feeding and receiving arrangements are provided on both sides of the machine so that the plates are utilised in both strokes of their oscillation.—J. F. B.

*Paper; Method and machine for making —.* C. E. Pope, Springfield, Mass., Assignor to Great Northern Paper Co., Millinocket, Me. U.S. Pat. 1,279,756, 24.9.18. Appl., 14.3.18.

To remove the paper web from the papermaking wire at the beginning of the operation of the machine or after a break, a narrow strip of the paper is formed at one edge of the wire and two oppositely directed transverse jets of air are directed against the two edges of the strip at the point where the strip leaves the wire, so as to meet behind the strip. The adhesion of the strip of paper to the wire is thus broken at this point, and the strip deflected from the roller so as to fall on to the press roll felt.—W. F. F.

*Paper; Manufacture of acid- and weatherproof —.* Heddernheimer Kupferwerk und Süddeutsche Kabelwerke A.-G., Abt. Süddeutsche Kabelwerke in Mannheim. Ger. Pat. 307,867, 5.1.18.

AN oxidising agent such as red lead or the like is incorporated with the paper pulp and the dry finished paper is subsequently impregnated with a drying oil, which is oxidised in the paper to a coherent protective layer capable of resisting acids and moisture.—J. F. B.

*Sulphite-cellulose waste lyes; Purification of — by treatment with flue gases.* H. Achenbach, Nussdorf. Ger. Pat. 306,898, 17.6.15.

THE spent liquor from the digester, which may undergo a preliminary purification by spraying, is fed into the top of a scrubbing tower packed with stones and allowed to trickle down in contact with ascending hot flue gases. The concentrated liquor is collected at the bottom of the tower, whilst the vapours containing sulphur dioxide and volatile acids pass away through the hood of the tower, together with the exhaust gases, into a horizontal tubular cooler containing arrangements which facilitate the maximum utilisation of the cooling water. The condensed water containing sulphur dioxide in solution collects in the lower part of the cooler and is drawn off to a reservoir for the production of fresh liquor. The gases, also containing some sulphur dioxide, pass through a pipe to an absorption tower.—J. F. B.

*Sulphite-cellulose waste lyes; Recovery of useful organic and inorganic substances from — by heating in autoclaves.* R. W. Strehlenert, Göteborg, Sweden. Ger. Pat. 308,144, 1.4.17.

THE waste lye is treated in an autoclave with gases or vapours of an acid character, for instance, the exhaust gases from the sulphite pulp digesters con-

taining sulphur dioxide. The gas is forced into the autoclave at a pressure which is substantially higher than that corresponding to the temperature of the liquor therein, for instance, with the liquor heated at 190° C. the gas pressure may amount to 20 atm. The liquor may be heated in a series of interconnected pressure vessels fed with gas from the autoclave in front of them. An oxidising gas, such as air, may be mixed with the acid gases employed for the reaction. The sulphur dioxide in the liquor is converted by the treatment, according to the equation  $3\text{SO}_2 = 2\text{SO}_3 + \text{S}$ , into sulphuric acid, which causes the precipitation of the organic matter.—J. F. B.

*Paper twine or yarn; Manufacture of* —. A. D. Spicer, and J. Spicer and Sons, Ltd., London. Eng. Pat. 129,236, 19.10.17. (Appl. 15,207/17.)

*Cellulose acetate and process of making same. Process of making cellulose acetate insoluble in chloroform and in chloroform-alcohol.* H. Dreyfus, Basle, Switzerland. U.S. Pats. 1,280,974 and 1,280,975, 8.10.18. Appls., 4.5.15 and 15.6.16.

SEE FR. Pat. 475,160 of 1914; this J., 1916, 40.

*Drying yarns, jute, etc.* Eng. Pat. 120,277. See XV.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

### PATENT.

*Fabric dyeing processes [using organic solvents].* W. P. Thompson, Liverpool. From V. Planté, Marseilles, France. Eng. Pat. 119,881, 17.8.17. (Appl. 11,825/17.)

Wool, silk, etc., are dyed with basic dyestuffs dissolved in carbon tetrachloride, to which alcohol may be added, in the presence of a mordant termed "gallo-oleate of ammonia." The mordant is prepared by mixing 55 parts of triolein, 5.5 of alcohol, 1 of gallic acid, and 13 of 22% ammonia solution, and stirring until saponification is complete. The dye-bath, suitable for dyeing 66 lb. of material, is composed of: carbon tetrachloride, 25.5 galls.; alcohol, 17 galls.; mordant, 44 lb.; basic dyestuff, 22 lb. The mixture is heated to 145° F. (62.5° C.) and dyeing is carried out in an iron vessel with occasional agitation for 3–4 hours. During the operation an electric current, preferably of 250 amps. at 450 volts, is passed through the bath. The dyed material is drained in a hydro-extractor, dried, and treated in a vat containing a solution of 6.5 lb. of "gallo-oleate of ammonia" in 23 galls. of petroleum spirit, and finally rinsed in fresh petroleum spirit. —J. F. B.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

*Nitre cake; Summary of the literature on the solubility of systems relating to* —. H. W. Foote. J. Ind. Eng. Chem., 1918, 10, 896–897.

A RÉSUMÉ of the literature dealing with the solubility relationships of the systems,  $\text{Na}_2\text{SO}_4\text{—H}_2\text{SO}_4\text{—H}_2\text{O}$ ,  $\text{Na}_2\text{SO}_4\text{—KSO}_4\text{—H}_2\text{O}$ , and  $\text{KSO}_4\text{—H}_2\text{SO}_4\text{—H}_2\text{O}$ .—J. F. S.

*Nitre cake; Recrystallisation of* —. B. Saxton. J. Ind. Eng. Chem., 1918, 10, 897–901.

Using the solubility data of D'Ans and Pascal (this J., 1906, 534; 1917, 594) equations have been developed for the efficient working of the following processes: (1) separation of any one solid phase of the system  $\text{Na}_2\text{SO}_4\text{—H}_2\text{SO}_4\text{—H}_2\text{O}$ , (2) separation of a stated amount of one solid phase of the same system, (3) separation of the maximum amount of each solid phase. These equations are for the temperatures 0° C. and 25° C. Leaching processes are outlined, by means of which sulphuric acid may be concentrated in the solution and sodium sulphate in the solid. It is found experimentally that this separation can be carried out more efficiently at 0° than at 25° C.—J. F. S.

*Kaolin rich in iron; Utilisation of* — [for manufacture of alum or aluminium sulphate]. J. Milbauer and F. Skutil. Chem.-Zeit., 1918, 42, 525–527.

For the manufacture of alum or aluminium sulphate from kaolin containing a large proportion of iron, the best method consists in heating the mineral at 700° C. with the addition of sawdust; air must be excluded during the heating. The calcined material is then heated with sulphuric acid (50° B., sp. gr. 1.53); the ferrous sulphate remains in solution when the aluminium sulphate is separated by crystallisation. Traces of ferric salts may be removed from the aluminium sulphate solution by adding potassium ferrocyanide and filtering, a small quantity of kieselguhr being added to facilitate the filtration. If, after the iron salts have been reduced, the aluminium sulphate is allowed to crystallise in an atmosphere free from oxygen, the crystals obtained contain but very little iron.—W. P. S.

*Carbon monoxide; Oxidation of* — in contact with copper. K. A. Hofmann. Ber., 1918, 51, 1334–1346.

In contact with metallic copper moistened with aqueous potassium hydroxide, carbon monoxide mixed with air is oxidised two or three times more rapidly than it is when in contact with a copper oxide surface of the same area. The copper is converted into a peroxide,  $\text{Cu}_2\text{O}_2$  or  $\text{CuO}_2$ , which is the active oxidising agent. The rate of oxidation is increased when the copper surface is coated with a trace of iridium. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Carbon monoxide; Activation of* — by metallic copper, the producer gas-cell, and the electro-motive activation of alkali formates. K. A. Hofmann. Ber., 1918, 51, 1526–1537.

The author's discovery (see preceding abstract) that carbon monoxide can be rapidly oxidised at the ordinary temperature has enabled him to realise the producer gas-cell  $\text{O}_2 | \text{Cu} | \text{alkali} | \text{Cu} | \text{CO}$ , and the drawbacks of most of such cells have been overcome (subject of a censored patent). The E.M.F. ( $\text{CO} \rightarrow \text{CO}_2$ ) is found to be 1.32 volts at 20° C., which compares well with the value 1.343 at 17° C. calculated by Nernst and von Wartenberg from the dissociation of carbon dioxide. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Hydrogen; Detection and determination of hydrogen phosphide in* —. J. Soyer. Ann. Chim. Analyt., 1918, 23, 221–225.

HYDROGEN prepared for aeronautical purposes by the action of sodium hydroxide on ferrosilicon always contains traces of hydrogen phosphide (from 1 in 15,000 to 1 in 40,000 vols., or less). The presence of



the hydrogen phosphide may be detected by the green coloration of the flame when the gas is burnt from a platinum jet, by the spectrum (phosphorus lines) exhibited by the flame, and by the detection of phosphoric acid when a drop of water is held in the flame for 15 secs. and then tested with molybdic acid reagent. To determine the hydrogen phosphide, from 2 to 20 litres of the hydrogen is conducted together with a large excess of air to a platinum jet fitted in a silica tube heated to bright redness. This tube is inclined slightly, and the lower end is cooled and connected with a series of absorption vessels containing water. The combustion of 20 litres of the gas takes about 1 hr. At the end of the operation, the contents of the absorption vessels are transferred to a basin, the silica tube is also rinsed out with water, and, after the addition of 5 grms. of ammonium nitrate, the solution is concentrated to about 40 c.c., and the phosphoric acid precipitated in the usual way with molybdic acid reagent. If the yellow precipitate obtained is very small, its quantity may be determined colorimetrically.—W. P. S.

*Gas purification.* Parrish. See IIA.

• *Lime-alumina-silica.* Neumann. See X.

*Colloidal silicic acid.* Mary. See XVII.

#### PATENTS.

*Sulphuric acid [and zinc]; Manufacture of —.* J. F. Cullen, Midvale, Utah, Assignor to United States Smelting, Refining, and Mining Co. U.S. Pat. 1,278,308, 10.9.18. Appl., 13.9.17.

In a cyclic process, zinc ore is dissolved in sulphuric acid, manganese dioxide (ore) is added to the solution, and the mixture is treated with sulphur dioxide to produce a solution of zinc and manganese sulphates. The latter is then electrolysed, an insoluble anode being employed, under conditions whereby zinc is deposited at the cathode while sulphuric acid and manganese dioxide are produced at the anode.—W. E. F. P.

*Evaporating furnaces for surface heating, particularly for the concentration of acids.* Chem. Fab. zu Schöningen, and R. Vetterlein. Ger. Pat. 307,564, 27.8.16.

THE liquid to be evaporated and the heating gases are passed in contact in opposite directions and in a horizontal zig-zag path through the evaporator, consisting of a metal pan built in a masonry setting, and through a preliminary evaporator consisting of a reaction tower built above one end of the metal pan. The liquid is sprayed into the top of the tower, which may be filled with packing, and meets the ascending exhaust gases from the main evaporator; it is collected in the pan beneath and flows in contact with the furnace gases through the pan, which is divided by partitions into zig-zag channels, to the outlet near the furnace. The pan may be constructed of wrought iron, and supported on pillars in the masonry setting. In the evaporating space all baffling obstructions are avoided, and doors are fitted in the walls at the ends of the channels to facilitate cleaning of the trough.—J. F. B.

*Aluminous compounds [from clay, etc.]; Production of —.* W. B. Llewellyn, H. Spence, and P. Spence and Sons, Ltd., Manchester. Eng. Pat. 119,924. (Appls. 15,711, 29.10.17 and 3152, 22.2.18.) Addition to 112,881, 11.5.17 (this J., 1918, 149 A).

A hot or boiling solution of sulphuric acid, or of aluminium sulphate containing free sulphuric acid,

is circulated repeatedly through a mass composed of small lumps of clay, fireclay, lithomarge, or similar material, preferably ignited previously, until the alumina is completely extracted from the material, and a concentrated, acid or basic solution of aluminium sulphate is obtained for use as such or for conversion into other compounds of aluminium. The process is more particularly applicable to clays, etc., possessing, naturally or as the result of ignition, a structure permitting of ready circulation of liquid through the mass. Aluminous minerals containing much potash are unsuitable.—W. E. F. P.

*Ammonium chloride from ammoniacal liquor; Recovery of —, with the extraction of cyanides and other valuable products.* H. Baker, Barrow-in-Furness. Eng. Pat. 119,971, 22.1.18. (Appl. 1211/18.) Addition to 112,329, 17.1.17 (this J., 1918, 88 A).

AFTER the removal of free ammonia by boiling, the liquor is concentrated or evaporated to dryness before recovering the cyanides and ammonium chloride by the original process. The waste gases from the ammonia-recovery plant are employed to precipitate any metals remaining in solution after recovering the cyanides.—W. E. F. P.

*Caustic soda and the like; Machine or apparatus for breaking up or pulverising —.* W. Blacker, Stalybridge. Eng. Pat. 120,309, 21.1.18. (Appl. 1154/18.)

In an apparatus for breaking up caustic soda, etc., which has been allowed to set in drums or other containers, the container is struck repeatedly, and in different places, by a power hammer having one or more striking heads, whilst supported upon and moved by a mechanical cradle actuated by the hammer device.—W. E. F. P.

*Cream of tartar from wine-ices.* H. Tobler, Hackensack, N.J. U.S. Pat. 1,278,257, 10.9.18. Appl., 9.11.17.

A MIXTURE of wine-lees and sulphuric acid of strength 10° B. (sp. gr. 1.074) is heated to about 60° C., with agitation, and filtered, and the solution is treated with potassium compounds and alkali to produce cream of tartar. The mother liquor, from which the latter is separated, is treated with a calcium salt to precipitate calcium tartrate which is added to a subsequent charge of wine-lees.

—W. E. F. P.

*Cyanogen compounds; Production of —.* J. D. Morgan, New York. U.S. Pat. 1,278,493, 10.9.18. Appl., 30.6.17.

AN intimate mixture of an alkali-metal compound and carbonaceous matter is showered into a heated reaction chamber in which an atmosphere consisting largely of nitrogen is maintained. The cyanogen compound formed adheres to the walls of the chamber, and is removed periodically while in a plastic condition.—W. E. F. P.

*Barium chloride, magnesium chloride, etc.; Process for the production of —.* S. H. Dolbear and J. W. Beckman, San Francisco, Cal. U.S. Pat. 1,279,090, 17.9.18. Appl., 5.10.15.

A BARIUM compound is mixed with sodium chloride, heated to 1500°–2500° F. (815°–1370° C.), and treated with water, whereby the sodium chloride dissolves and the barium compound remains in a finely divided condition. The mixture is evaporated to dryness, mixed with silica, and again heated to the above temperature.—W. F. F.

*Alum; Process of making* —. E. W. Haslup, Bronxville, N.Y., and B. A. Peacock, Philadelphia, Pa. U.S. Pat. 1,279,109, 17.9.18. Appl., 7.11.17.

AN alkali-metal alum is made from a mixture of a mineral containing alkali metal compounds (e.g., "green sand") and bauxite by digesting with sulphuric acid in a closed heat-insulated vessel. —C. A. K.

*Potassium sulphate; Process of obtaining* —. B. A. Peacock, Philadelphia, Pa., Assignor to Haslup and Peacock, Inc., New York. U.S. Pat. 1,279,145, 17.9.18. Appl., 8.2.18.

POTASSIUM in the form of sulphate is extracted from a hydrous mineral, such as glauconite, by mixing it with aluminium sulphate, the latter being present in a quantity only slightly in excess of that required to supply sufficient sulphuric acid to combine with the potassium present. The mixture is heated to a temperature above 100° C. to form potassium sulphate, which is then extracted by means of water.—B. N.

*Cerium-group metals; Process of obtaining compounds of* — from monazite sands. W. S. Chase, Lakewood, Ohio, Assignor to National Carbon Co., Inc. U.S. Pat. 1,279,257, 17.9.18. Appl., 20.11.16.

COMPOUNDS of metals of the cerium group are obtained by decomposing monazite sands by sulphuric acid, and then adding sodium bisulphate to precipitate double sulphates of sodium and these metals.—B. N.

*Perhalates [sodium perchlorate]; Process of producing* —. E. P. Schoch and R. H. Pritchett, Austin, Tex. U.S. Pat. 1,279,593, 24.9.18. Appl., 18.4.18.

SODIUM perchlorate is produced by electrolysis of an acid solution of sodium chlorate, an anode of gas retort carbon being employed.—W. E. F. P.

*Catalytic substances; Manufacture of active* —. F. Müller, Freiburg. Ger. Pat. 307,380. 26.3.16.

COMPLEX salts of the heavy metals, known as "Werner salts," are heated in absence of air at temperatures not exceeding those at which the catalytic reaction is to be carried out. The salts may be heated either in their original form or distributed on carrier masses in a current of nitrogen, or in an atmosphere of one or more of the gases with which the catalytic agent is to be used. Alternatively, the "Werner salts" may be finely divided and heated in the presence of liquids, except in the case of the complex salts of the platinum metals intended for use as catalysts in the process of fat hardening. The "Werner salts" comprise double salts and complex salts, such as potassium chromicyanide, ammonium ferrimolybdate, nitro-purpureonitrile, sodium-ammonium cobalticyanide, and carbonatopentammine cobalt nitrate for ammonia synthesis, cerocobalt nitrate for the production of formaldehyde from methane and oxygen, hexaquo-chromium acetate for the production of indole from methyl-o-toluidine and hydrogen, hexaquo-chromium propionate for production of camphor from a solution of borneol in paraffin oil, luteo-chromium ferri-cyanide for oxidation of ammonia, and cobalt hydroxynitrite for production of sulphur trioxide from sulphur dioxide and excess of oxygen.—J. F. B.

*Chalk; Light precipitated* — and processes of making same. N. Statham, Hastings-on-Hudson, N.Y., U.S.A. Eng. Pat. 120,219, 26.7.17. (Appl. 10,780/17.)

SEE U.S. Pat. 1,266,339 of 1918; this J., 1918, 415 A.

*Tungsten and nitrogen; Producing compounds containing* —. C. Bosch and A. Mittasch, Assignors to Badische Anilin und Soda Fabrik, Ludwigshafen, Germany. U.S. Pat. 1,278,580, 10.9.18. Appl., 11.11.12.

SEE Ger. Pat. 259,647 of 1911; this J., 1913, 657.

*Sodium sulphate; Process of manufacture of* —. L. P. Basset, Paris. U.S. Pat. 1,279,499, 24.9.18. Appl., 3.6.15.

SEE Fr. Pat. 477,353 of 1914; this J., 1916, 601.

*Acid-resisting cement.* Eng. Pat. 119,966. See IX.

*Manganese sulphate.* U.S. Pat. 1,279,110. See X.

## VIII.—GLASS; CERAMICS.

### PATENTS.

*Glass-furnace.* C. R. Hook, Coffeyville, Kans. U.S. Pat. 1,279,697, 24.9.18. Appl., 5.7.17.

TWO hot-air flues leading from the regenerators at opposite sides of a glass furnace communicate with an air-passage, having an outwardly flared end, connected to the chimney. A vertical "butterfly" damper is arranged so as to connect either of the two hot-air flues with the chimney and the other one with the air-passage.—A. B. S.

*Glass manufacture.* R. H. Bolln, Rochester, N.Y. U.S. Pat. 1,280,139, 1.10.18. Appl., 17.2.15.

GLASS is allowed to flow from a stationary tank furnace into a movable receptacle surrounded by an air-chamber through which the glass is drawn in a continuous sheet by a pair of revolving bodies, one on each side of the chamber, which press on the edges of the sheet. The latter is supported on a column of air under pressure moving in the same direction as the glass. The tension on the glass is varied by altering the level of the glass in the receptacle.—A. B. S.

*Separators; Centrifugal* — [for treating clay "slip"]. R. S. Brownlow, Manchester. Eng. Pat. 120,319. (Appls. 2564, 13.2.18, and 10,231, 21.6.18.)

A CYLINDRICAL rotating vessel with its axis vertical is provided with a removable lining flanged externally at the top so as to rest on the rim of the vessel and rotate with it. The solid constituent of the mixture treated is deposited on the lining, which may then be raised out of engagement with the vessel by means of a framework of vertical rods co-operating with the flange of the lining, so that the solids may be removed. A complete withdrawal of the lining may be avoided by providing a piston at the bottom of the vessel, which may be raised by a vertical rod, thus carrying the solids with it. The solids may be consolidated before removal by providing the lining with a removable internal flange at the top, against which the solids may be compressed by the piston. In another modification two or more lifting pistons of different diameters may be provided to withdraw the solids in portions of different densities, and in a further modification



pistons may be provided at different depths to separate the solids according to their axial grading. The apparatus is particularly applicable for treating clay "slip."—W. F. F.

*Continuous kiln for burning bricks, terra-cotta and the like.* E. Glossop and J. Sherlock, Ambergate. Eng. Pat. 120,052, 23.10.17. (Appl. 5764/17.)

A CONTINUOUS chamber kiln of the transverse arch type is provided with a longitudinal hot-air flue, the bottom of which is connected by a series of small damper-controlled flues with the several chambers of the kiln, and the top of it by a series of overhead flues and down-cast flues to openings in the walls of the chambers, and to the flue which runs longitudinally in the sole of the kiln. In order to dry freshly-set goods, a current of air is drawn from a chamber containing cooling goods, through an opening near the top of the longitudinal hot-air flue, and is discharged into the chamber containing the freshly-set goods, through an opening connected to the bottom of the hot-air flue. As this warm air becomes saturated with steam and moisture, it is drawn off through flues in the partition walls and through an opening near the floor of the chamber into the main flue, and thence to the chimney. By withdrawing the moisture at positions equally distributed across the kiln, scum is prevented and the faces of the goods are kept clean.—A. B. S.

*Tunnel-kilns; Method of and means for balancing draught in* —. J. B. Owens, Metuchen, N.J. U.S. Pat. 1,278,991, 17.9.18. Appl., 16.11.17.

THE ware is conveyed through the kiln by means situated below a horizontal platform extending across the kiln and engaging in longitudinal grooves at the sides, so that the kiln is divided into two separate compartments. The upper compartment containing the ware to be burned is strongly heated, and cooling air is admitted in regulated amount to the lower compartment to protect the conveying means. The air leaks slowly into the upper compartment through the spaces between the platform and the walls.—W. F. F.

*Carbon blocks; Manufacture of* —. T. W. S. Hutchins, Middletown. Eng. Pat. 120,255, 31.10.17. (Appl. 15,885/17.)

CARBON blocks are made by compressing the finely ground material in hydraulic or other presses, the cylinder of the latter being coated with a film of graphite or a mixture of graphite with oil or grease, applied either before or during the charging of the cylinder with carbon, so that the binding of the charge during compression will be minimised and the whole body be carried forward by the plunger of the press and form a uniform non-porous mass. This is accomplished by fitting a tubular liner to the cylinder, applying the lubricant to the outside of the liner, whilst the charge is fed through the centre of it. The lower part of the liner may be made larger than the upper, and may be provided with hinged tongues.—A. B. S.

*Abrasive.* F. M. Becket, Assignor to Electro Metallurgical Co., Niagara Falls, N.Y. U.S. Pat. 1,279,828, 24.9.18. Appl., 10.4.16.

CLAIM is made for an abrasive composed of irregularly shaped grains, of approximately uniform size, of the alloy described in U.S. Pat. 1,245,552 of 1917 (this J., 1918, 13a), and for abrasive wheels, etc., containing bonded grains of the alloy.

## IX.—BUILDING MATERIALS.

*Wood paving blocks; Increasing the resistance of* —. W. Ritter. Bitumen, 1917, 15, 148—150. Z. angew. Chem., 1918, 31, Ref., 308.

WOOD paving blocks in busy streets require renewal every 5—6 years, and attempts have been made to increase their strength by suitable impregnation. Tests were made of crushing resistance calculated per sq. cm. on prismatic blocks 75-75 sq. mm. in base area and 120 mm. in height, the pressure being applied in the direction of the fibres. Experiments in which sodium or potassium carbonate and sodium silicate in various proportions were added to an impregnating bath of pine wood tar showed no advantage, but increasing the temperature of the bath with the dry samples gave a 12% increase in strength. Experiments were also made by substituting heavy coal tar oil for the wood tar at temperatures from 112° to 140° C. At the higher temperature these trials gave as good results as those with wood tar, but the blocks impregnated at the lower temperature showed a crushing resistance inferior to that of the untreated blocks. Sodium carbonate alone, or still better sodium silicate alone, gave more favourable test results than the mixture of carbonate and silicate. The chemical composition of the impregnating bath, however, is of less importance than its temperature. The temperature should not be carried above 200° C. on account of the danger of splitting the blocks. The best strengthening results are obtained by impregnating for 3 hours with gradual increase of temperature up to 200° C. A test block showed a crushing strain of 323 kilos. per sq. cm. In the untreated condition; this was increased by impregnation at 100° C. to 330 kilos., at 120° C. to 345, at 140° to 487, at 150° to 516, and at 200° to 536 kilos.—J. F. B.

*Lime-alumina-silica.* Neumann. See X.

### PATENTS.

*Insulating material for cold storage.* L. G. Surie, and Surlstone, Ltd., London. Eng. Pat. 119,783, 17.1.18. (Appl. 992/18.)

GRANULATED cork, cork dust, sawdust, or the like, 2 lb., silica wool, 0.25 lb., casein, 1 lb., lime, 0.5 lb., and water, 5 lb., are mixed and moulded into slabs, which are hardened in a bath of formaldehyde, and dried. The dried slabs are treated with calcium tannate solution to render them damp-proof.

—W. F. F.

*Heat-insulating material and method for manufacturing same.* P. A. Boeck, New York, and W. L. Jordan, Glendale, Cal. U.S. Pat. 1,279,975, 24.9.18. Appl., 12.3.18.

A HEAT-INSULATING material is produced by forming a suspension of hydrated basic magnesium carbonate in water, adding an alkali hydroxide, heating the mixture, and then adding diatomaceous earth and fibrous material. Part of the magnesium reacts with the silica in the diatomaceous earth, forming magnesium silicate.—A. B. S.

*Cement; Acid-resisting* —. Chance and Hunt, Ltd., A. E. Holley, and H. W. Webb, Oldbury. Eng. Pat. 119,966, 12.1.18. (Appl. 749/18.)

ACID-RESISTING cement, consisting of an acid-resisting silicious material and an aqueous solution of sodium silicate, is made more quick-setting by adding about 1.5% of lead carbonate or basic lead carbonate.—W. F. F.

*Gypsum; Process of burning* — E. Fischer, Pilsen, Bohemia. Ger. Pat. 307,808, 1.1.18. Int. Conv., 15.11.15.

PLASTER of Paris of uniform character is produced from gypsum of varying density by burning the latter in pieces the size of which is in inverse ratio to their density.—J. H. L.

*Cement; Processes of producing — and recovering potassium compounds.* F. W. Huber and F. F. Reath, Riverside, Cal., U.S.A. Eng. Pat. 110,540, 27.8.17. (Appl. 12,289/17.) Int. Conv., 26.9.16.

See U.S. Pat. 1,219,315 of 1917; this J., 1917, 458.

*Artificial asphalts.* Ger. Pat. 307,961. See IIa.

*Carbon blocks.* Eng. Pat. 120,255. See VIII.

## X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

*Cementite transformation; Study of — and of the equilibrium diagram of the iron-carbon system by means of electric resistance measurement.* I. Iitaka. Sci. Rep. Tôhokô Imp. Univ., 1918, 7, 167—175.

By a highly sensitive arrangement of apparatus the author was able to detect the cementite conversion in steel by means of the change in electrical resistance, and also to plot a line where, during cooling, cementite begins to separate from austenite, or during heating, the dissolution of free cementite in ferrite terminates (the so-called SE line). The test specimen, in the form of a cylindrical rod 20 cm. long and 5 mm. diam., enclosed in an evacuated silica tube, formed part of a circuit carrying a constant current of 5 amperes, and a differential galvanometer, connected through two resistances, indicated any difference of potential between two points on the rod 8 cm. apart. The silica tube was heated in a resistance furnace, and the resistances in circuit with the galvanometer were so adjusted that a zero reading was obtained when a current of 5 amperes was passing through the specimen. As the temperature was raised, the deflection of the galvanometer gradually increased with a rapid alteration at the transition point. The mean values for the  $A_0$  point obtained with steels containing 0.31 to 1.50% C were 205° C. ( $Ac_0$ ) and 197° C. ( $Ar_0$ ), which are a little lower than when determined magnetically (215° C.). In a similar manner abnormal changes in resistance were observed during the  $A_1$ ,  $A_2$ , and  $A_3$  transformations, and a curve, ascending rapidly from the eutectoid point, with a curvature of downward concavity, was obtained as the solubility line (SE) of cementite in ferrite.—C. A. K.

*Gold bullion; Refining — with chlorine gas and air.* R. R. Kahan. Bull. 170, Inst. Min. Met., Nov., 1918. [Advance copy.] 4 pages.

In the chlorine process of refining, the greater part of the gold loss occurs in the final stages, and, as metal containing lead is too brittle for coinage purposes, the thorough chlorine treatment necessary for the complete removal of the lead causes a large loss of gold. It is desirable that the last of the base metals be removed while there is still considerable silver in the bullion. The following method, besides being shorter, resulted in a distinct reduction of the gold loss. Two clay pipe stems were introduced into the molten metal, for the delivery of air and chlorine respectively. At the beginning of the operation a maximum amount of air and a very slow stream of chlorine were used. The volume of air

was then diminished and that of the chlorine increased; finally the air pipe was removed. It was found an advantage in this process to mix brittle bullion with the most silvery metal available.

—W. R. S.

*Alloys high in zinc.* L. Guillet and V. Bernard. Rev. Mét., 1918, 15, 407—425.

The properties of alloys of zinc containing up to 8% Al and 8% Cu have been examined by mechanical and microscopical methods. The cast alloys do not present any interesting features in the mechanical tests; a breaking strain of 17 kilos. is attained when 8% Cu is present. A rolled bar containing 3% Al has a breaking strain of 23 kilos., with an extension of 15%, and corresponding figures for an alloy containing 2% Al and 3.6% Cu are 32 kilos. and 5%. When drawn down the alloys generally show a higher breaking strain and a greatly increased elongation. An alloy possessing somewhat similar mechanical properties to brass had the composition Zn (Pb 1%) 97.5 to 98%, Cu 1.5 to 2.0%. The elasticity, however, was much less than that of brass. The micrographic structure of zinc-aluminium alloys shows that two solid solutions are formed, one containing 0—4% Al, and the second 50—100% Al. These form a eutectic containing 5% Al (Shepherd, this J., 1905, 972), though it has been observed in an alloy containing only 1% Al, evidently due to heterogeneity of the metal. In the zinc-copper series there is a solid solution ' $\eta$ ,' with 2.5% Cu, and where the copper present is between 2.5% and 13% a further solid solution ' $\epsilon$ ' forms, increasing with increase in the copper content. The latter solid solution gives rise to brittleness, and its formation should be restricted. A complete examination of the zinc-aluminium-copper system has not been made, but it appears that the solid solutions of zinc with aluminium, and of zinc with copper, are formed independently, and that the properties of the alloys correspond with the separate properties of these solutions. Macrographic examination shows large elongated crystals in the cast alloys, but the crystal structure is not so apparent in rolled or drawn metal. Heterogeneity is generally revealed, and is not influenced by annealing. The hardness of the alloys diminishes with increasing temperature, and the best temperatures for forging or working is given as 125° C. to 130° C.—C. A. K.

*Metals; Occlusion of gases in —.* A. W. Porter. Faraday Soc., Nov. 12, 1918. [Advance proof.] 6 pages.

A REVIEW of the available data on the occlusion of gases in metals. The term "occlusion" is used to cover a number of different phenomena, which may be classified under the following heads: (1) Chemical combination of gas with metal, *e.g.*, hydrogen with sodium, potassium, or palladium; (2) solid solution in conjunction with chemical combination, as in the occlusion of hydrogen by lithium; (3) solid solution, *e.g.*, hydrogen in palladium; (4) solution accompanied by surface adsorption; (5) surface condensation under molecular forces, unaccompanied by solution; (6) inclusion, as when bubbles become entangled in the molten viscous metal. The term adsorption should probably be restricted to cases in which true solution takes place, but in which there is a modification of the concentration in the surface film, due to the influence of the dissolved body upon the surface tension. This phenomenon is quite distinct from surface condensation without solution. The Volta effect has often been attributed to condensed gases on the surface of the metals, but Langmuir (Trans. Amer. Electrochem. Soc., 1916, 29, 129) has shown that contact potentials of large magnitude exist between pure metals, even in a practically perfect vacuum.



*Metals; Properties of — as affected by their occluded gases.* C. Johns. Faraday Soc., Nov. 12, 1918. [Advance proof.] 4 pages.

REFERENCE is made to the widely differing phenomena which have been included under the term "occlusion of gases in metals," and it is pointed out that the properties of metals used for constructional purposes may be affected by their gaseous constituents more profoundly than is generally admitted. All the more complete investigations of metals used for industrial purposes have been made on specimens containing gaseous constituents, and there is no knowledge of the properties which the pure metals or alloys would possess if they could be produced.

*Scrap metals and dross, etc.; Analysis [sampling] of —. Analysis of cement copper.* O Binder. Chem.-Zeit., 1918, 42, 546.

SPECIMENS of scrap metals or dross, etc., usually contain large pieces of metal more or less coated with oxide, and it is impossible to obtain a fair average sample for analysis; it is recommended that, after the finer portions of the sample have been separated by a sieve, the larger pieces should be melted together and then filed so as to obtain a portion for weighing. In the determination of chlorine in cement copper, solution of the sample in nitric acid brings about loss of chlorine, and the copper is not dissolved completely.—W. P. S.

*Flotation; Development of galena — at the Central Mine, Broken Hill.* R. J. Harvey. Bull. 170, Inst. Min. Met., Nov., 1918. [Advance copy.] 17 pages.

THE ore is crushed to  $\frac{1}{2}$ -in. mesh and jigged. The tailings are ground to 40-mesh size, and classified into slimes and "roughs." The latter product is treated with a mixture of coal tar and eucalyptus oil (2 oz. per ton) in "lead cascades," consisting of a series of 9 boxes (36 in. long, 18 in. wide, 28 in. high) set one above the other. The pulp, in descending from one box to another, passes through three nozzles and draws in the required amount of air. Two units of 9 boxes are in use. The slimes from the re-grinding are thickened and floated together with the "roughs." The successful application of this flotation process has resulted in the discarding of 24 tables and 30 vanners, and an increase of 8% in the effective lead recovery, i.e., to 85.6%. The silver recovery increased from 49.2 to 64.4%.

—W. R. S.

*Tin ores; Effect of heating, and heating and quenching Cornish — before crushing.* A. Yates. Bull. 170, Inst. Min. Met., Nov., 1918. [Advance copy.] 3 pages.

THE investigation was made in order to ascertain whether heating, and heating and quenching the ores before crushing, assisted the operation of crushing, diminished the breaking up of coarse particles of cassiterite and the production of slime, and facilitated the liberation of cassiterite grains from the gangue. It was found that the ores after heat treatment were more easily crushed, and contained a smaller proportion of the very fine slime from which very little tin is recoverable; but an improved recovery of tin was not obtained except in one case.—W. R. S.

*Lime-alumina-silica; The system — and its relation to blast-furnace slags and Portland cement.* B. Neumann. Stahl u. Eisen, 1918, 38, 953–960.

THE author gives a brief review of the present state of knowledge in regard to the ternary system

$\text{CaO-Al}_2\text{O}_3\text{-SiO}_2$ , and its relation to blast-furnace slags and Portland cement, based principally on the results of recent investigations at the Carnegie Institution, Washington (this J., 1911, 543; 1915, 139). Blast-furnace slags and Portland cements occupy only small regions in the triangular diagram representing the complete ternary system. Johnson's work on blast-furnace slags (Met. and Chem. Eng., 1916, 363) is referred to, and the differences obtaining in blast-furnaces using charcoal and coke respectively are pointed out. In furnaces heated by coke, the sulphur in the latter necessitates the use of more lime ( $\text{CaO} : \text{SiO}_2 = 1.5 : 1$ ) as flux, and a corresponding rise of about  $200^\circ\text{C}$ . in the flowing temperature of the slags. Calcareous slags are of low viscosity compared with the viscous, tough silicious slags. The temperature at which calcareous slags flow freely is only slightly above their softening point; silicious slags require a difference in temperature of  $200^\circ\text{--}300^\circ\text{C}$ . (compare Fulton, this J., 1913, 89). If the silica content is increased to 37% ( $\text{CaO} : \text{SiO}_2 = 1.35 : 1$ ) the slag flows readily at a lower temperature, but the metal is not desulphurised; if the silica is reduced below 32%, both the furnace temperature and the amount of slag must be increased. Thus increasing the proportion of lime beyond a certain limit does not further improve the desulphurisation, the increased basicity being more than counterbalanced by the greater "stiffness" of the slag. Portland cements of good quality are comprised within the lines in the triangular diagram representing  $3\text{CaO}, \text{SiO}_2$ ,  $3\text{CaO}, \text{Al}_2\text{O}_3$ , and  $2\text{CaO}, \text{SiO}_2$ . Portland cement made from pure materials and burned at a sufficiently high temperature would consist solely of these three substances. In commercial cements, the natural impurities in the materials result in free lime or free  $5\text{CaO}, 3\text{Al}_2\text{O}_3$  being present, together with small quantities of magnesia, iron oxide, etc. As no ternary compound is present in the area of the triangular diagram occupied by cements, the theory of Jänecke (this J., 1911, 1453) and others that Portland cements consist chiefly of alite ( $\text{SCaO}, \text{Al}_2\text{O}_3, 2\text{SiO}_2$ ) and other ternary compounds is untenable. Rankin's work on Portland cement (this J., 1916, 842) is reviewed and the following summary of the compounds formed in the production of Portland cement is given:—

Cement	Composition of raw materials	%	Burning temp.	Constituents in finished cement
Chemically pure cement	$\text{CaO}$	63.4	$1630^\circ\text{C}$ .	$2\text{CaO}, \text{SiO}_2$
	$\text{Al}_2\text{O}_3$	8.0		$3\text{CaO}, \text{SiO}_2$
	$\text{SiO}_2$	23.6		$3\text{CaO}, \text{Al}_2\text{O}_3$
White cement	$\text{CaO}$	66.2	$1525^\circ\text{C}$ .	$2\text{CaO}, \text{SiO}_2$
	$\text{Al}_2\text{O}_3$	6.4		$3\text{CaO}, \text{SiO}_2$
	$\text{SiO}_2$	25.0		$3\text{CaO}, \text{Al}_2\text{O}_3$
	$\text{MgO}, \text{Fe}_2\text{O}_3, \text{Na}_2\text{O}, \text{K}_2\text{O}$	2.4		Trace $\text{CaO}$
Commercial (grey) cement	$\text{CaO}$	63.2	$1425^\circ\text{C}$ .	$2\text{CaO}, \text{SiO}_2$
	$\text{Al}_2\text{O}_3$	7.7		$3\text{CaO}, \text{SiO}_2$
	$\text{SiO}_2$	22.4		$3\text{CaO}, \text{Al}_2\text{O}_3$
	$\text{MgO}, \text{Fe}_2\text{O}_3, \text{Na}_2\text{O}, \text{K}_2\text{O}, \text{SiO}_2$	6.7		Trace $\text{CaO}, \text{CaO}, 3\text{Al}_2\text{O}_3$ and ferrite

If the clinker is insufficiently burned, there will be a deficiency of  $3\text{CaO}, \text{SiO}_2$ . The tricalcium silicate appears to be the most valuable constituent of the cement; the dicalcium silicate hardens too slowly and the aluminate too rapidly. As the temperature of formation of tricalcium silicate is about  $1700^\circ\text{C}$ ., it is desirable to find some flux by means of which it can be produced at a lower temperature; ordinarily, calcium aluminate appears to have this effect. Rankin has suggested that the substitution of iron oxide for part of the alumina may lead to important industrial developments, and the author suggests that an investigation of the properties of the iron Portland cements is also desirable.

—A. B. S.

*Electric resistance; Variation of* — during the fusion of metals. H. Tsutsumi. Sci. Rep. Tôhokô Imp. Univ., 1918, 7, 93—105.

THE electrical resistance of a column of metal contained in a silica tube of small diameter was measured, at various temperatures, by a potentiometer method, the metal being protected from oxidation. Mercury, tin, lead, zinc, aluminium, silver, and copper show a considerable increase in electrical resistance during fusion, the ratio of the specific resistances of the metals in the liquid and solid states at the melting point being approximately equal to 2. Bismuth and antimony show a contrary effect, and a similar ratio for these metals is roughly  $\frac{1}{2}$ , which is attributed to a transformation in the molecular state during fusion.—C. A. K.

*Chromium; Passivity of* —. A. H. W. Aten. Proc. K. Akad. Wetensch. Amsterdam, 1918, 21, 138—150.

A DISCUSSION of the reasons for the activation of passive chromium by cathodic polarisation.

—J. F. S.

*Silver; Colloidal* —. A. Pickles. Chem. News, 1918, 117, 358.

COLLOIDAL solutions of silver are produced by adding 60% solutions of formaldehyde to silver oxide in water at 35° C. The solutions are very stable, and vary in colour from pale lilac to rich ruby red. Acetaldehyde has no action on silver oxide. The colour is discharged slowly by solutions of salts and by nitric acid. Hydrogen peroxide is slowly decomposed by the solution.—J. F. S.

*Electrodeposition of metals.* François. See XXIII.

#### PATENTS.

*Steel-making furnaces, or mixers and the like.* Wellman, Seaver, and Head, Ltd., London, and F. G. Smith, Purley. Eng. Pat. 120,114, 12.11.17. (Appl. 16,585/17.)

IN order to connect or seal various parts of the plant, such as the furnace and regenerator ports, the trough of the seal surrounding the lower port is provided with an independent inner trough, which contains a liquid into which enters a projection surrounding the upper port. The inner ring may be raised or lowered by mechanical means, or by causing it to float, by means of an enclosed air space, on a liquid in the outer trough, from which the liquid may be run off or admitted as required. Suitable guides for the inner ring are provided.

—C. A. K.

*Iron and steel; Method of preparing pickled — for painting.* J. H. Gravell, New York. U.S. Pat. 1,279,101, 17.9.18. Appl., 30.4.18.

SUBSEQUENT to the customary pickling of the metal in sulphuric acid, rapid rusting is prevented by removing the surface metal by a solution of phosphoric acid containing calcium phosphate.—C. A. K.

*Metals; Cleaning* —. J. H. Gravell, New York. Eng. Pat. 119,618, 22.2.18. (Appl. 5113/18.)

SEE U.S. Pat. 1,279,101 of 1918; preceding. An alcoholic solution of phosphoric acid containing calcium phosphate is used.

*Iron and steel; Pickling* —. J. H. Gravell, New York. U.S. Pat. 1,279,331, 17.9.18. Appl., 11.5.18.

THE metal is subjected to the action of a mixture of sulphuric and phosphoric acids, then to the action of an aqueous solution of a blebromate.—T. H. B.

*Iron alloy.* H. K. Sandell, Assignor to H. S. Mills, Chicago, Ill. U.S. Pat. 1,279,448, 17.9.18. Appl., 18.6.17.

AN alloy of 45 parts by weight of Ni, 50 parts Cu, 100 parts Fe, and a relatively small proportion of Mn.—T. H. B.

*[Iron] pipes; Heat treatment for centrifugally cast* —. D. S. de Lavaud, New York. U.S. Pat. 1,280,418, 1.10.18. Appl., 30.7.17.

MOLTEN iron is fed into a rotary metal mould, and the mould rotated to compress the molten metal against its sides by centrifugal force, while congealing the metal within six seconds. The casting is withdrawn as quickly as possible, heated to at least 1800° F. (980° C.), and then allowed to cool. The graphite is very fine on the outside of the casting, increasing in coarseness towards the centre, and the outer portion is easily worked by any tool. The structure of the casting comprises dendrites and grains of silicoferrite and grains of ferrite containing fine graphite surrounded by a network of pearlite.—T. H. B.

*Ores; Process and apparatus for treating — in the blast furnace.* L. P. Basset, Paris. Eng. Pat. 109,452, 7.9.17. Int. Conv., 7.9.16.

THE carbon used as fuel is injected in the form of dust into the lower part of the blast furnace, air being admitted in such proportion as to generate carbon monoxide only, the fraction of the carbon escaping combustion acting as a reducing agent. Wrought iron, cast iron, or steel can be produced as desired by varying the proportions of air and carbon injected, and carbon in coarser form may be added to the ore in suitable proportion to effect a direct reducing action.—T. H. B.

*Furnaces for annealing, hardening, or heat treatment of metals, and the like.* A. Smallwood, London. Eng. Pat. 120,266, 5.11.17. (Appl. 16,159/17.)

A FURNACE has an elongated heating chamber, and in allment with this is a charging chamber of similar size, which extends downwards into a wider chamber capable of being filled with water so as to seal the charging and heating chambers. A trolley car in this chamber, but outside the wall of the charging chamber, is loaded with material for heat treatment, and is moved laterally on rails beneath the charging chamber, and elevated into it above the water level. Charging mechanism then conveys the material from the trolley car longitudinally into the heating chamber.—C. A. K.

*Billet-heating and like furnaces.* A. Crowcroft and F. Morris, London. Eng. Pat. 120,348, 15.5.18. (Appl. 8137/18.)

IN a billet-heating furnace of the continuous type, heated by two fires on opposite sides of the furnace, the fire-holes are set not directly opposite one another, the gases of combustion thereby causing a circulatory motion over the centre of the hearth before their discharge through ports on either side of the combustion chambers. Air, or a mixture of air and steam, is blown through the fire-grates, and a secondary supply of preheated air is supplied above the fires.—C. A. K.

*Metal-melting crucibles; Heating device for* —. G. L. Fort, Assignor to Fort-ified Manufacturing Co., Kansas City, Mo. U.S. Pat. 1,279,682, 24.9.18. Appl., 18.7.17.

A CRUCIBLE provided with a delivery spout is in contact on its under side and along the delivery spout with an electrical heating unit. The whole is sur-



rounded by an insulated metal casing having an opening in the base, through which the heating element may be removed or inserted.—C. A. K.

*Heating furnace.* C. W. Lummis, Assignor to Morgan Construction Co., Worcester, Mass. U.S. Pat. 1,279,889, 24.9.18. Appl., 27.4.14.

A CONTINUOUS billet-heating furnace is divided into a number of narrow compartments by means of transverse partitions carried from the crown of the furnace, sufficient space being allowed above the hearth to enable the billets to be moved longitudinally along the furnace. Products of combustion travel parallel to the partitions.—C. A. K.

*Antimonial and arsenical gold ores; Treatment of* —. J. Penhale, Johannesburg, and W. H. Treloar, Langlaagte, Transvaal. Eng. Pat. 120,287, 3.12.17. (Appl. 17.884/17.)

GOLD ores containing antimony sulphide and/or arsenic sulphide are crushed and slimed in an aqueous solution of an alkali sulphide; after separation of the liquid, the remaining ore is ready for cyaniding, or other extraction process; the base metals may be recovered by acidifying the alkali sulphide solution.—T. H. B.

*Copper ores; Process of treating* — by lixiviation. N. C. Christensen, Salt Lake City, Utah, Assignor to J. E. Barlow, Habana, Cuba, and Big Indian Copper Co. U.S. Pat. 1,278,854, 17.9.18. Appl., 1.9.16.

COPPER ores are lixiviated with a concentrated solution of sulphur dioxide made by first bringing the solution in contact with gases relatively poor in sulphur dioxide and then successively in separate absorbers, with gases relatively stronger in sulphur dioxide. A lime compound may be added to the solution during the lixiviation in order that all of the copper may be recovered as copper sulphite.

—C. A. K.

*Concentrating ores by flotation; Method and apparatus for* —. C. C. Thomas, Baltimore, Md. U.S. Pat. 1,279,040, 17.9.18. Appl., 28.6.16.

ORE pulp is agitated by means of heated products of combustion, such as the exhaust gases from an internal combustion engine. The gases are discharged into the flotation cell through an atomising device.—C. A. K.

*Drying mechanism for electro-plating apparatus.* M. F. Davoran, Assignor to The Crane and Breed Manufacturing Co., Cincinnati, Ohio. U.S. Pat. 1,279,086, 17.9.18. Appl., 3.8.17.

A HORIZONTAL drying chamber is provided at one end with means for discharging a forced blast of air away from the chamber in a direction inclined to its axis. Air passes through the chamber towards the air blast, and is deflected at several points in the chamber into contact with heating devices. The articles to be dried are conveyed past the air blast and through the chamber.—W. F. F.

*Manganese from lean ores; Process of extracting* —. *Process of producing manganese sulphate.*

(A) E. W. Haslup, Bronxville, N.Y., and B. A. Peacock, Philadelphia, (B) E. W. Haslup, Assignor to Haslup and Peacock, Inc., New York. U.S. Pats. (A) 1,279,108 and (B) 1,279,110, 17.9.18. Appls., 7.11.17 and 2.2.18.

(A) A MIXTURE of the ore and a material capable of producing an exothermic reaction in the process, e.g., a potassium compound, is finely ground and

digested with sulphuric acid of not more than 55° B. (sp. gr. 1.616) in a closed heat-insulated vessel for not less than 15 hours. A temperature of 150° C. is attained by the heat of reaction and the sulphates formed are lixiviated with water and the metal values recovered. (B) Low-grade material containing manganese oxide is finely divided and digested in a solution of a metal sulphate capable of attacking the manganese oxide present. Ferrous sulphate or aluminium sulphate, derived for example from "greensand," may be employed at a reaction temperature of 60° C.—C. A. K.

*Ores; Apparatus for reducing* —. H. N. Tracy, Los Angeles, Cal. U.S. Pats. (A) 1,279,180 and (B) 1,279,181, 17.9.18. Appls., 11.1 and 1.3.17.

(A) THE ore is continuously heated in a tall and narrow vertical stack, and a gaseous reducing agent is continuously introduced near the middle of the column of material. The reduced ore is withdrawn at the bottom, and charging and withdrawal are so arranged as to keep the height of the column of ore constant. (B) The ore is continuously cooled at the bottom of the column, and means are provided for transferring heat from the cooling ore to fresh ore which is thus preheated prior to its introduction into the top of the stack.—T. H. B.

*Zinc; Apparatus for treating* —. C. A. H. de Saulles, New York. U.S. Pat. 1,279,292, 17.9.18. Appl., 10.7.17.

PROVISION is made for the transference of molten zinc from a melting furnace, in which separation of lead takes place by gravitation, to a distillation plant in the condensing part of which liquid zinc is produced. The molten zinc is discharged from the condensers into a mixing furnace, from which it may be returned to the distilling furnace.—C. A. K.

*Metals; Case-hardening of* —. P. W. Shimer, Easton, Pa. U.S. Pats. (A) 1,279,457 and (B) 1,279,458, 17.9.18. Appl., 3.1.18.

(A) THE metal is immersed in a bath of fused salts in which calcium cyanamide has previously been immersed until a vigorous evolution of gas has occurred and then removed. (B) A mixture of calcium cyanamide and hard pitch or other coking binder is coked, and the residual mass is broken up into fragments of suitable size for use in case-hardening.—T. H. B.

*Electrolytic process [for impure copper solutions].* W. E. Greenawalt, Denver, Colo. U.S. Pat. 1,279,860, 24.9.18. Appl., 24.2.14. Renewed 26.2.18.

A PORTION of the copper solution, containing salts of iron, is separated from the electrodes, and a finely-divided sulphide reducing agent, e.g., hydrogen sulphide in an atomised form, is agitated in contact with the separated electrolyte. The iron salts are reduced to the ferrous condition, the insoluble matter filtered off, and the filtered electrolyte returned to the electrodes.—B. N.

*Electroplating; Method of and apparatus for* —. C. A. Hach, Oak Park, Ill., Assignor to Western Electric Co., Inc., New York. U.S. Pat. 1,280,213, 1.10.18. Appl., 12.1.17.

A CONSTANT stream of gas is delivered through an inert porous material, forming bubbles which remove the hydrogen bubbles generated on the article connected to the cathode during the plating operation.—B. N.

*Plating; Method of and apparatus for [electro] —.* G. A. Landry, Chicago, Ill., Assignor to Western Electric Co., Inc., New York. U.S. Pat. 1,280,249, 1.10.18. Appl. 15.1.17.

THE cathode is completely enclosed in a perforated cup-shaped anode, the interior contour of which is shaped to conform substantially to the shape of the cathode, and the latter is rotated so as to agitate the solution in the anode.—B. N.

*Ores; Method of sintering —.* E. J. Hellman, Perrysburg, Ohio. U.S. Pat. 1,280,221, 1.10.18. Appl., 3.1.18.

A LAYER of the ore is spread on a moving surface, the upper surface of the ore ignited, a second layer then spread over the first one, and its upper surface ignited, air being drawn downwards through both layers simultaneously to promote combustion.

—T. H. B.

*Metal article; Corrosion-resisting — and method of making same.* W. J. Merten, Pittsburgh, Pa., Assignor to G. D. Breck, Cleveland, Ohio. U.S. Pat. 1,280,268, 1.10.18. Appl., 4.1.18.

THE opposite sides of metal articles which are subjected to different corroding influences are treated simultaneously by different corrosion-inhibiting processes, which require for their performance the same elevated temperature. For example, in the case of iron tubes or the like, the opposite sides of which are subjected to heat and moisture respectively, as in steam generating apparatus, the side which is to be subjected to moisture is sherardised and the opposite side is simultaneously coated with magnetic oxide.—T. H. B.

*Ores; Process of roasting —.* A. B. Newman, Langeloth, Pa., Assignor to Metallurgical Co. of America, New York. U.S. Pat. 1,280,283, 1.10.18. Appl., 23.3.16.

IN roasting ores in a multiple hearth furnace, the roaster gases are enriched in sulphur dioxide, by burning sulphur in the lower levels of the furnace with insufficient air for complete combustion, and subsequently introducing further amounts of air to complete the combustion, whereby heat is supplied directly to the ore bed and the furnace gases are enriched in sulphur dioxide.—T. H. B.

*Coating metal; Processes for —.* B. P. Allen, Great Neck Station, N.Y., U.S.A. Eng. Pat. 113,602, 31.10.17. (Appl. 15,867/17.) Int. Conv., 19.2.17.

SEE U.S. Pat. 1,233,633 of 1917; this J., 1917, 1015.

*Zinc; Furnace for treating —.* L. Van Gulek, Swansea. U.S. Pat. 1,280,593, 1.10.18. Appl., 21.7.17.

SEE Eng. Pat. 113,393 of 1917; this J., 1918, 185 A.

*Metal [tungsten wire]; Process of treating —.* A. Pacz, Cleveland, Ohio, Assignor to General Electric Co. U.S. Pat. 1,280,825, 8.10.18. Appl., 13.4.14. Renewed 18.5.18.

SEE Eng. Pat. 13,549 of 1914; this J., 1915, 803.

*Solder or the like and process for preparing the same.* A. P. Bevan, Tavistock. U.S. Pat. 1,281,126, 8.10.18. Appl., 9.8.17.

SEE Eng. Pat. 108,916 of 1916; this J., 1917, 1100.

*Sulphuric acid [and zinc].* U.S. Pat. 1,278,308. See VII.

*Resistance alloy.* U.S. Pat. 1,279,252. See XI.

## XI.—ELECTRO-CHEMISTRY.

*Producer gas cell.* Hofmann. See VII.

*Electrodeposition of metals.* François. See XXIII.

### PATENTS.

*Furnace; Electric —.* S. Peacock, Philadelphia, Pa., Assignor to Haslup and Peacock, Inc., New York. U.S. Pat. 1,279,146, 17.9.18. Appl., 1.3.18.

THE furnace comprises a water-jacketed tube, open at the ends, within which is a hollow resistor surrounded by a heat-shield. Detachable end pieces connected to flanges on the tube carry holders for the resistor and form open chambers communicating with the interior of the resistor, and provided with readily-detachable end pieces to seal them, and with means for exhausting the air from the chambers and the resistor.—B. N.

*Furnace; Three-phase electric — and method of operating the same.* F. T. Snyder, Oak Park, Ill. U.S. Pat. 1,279,928, 24.9.18. Appl., 11.3.18.

THE furnace is provided with two upper electrodes, a bottom contact, and a source of polyphase current for the formation of two arcs between the electrodes and the bath. A reactance is provided in the path of lowest resistance, of such an amount as automatically to equalise the currents flowing in the different phases of the system.—B. N.

*Electrode.* F. G. Wheeler, Assignor to Bleach Process Co., Appleton, Wis. U.S. Pat. 1,279,192, 17.9.18. Appl., 21.7.16.

A METALLIC terminal, in the form of a bolt, extends into a screw-threaded recess in the carbon portion of the electrode, and fusible metal is introduced into the space between the bolt and the carbon, the portion of the carbon adjacent to the metal being rendered impervious to the electrolyte or its decomposition products by impregnation with a mixture of asphaltum and a glyceride. The portion of the terminal projecting from the carbon is encircled by a gasket, which lies between the carbon body of the electrode and the internal face of the cell cover, and contact between the gasket and the carbon body to form a liquid-tight joint is secured by means of a nut screwing on to the end of the terminal.—B. N.

*Resistance element.* J. A. Capp, Schenectady, N.Y., Assignor to General Electric Co. U.S. Pat. 1,279,252, 17.9.18. Appl., 18.4.16.

A HIGH-RESISTANCE core wire of an alloy of iron (55%), nickel (35%), and manganese (10%) is enclosed within a tubular metallic sheath, with an intervening layer of compact, powdered, insulating material in which the resistance element is embedded.—B. N.

*Battery cell; Secondary —.* H. Csanyi, New York. U.S. Pat. 1,279,280, 17.9.18. Appl., 27.2.17.

A SECONDARY battery comprises a positive electrode, a container forming the negative electrode, an electrolyte of ammonium and zinc chlorides, and a depolariser of ammonium chloride, graphite, and red lead. On charging the battery, zinc is deposited



at the negative electrode and chlorine liberated at the positive, the chlorine forming lead chloride and peroxide with the red lead. The electrical discharge of the cell produces chemical actions which restore the electrolyte and depolariser to the conditions existing prior to charging.—B. N.

*Battery; Storage* —. F. Wright, Assignor to Wright Storage Battery Co., Poughkeepsie, N.Y. U.S. Pat. 1,279,508, 24.9.18. Appl., 9.1.14. Renewed 6.2.18.

THE grid of a lead storage battery plate is pasted with a mixture of equal parts by weight of "battery lead" and red lead, moistened with dilute sulphuric acid. The pasted plates are dried at the ordinary temperature, and, before being "formed," are painted over lightly with a small quantity of concentrated sulphuric acid, to make the active material denser and harder and to facilitate the "forming" treatment.—B. N.

*Electric furnace*. C. H. vom Baur, Douglaston, N.Y., U.S.A. Eng. Pat. 120,444, 10.11.17. (Appl. 16,507/17.)

SEE U.S. Pat. 1,252,633 of 1918; this J., 1918, 156 A.

*Electric furnaces; Electrode-holder for* —. S. Steinberg and I. Gramolin, Motowilichi, Russia. U.S. Pat. 1,280,576, 1.10.18. Appl., 19.7.16.

SEE Eng. Pat. 111,679 of 1916; this J., 1918, 61 A.

*Treating gases*. U.S. Pat. 1,280,471. See I.

*Perchlorates*. U.S. Pat. 1,279,593. See VII.

*Carbon blocks*. Eng. Pat. 120,255. See VIII.

*Electrolytic process*. U.S. Pat. 1,279,860. See X.

## XII.—FATS; OILS; WAXES.

*Palm oils; Rapid hydrolysis of* —. Balland. Comptes rend., 1918, 167, 673—674.

THREE samples of palm oil, representing consignments imported into France as food for the Colonial troops, were examined after 4, 3, and 2 months, and found to contain respectively 61.2, 19.4, and 8.3% of free fatty acids, as oleic acid. On keeping for still longer periods the hydrolysis proceeded until the free fatty acids reached 75%.—C. A. M.

### PATENTS.

*Oils from seeds, beans, nuts, offal, or the like; Apparatus for extracting* —. A. McC. Winters. London. Eng. Pat. 120,155, 24.1.18. (Appl. 1412/18.)

THE apparatus consists of several (e.g., three) horizontal extracting chambers connected together in series one above the other. The uppermost chamber is connected with a charging hopper. Each extraction chamber is provided internally with a screw conveyor to lead the material to one end where it falls by gravity to the chamber below, the extract being separated from the material by passage through a screen acting as a false bottom in the lower half of each chamber. Solvent recovered continuously from the oil-bearing extract draining through the screen in each of the three chambers is supplied to the first (upper) chamber, and solvent

similarly recovered from the extracted residue is supplied to the second (intermediate) extracting chamber. The solvent is supplied either through perforations in the shaft of the screw conveyor or preferably through perforations in the upper portion of the extracting chamber, in this case the upper half of the chamber being fitted with a jacket. To recover the solvent, the extracts draining from the chambers are united and allowed to fall by gravity over oppositely inclined baffles and grids or sieves in a chamber into which steam enters at the bottom, the steam and solvent vapour being condensed and the water separated in an apparatus, the upper outlet of which (for recovered solvent) is above the level of entry into the first extraction chamber. The extracted material is delivered by the screw conveyor in the last and lowest of the extracting chambers into a similar solvent-recovery vessel, the oppositely-inclined baffles of which consist of sieves. The volatilised solvent from this latter vessel is condensed and freed from water in apparatus of which the outlet is above the level of entry into the second extraction chamber.

—A. de W.

*Hydrogenated-oil compositions. Product containing hydrogenated oil*. C. Ellis, Montclair, N.J. U.S. Pats. (A) 1,276,507, (B) 1,276,508, and (C) 1,276,509, 20.8.18. Appls., 6.1.13 (renewed 1.8.16), 21.4.17, and 10.4.18.

(A) A MIXTURE of a hydrogenated animal, fish, or vegetable oil having a "titre" not materially less than 59 and an iodine value not exceeding 25, with carnauba wax or paraffin wax is used for making phonograph records. (B) A mixture of hydrogenated fatty oil (e.g., cottonseed oil) with a non-abrasive mineral substance (e.g., talc, barytes, clay) is used for making phonograph records. (C) Mixtures of hydrogenated fatty oil with non-abrasive powders are used as lubricants, toilet powders, etc., and mixtures with abrasive powders as grinding and polishing compositions.

*Hydrogenation; Process of* —. A. Schwareman, Assignor to Kellogg Products, Inc., Buffalo, N.Y. U.S. Pat. 1,280,315, 1.10.18. Appl., 20.8.17.

A VERTICAL shaft carrying a number of hollow perforated arms at different levels is rotated in a mixture of oil and "compound gas-occluding catalyst" at a speed sufficient to induce cavitation, hydrogen being supplied through the arms to the cavities immediately on their production.—A. de W.

*Catalyst*. U.S. Pat. 1,280,314. See I.

*Catalytic substances*. Ger. Pat. 307,380. See VII.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

*Pine resin*. W. Fahrion. Chem. Umschau, 1918, 25, 3—5. Z. angew. Chem., 1918, 31, Ref., 306.

THE resin obtained by the extraction of pine resin residues with caustic soda and acidification of the filtrate is light chocolate-brown in colour, without lustre, friable, and has a faint aromatic herb-like odour. It contains 4% of water and is soluble to the extent of 5.8% in light petroleum spirit, 34.5% in ether, and 55.7% in alcohol. The portion soluble in light petroleum spirit contains, together with neutral substances, some petroleum-soluble derivatives of hydroxyabietic acids. The portions soluble in ether and alcohol are formed by the auto-oxida-

tion of the substances soluble in light petroleum spirit. It is obvious that such a highly oxidised resin cannot in all cases fulfil the functions of normal colophony.—J. F. B.

*Rosin from pine scrapings; Process for the recovery of —.* W. Schaefer. Chem. Umschau, 1918, 25, 25—26. Z. angew. Chem., 1918, 31, Ref., 306.

Pine scrapings were formerly used to a small extent as an addition to brewers' pitch. Rosin and turpentine oil can be recovered from this material by extraction with solvents, but the process presents considerable disadvantages. The new process proposed consists in heating and emulsifying the crude oleo-resin with water and separating the emulsion from the residue by frothing it over. The product which is floated off is passed into a melting vessel and then into a still from which the turpentine oil is distilled off with the water under vacuum.

—J. F. B.

#### PATENTS.

*Paint-vehicle and method of making same.* W. G. Hall, California, Pa. U.S. Pat. 1,279,106, 17.9.18. Appl., 20.9.17.

AN elastic oil suitable as a vehicle for paints is produced by distilling a mixture of about equal parts of "green woods" and crude turpentine. The more volatile constituents are condensed and are mixed while hot with a proportion of the heavier oils remaining in the distillation vessel.

—C. A. K.

*Paint; Method of recovering materials from —.* J. F. Lostumbo and A. H. Olander, Chicago, Ill. U.S. Pat. 1,279,888, 24.9.18. Appl., 11.2.18.

PAINT refuse, or paint which has become more or less dried, is treated with a solvent, and the oil constituent saponified by the addition of alkali. The solid and liquid portions are separated, and after grinding, the pigments may be utilised again for paint making.—C. A. K.

*Paint-dissolver.* H. Catlett, Assignor to Schalk Chemical Co., Los Angeles, Cal. U.S. Pat. 1,280,162, 1.10.18. Appl., 24.4.17.

A PAINT dissolver consists of a mixture of trisodium phosphate, 93.25%, and sodium chloride, 6.75%, with sufficient water to dissolve the two constituents.—A. de W.

*Plastic compositions.* J. W. Aylsworth, East Orange, N.J., Assignor to Condensite Co. of America. Reissues 14,530 and 14,531, 8.10.18, of U.S. Pat. 1,087,422, 17.2.14. Appls., 8.1.16 and 26.8.18.

SEE Eng. Pat. 3498 of 1911; this J., 1912, 347.

*Preparing iron for painting.* U.S. Pat. 1,279,101. See X.

#### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

*Sulphur; Determination of — in vulcanised rubber.* H. P. Stevens. Analyst, 1918, 43, 377—378.

ABOUT 0.5 gm. of the sample is digested with 20 c.c. of nitric acid (sp. gr. 1.42) and 0.5 gm. of potassium chlorate, the liquid then boiled for 2 to 3 hours beneath a reflux condenser, and subsequently evaporated to dryness in a basin, after

the addition of 3 grms. of pure magnesium nitrate. The residue is cautiously heated over a naked flame, the presence of the magnesium salt moderating the combustion, and any unburnt carbon is destroyed by digestion with nitric acid and potassium chlorate, and the excess of acid evaporated. After the addition of 10 c.c. of strong hydrochloric acid, the dish is covered with a clock-glass and gently heated until red fumes cease to appear. The liquid is then diluted, filtered, made up to 300 c.c., and heated to boiling on a hot plate, the sulphuric acid precipitated by the addition of 5 c.c. of 10% barium chloride solution, and the precipitate allowed to stand overnight before filtration.—C. A. M.

*Isoprene monohydrochloride.* Aschan. See XX.

#### PATENT.

*Elastic mass [rubber substitute] and manufacture of the same.* E. S. Ali-Cohen, The Hague, Netherlands. U.S. Pat. 1,280,129, 1.10.18. Appl., 14.4.17. SEE Eng. Pat. 103,824 of 1917; this J., 1917, 1140.

#### XV.—LEATHER; BONE; HORN; GLUE.

*Tannins; Estimation of —.* New methods and their practical value compared with the hide powder process. R. Lauffmann. Collegium, 1918, 185—190.

A CRITICAL review of the new processes not included in Dekker's "Tannins" (1913). In the methods of Zwick, Vanicek, and Eglén the tannin is estimated by determining the refractivity, acidity, and proportion of total solubles to tans respectively of original and detannised solutions, the figures obtained in each case being multiplied by a factor which is assumed constant for each tannin. Gawalowski (this J., 1916, 187) precipitates the tannin with basic copper acetate, and Levi and Ortlmann use a chromium compound as precipitant, a factor being applied to the weight of dry precipitate to obtain tannin content. These conversion factors, however, are not constant for the same material and are useless for application to mixtures of tannins. Kohn-Abrest and Singh and Ghose (this J., 1916, 159) recommend the adoption of inorganic detannisers. These methods seem very arbitrary since the detanniser is so entirely different from hide powder. No materials not in use in the tannery are employed in the hide powder process.—D. W.

*Chromium [in tanning liquors]; Influence of iron and organic matter on the iodometric estimation of —.* R. Lauffmann. Collegium, 1918, 222—228.

THE author confirms the results obtained by Schorlemmer (this J., 1918, 445A) and Lamb and Harvey (this J., 1916, 1028). Hydroxide precipitates separating out after the peroxide oxidation of chrome liquors adsorb some of the chromate even if filtered off and well washed. If they do not liberate iodine from potassium iodide and are soluble in acid, titration is unnecessary as they will dissolve in excess acid. Large amounts of organic matter in a chromium solution lead to inaccurate results by the peroxide oxidation method, since they retard the separation of the iodine and obscure the end point. Organic matter has no influence on estimations in which the chromium is oxidised by fusion.—D. W.



*Tannage; Quinone* —. I. and II. W. Moeller. Collegium, 1918, 71—78, 210—213, 241—242.

ALL compounds of a quinone character are not easily oxidised or reduced. Anthraquinone and its derivatives do not give the ordinary quinone reactions. The quinone characteristics which appear to be missing are present in a latent form. The quinonic or non-quinonic nature of the phlobaphenes cannot be determined by studying their behaviour in ordinary quinone reactions but only by experimental determination of their constitution. Nierenstein, Powarnin, and Koerner have all furnished evidence that tannins have a quinone nature. Active quinonic properties, the tendency to form complex colloids, and tanning properties all seem interdependent. In vegetable tannage the peptiser, a polyphenol, is oxidised; this disturbs the colloid-chemical character of the solution, and peptised phlobaphenes are precipitated along with dark-coloured humins. An intermediate product of this oxidation is always benzoquinone, the quinhydrone of which colours the solution an intense red, to which may be attributed the colour of the phlobaphenes. In the pure benzoquinone tannage the first phase is the oxidation by the quinone of the  $\alpha$ -amino-acids in the interfibrillary tissue to aldehydes. The quinone is thereby converted into quinhydrone (red colour) and further into complex colloidal humins (brown) which are the effective tanning agents. Free quinone can never be detected in quinone-tanned leather.—D. W.

*Leather formation; Theory of* —. V. W. Fahrion. Collegium, 1918, 173—178, 213—214.

A REPLY to Moeller's criticisms on the chemical theory of tannage (Collegium, 1918, 25—40, 61—78, 93—105; J. Soc. Leather Trades Chem., 1918, 188—190). Moeller draws a false analogy between the behaviour of formaldehyde with albumins and with collagen, since the latter is not an albumin but an albuminoid. Water is necessary for Moeller's aldehyde tannage theory; formaldehyde however tans in alcoholic solution. The increased tanning properties of aldehydes containing polymers can be explained by catalysis without the aid of the peptisation theory. Moeller's observation of free aldehyde in pelt after tanning with aldehydes and washing, can be explained by reversible chemical combination or formation of additive compounds. Quinone can tan hide powder by being rubbed together with it in a mortar, without the presence of any solvent. The decomposition products of quinone in aqueous solution are thus proved unnecessary for tanning, hence Moeller's peptisation theory is superfluous and incorrect.

—D. W.

*Leather fibre; Elementary structure of* —. Principles of molecular physics of leather. W. Moeller. Collegium, 1918, 157—173, 202—210, 230—240.

THEORETICAL deductions from ultramicroscopic observations on hide and leather. The fine fibrils of hide tissue consist of parallel chains of collagen micells with their optical axes all arranged in the direction of the chains, hence the property of anisotropism in hide tissue. Like all organised substances hide tissue consists of two chemically different substances, collagen and its hydrolytic decomposition products. The collagen micell is polyhedral and somewhat elongated. Swelling disturbs the arrangement of the micells. A comparison is drawn between anisotropy in crystals and in the hide tissue which leads to the assumption of a lattice-work arrangement of the micellar chains. The astringent effect of tannins is seen in their action on micellar chains which are subjected to torsion as a result. The optical axes are displaced and the anisotropy disappears. Hide fibres under tension cannot be tanned.—D. W.

## PATENTS.

*Tanning hides; Process and apparatus for* —. V. Qnaedvlieg, Keremeos, B.C., Canada. U.S. Pat. 1,280,544, 1.10.18. Appl., 18.9.16.

HIDES are suspended from drums mounted in series in a vat containing tanning solution; the drums can be rotated alternately in opposite directions to secure gradual and uniform bending of the hides, thus opening their pores and ensuring free flow of tanning liquor over their surface.—D. W.

*Tanning animal hides; Process for* —. Badische Anilin und Soda Fabr. Ger. Pat. 305,855, 11.2.15. Addition to 281,484 (see Addition to Fr. Pat. 443,730; this J., 1914, 209).

SUBSTANCES are claimed which fulfil the conditions specified in the principal patent and contain in their molecule two or more aromatic nuclei, some or all of which are different from the others. Tanning agents which are covered by the present addition comprise the condensation products of 2-naphthol-6-sulphonic acid with benzyl alcohol, of 2,5,7-aminonaphtholsulphonic acid with *p*-toluenesulphochloride, and of sodium 2-naphthol-6-sulphonate with *p*-cresol.—J. F. B.

*Leather, yarns, jute or like materials; Machines for drying* —. C. Stephens, Leeds. Eng. Pat. 120,277, 15.11.17. (Appl. 16,779/17.)

By means of a conveyor, the material to be dried is drawn longitudinally through a long horizontal chamber, fitted with two flat sets of steam-heated coils, arranged near the top and bottom of the chamber respectively. Air is blown into the chamber through a tapered inlet duct arranged along the bottom. Baffle-plates direct the air vertically upwards or diagonally, thus ensuring its being continuously heated and reheated as it circulates over the steam coils. An exhaust fan at one end extracts a predetermined proportion of the moist air.—D. W.

## XVI.—SOILS; FERTILISERS.

*Soil acidity methods*. R. E. Stephenson. Soil Sci., 1918, 6, 32—52.

FROM a comparison of the Hopkins, Veitch, Jones, MacIntire, Truog, and Tacke methods of studying soil acidity (this J., 1904, 762; 1915, 1103, 1156; 1916, 699), the author concludes that the Tacke method is the most satisfactory. Pure water is a satisfactory medium for the reaction between the acid soil and the calcium carbonate, although the use of dilute solutions of calcium or sodium chloride hastens the reaction to a limited extent. Concentrated solutions of these chlorides, on the other hand, depress the reaction. The presence of an anti-septic, such as toluene, is not necessary. The most important factors are the duration of the reaction, which should not be less than 5—10 hours; the rate of aeration, which should be maintained at a maximum; the vigour of shaking, which should be thorough. The method is consistent in indicating total acidity, and, in a limited way, also measures toxicity of the soil acids.—W. G.

*Potash residues in Hagerstown silty loam soil; Condition of fertiliser* —. W. Frear and E. S. Erb. J. Agric. Res., 1918, 15, 59—81.

A LOAM soil in Pennsylvania was selected for study, part of which had received 100 lb. of fertiliser potash biennially for 36 years and the other part had been unmanured, but both had been cropped

alike. The condition of the potash present was determined by several solvents, concentrated hydrochloric acid being used for a strong solvent and  $N/5$  acid, distilled water, carbonated water, and ammonium chloride solution for weak solvents. The potash was determined by the platine chloride method. The total potash in the untreated soil was 3.821%. Concentrated acid dissolved a slightly larger proportion of the total potash from the fertilised than from the unfertilised soil, whereas weak solvents dissolved twice as much from the fertilised as from the unfertilised soil. The  $N/5$  acid was the most active of the weak solvents. If a second extraction was made after the first with a weak solvent, the amount of potash dissolved on the second occasion was much less than on the first, but was about equal from the two soils, showing that the potash was more readily available in the fertilised soil. The clayey and non-clayey portions of the soil were also examined separately. The clay contained less potash than the non-clay, and both fractions of the fertilised soil were richer in potash than the corresponding fractions of the unfertilised soil. The crops on the fertilised soil showed little increase over those on the unfertilised soil, owing to the soil being naturally rich in potash. The application of potash, however, caused the amount of potash taken up by the crops to be increased to the extent of 40%. The conclusion reached is that much of the potash applied remained in the surface soil in a highly available condition, but more still in a condition of lower availability; and that the loss by drainage was not great.—J. H. J.

*Legume-infecting bacteria; Report on the examination of commercial cultures of —.* C. R. Fellers. *Soil Sci.*, 1918, 6, 53—67.

SOME thirty official and twenty unofficial samples of cultures were examined. The culture medium for the plate tests was prepared by adding 12 grms. of mannitol, 2 grms. of monopotassium phosphate, 0.2 gm. of magnesium sulphate, 0.2 gm. of sodium chloride, 0.1 gm. of calcium sulphate, and 1 gm. of calcium carbonate to 1000 c.c. of distilled water and 15 grms. of agar. This was found to be very satisfactory for general plating of all varieties of *B. radiclecola*. The purity and general condition of the official samples examined were, generally speaking, very good, only two being classed as "poor" and four as "partly poor." Of the legumes examined, soya beans seemed to be harder to inoculate than most of the common varieties, and for these beans the soil-transfer method of inoculation is recommended. Soil or "muck" cultures are excellent carriers of legume bacteria. The plate method of testing pure cultures gives a good indication of the infecting ability of the organisms, but the results need to be verified by growing the plants themselves and examining the roots for nodules.—W. G.

*Legume inoculation; Tests of commercial cultures for —.* H. A. Noyes and C. O. Cromer. *Soil Sci.*, 1918, 6, 69—79.

IN the case of sweet clover, inoculation by commercial cultures, or by soil taken from a field where the clover had been recently successfully grown, at a dilution such that seed obtained either its exact proportion of the commercial culture, or its proportion of bacteria in the weight of soil used per acre, gave equally successful results. With soya beans, cowpeas, and hairy vetch, satisfactory inoculation was not secured with either commercial cultures or soil.—W. G.

*Seeds; Resistance of — to desiccation.* G. T. Harrington and W. Crocker. *J. Agric. Res.*, 1918, 14, 525—532.

SEEDS of wheat, barley, Sudan grass, Kentucky blue-grass, and Johnson grass were dried under varying conditions and for varying lengths of time and it was found that the percentage germination was not materially changed when the seeds were dried to a moisture content of less than 1%. The percentage germination of the two last-named grasses was not affected when the moisture content was reduced to 0.1%, although the vigour of the blue-grass seedlings was greatly reduced. The vigour of the seedlings was further reduced by drying the blue-grass seeds in a vacuum at 100° C. for six hours, but the percentage of germination was not materially affected.—W. G.

*Drying plant materials.* André. *See* XXIII.

#### PATENT.

*Fertiliser and fertilising.* C. Bosch, Assignor to Badische Anilin und Soda Fabr., Ludwigshafen, Germany. U.S. Pat. 1,280,650, 8.10.18. Appl., 24.11.14.

SEE Ger. Pat. 285,491 of 1914; this J., 1916, 190. Specific claim is made for a fertiliser containing calcium urea phosphate and ammonia.

### XVII.—SUGARS; STARCHES; GUMS.

*Sugars; Valuation of raw — from the point of view of the refiner.* W. D. Horne. *J. Ind. Eng. Chem.*, 1918, 10, 809—812.

THE efforts which have been made during recent years to elaborate a rough method of standardising raw sugars destined for refining, based on the size and hardness of the grain, the amount of insoluble matter, the reaction, and the odour, in addition to the usual data, such as the polarisation and the ash, are based on assumptions frequently not borne out in practice, and overlook important variations of quality radically affecting the value of the product for refining purposes. On account of the defective nature of some of the raw sugars recently imported into the States, the author has examined the behaviour of the raw sugar when subjected to certain tests that imitate as closely as possible the conditions actually obtaining in the refinery. For example, to obtain an indication of its "adhesibility," or the completeness with which the adhering film of molasses can be removed from the crystals, 200 grms. of the sample is mixed into a magma with 92 c.c. of a wash-liquor composed of a saturated solution of the sample itself, made by shaking 100 grms. with 45 c.c. of water for 10 minutes and decanting; this magma is spun in a 5-in. laboratory centrifuge for 2 minutes, and the yield and the polarisation (after drying) of the washed sugar determined. Under these conditions, a good centrifugal raw sugar which during manufacture has not been mixed with "seconds," will polarise about 99.4°, and an ordinary centrifugal sugar, about 99°. If the purity of the washings is below 80°, a deduction from the basic price of the raw sugar may be assessed. Secondly, the behaviour of the raw sugar during defecation and filtration is examined by treating a 45% liquor made from the sample with the minimum amount of acid calcium phosphate and calcium succinate to produce a clear solution, heating to 190° F. (88° C.), and filtering through a folded cloth in a 3 in. perforated brass cone, the time taken to collect 70 c.c. of the filtrate being noted. Sugars filtering freely



in the refinery will require 5 min. or less in this test, those of medium quality, from 5–10 min., poor samples, 10–15 min., and bad "gummy" samples, 15–20 min., or even longer, and, depending upon this rate of filtration, the deduction to be made from the basic price may be fixed. Thirdly, the readiness with which a raw sugar can be decolorised by animal charcoal is found by dissolving 10 grms. in 30 c.c. of water, adding 0.25 gm. of kieselguhr and 2 grms. of good animal charcoal in powder (which all passes through a 60-mesh sieve), heating gently to boiling point, and filtering through paper, colorimetric tests of the filtrate and of a solution submitted to similar treatment in the absence of the animal charcoal affording the means of comparison. A sugar of average quality will lose in this test about 75% of its colour, and according to the result of the test compared with the result obtained with a standard sample, the deduction to be made from the basic price may be ascertained.—J. P. O.

*Cane juice; Role of oxidases and of iron in the colour changes of sugar* — F. W. Zerban. *J. Ind. Eng. Chem.*, 1918, 10, 814–817.

PREVIOUS work has indicated that the colouring matter in the sugar cane is composed of chlorophyll, saccharetin, and in the case of dark varieties also anthocyanin. Of these, the first two are insoluble, passing mechanically into the juice during milling, while the saccharetin during the subsequent treatment with lime is rendered soluble, and develops a yellow colour. Anthocyanin is soluble, and imparts a dark colour to juice expressed from cane containing much of it. C. A. Browne (*Louisiana Bull.*, 91, 9) has pointed out the presence in the cane of so-called "tannins," that is, of aromatic compounds giving the well-known colour reaction with ferric chloride, which may not necessarily be true tannins, but may be any of the numerous polyphenols or phenolcarboxylic acids. M. C. Schneller (*Louisiana Bull.*, 157) has suggested that the greyish tint of some sugars and the abnormally dark colour of the less pure factory products may be due to the action of iron salts on the polyphenols. Experiments now carried out by the author with young cane shoots (containing neither chlorophyll nor anthocyanin, but rich in polyphenols) demonstrate that the extracted juice has a dark brown colour, quite unlike the dirty green of raw mill juice. When, however, ferrous sulphate is added to such juice the usual colour of mill juice is reproduced after a short time, showing this to be due to the contact of the juice with the iron of the milling plant. The brown colour formed in the absence of iron is due to the action of a laccase on the polyphenols, and to a small extent also to that of tyrosinase upon the tyrosine of the cane. The dirty green colour of mill juice is attributed to the interaction of the laccase, the polyphenols, and the ferrous salts formed by the action of the organic acids upon the iron of the rollers, the ferrous salts being rapidly oxidised by the oxidases of the cane to the ferric state.—J. P. O.

*Decolorising carbon; Preparation of an active* — from kelp [for use in sugar refining]. F. W. Zerban and E. C. Freeland. *J. Ind. Eng. Chem.*, 1918, 10, 812–814.

By carbonising kelp rapidly at a comparatively high temperature in such a way that the gases can easily escape, using preferably an open vessel, subsequently heating the product to redness for 2 hours, and afterwards washing it successively with water, hydrochloric acid, and again with water, a preparation more powerful than "Norit" is produced; if the kelp be washed with water before

heating to redness, so as to remove the soluble salts, the results are much less satisfactory. It is suggested that the high nitrogen content of kelp may explain the efficiency of the carbon prepared from it, and it was noticed in every case in which an active product was obtained that Prussian blue was formed on extracting with hydrochloric acid.—J. P. O.

*Sucrose; Inversion of* — by colloidal silicic acid. A. and A. Mary. *Comptes rend.*, 1918, 167, 644–646.

COLLOIDAL silicic acid, produced by interaction of hydrochloric acid and sodium silicate solutions, is capable of inverting sucrose. The hydrolytic activity appears to depend on the degree of dispersion of the colloid, and is destroyed by coagulation. It increases with rise in temperature to a certain point which is probably determined by coagulative changes. Dialysed solutions lose their activity after a period which varies inversely with their concentration. (See also *J. Chem. Soc.*, Jan., 1919.)—J. H. L.

*Xylose; Preparation of* — from corn [maize] cobs. C. S. Hudson and T. S. Harding. *J. Amer. Chem. Soc.*, 1918, 40, 1601–1602.

THE authors have previously described the preparation of crystalline xylose from cottonseed hulls (this *J.*, 1917, 730) and now find an even more suitable source in maize cobs. The yield is about 12%. The crushed cobs are boiled for two hours with 7% sulphuric acid; the residue is strained off and the filtrate exactly neutralised with calcium hydroxide, care being taken that the solution never becomes alkaline. The calcium sulphate is removed and the filtrate is decolorised with carbon or bone char after having been made just acid to litmus with phosphoric acid. After concentration under diminished pressure, alcohol is added to the solution; the precipitated calcium sulphate is removed, and the liquid is concentrated to a syrup from which, by suitable treatment with alcohol, the xylose is obtained in the crystalline form.—H. W.

*Lactose*. Panchaud and Auerbach. See XIXA.

## XVIII.—FERMENTATION INDUSTRIES.

*Beers; Belgian mashing system suitable for light* — G. M. Johnson. *J. Inst. Brew.*, 1918, 24, 237–246.

FOLLOWING an historical sketch tracing the influence of fiscal conditions on the development of Belgian mashing methods, the author describes the "turbid wort" system which is widely employed for the production of full-flavoured light beers. This system, carried out with plant similar to that used in this country, produces dextrinous worts by restricted conversion. The following is a brief outline. A stiff mash is held at 100°–110° F. (38°–43° C.) for 30–45 mins. for peptonisation, and then raised to 120°–125° F. (49°–52° C.) by underletting water at 150°–175° F. (65°–79° C.), after which the turbid wort is drawn off and gradually heated to boiling, with a pause for saccharification. The goods in the tun are meanwhile heated to 158° F. (70° C.) by introduction of hot water, and after saccharification a bright wort is obtained and drawn off to the bright wort copper. The boiling turbid wort is then returned to the tun, so as to raise the temperature of the mash to 167° F. (75° C.), at which saccharification is completed, the clear wort

thus obtained being then added to that already in the bright wort copper. Worts produced by this system do not weaken the yeasts employed. Belgian beers of gravity 1020–1040 produce at least 10 lb. of yeast (as collected at the troughs) per 100 lb. of malt, and often half as much again. It is suggested that the difficulties experienced with yeast in English breweries owing to the reduction of wort gravities, represent a passing phase due to a "displacement" of the characters of the yeast. Pitching yeast is regarded as a mixture of different types, and a reduction of working gravities may favour the predominance of a type previously present in only small proportions, and so change the character of the yeast and the flavour of the beer. To avoid this it is advisable, at each brew, to pitch a small tun of wort of normal gravity and to use the yeast therefrom for the lighter worts.—J. H. L.

*Brewery: Tracing infection in the —.* H. F. E. Hulton. *J. Inst. Brew.*, 1918, 24, 258–260.

A RATIONAL method of tracing infection deleterious to beer—devised by the author in conjunction with J. L. Baker—which has given good results, is described. Forcing tests are carried out on a series of wort samples taken at numerous stages of brewing from the turn-out of the copper onwards. Samples taken prior to pitching are fermented with a little pure culture yeast before the forcing test. Practical details are given. It has been observed that the liability of wort to infection varies inversely as its gravity.—J. H. L.

*Beers: Decarbonation of water used for diluting war —.* H. Krumhaar. *Woch. Brau.*, 1918, 35, 121.

THE author discusses the possible effects of carbonates present in waters used for diluting beers. The formation of precipitates by interaction with the salts in the beer is not to be feared, since the concentration of the reacting substances is very low and the temperature also is low. Carbonates will, however, reduce the acidity of the beer by partially neutralising free lactic acid. In some cases, e.g., lager beer, this is not of great importance; but where the character of a war beer depends on the presence of free acid, diluting water rich in carbonates should be decarbonated.

—J. H. L.

*Beers: Iron-sickness in thin —.* W. Windisch. *Woch. Brau.*, 1918, 35, 69–70, 95–96, 109–110.

IN Germany war beers of very low gravity have proved subject to a malady resembling the iron-sickness of wines, which is due to the formation of ferric tannate. The beers affected become greenish and eventually blackish in colour, and acquire an inky flavour. It is suggested that the iron is probably dissolved by the wort after fermentation owing to contact with naked metal, or is introduced into the beer in the water used in some cases for dilution. Decarbonation by boiling appears to render some waters more "aggressive" towards iron, so that contact of such waters with the metal, during cooling or afterwards, becomes a source of danger. (See following abstract.)—J. H. L.

*Beers: Maladies of thin —.* W. Windisch. *Woch. Brau.*, 1918, 35, 153–156.

THE war beers of low gravity produced in Germany have been remarkably free from maladies due to micro-organisms. The chief troubles have arisen from tannin-protein turbidity and iron-sickness (see preceding abstract), in both of which tannin

plays an essential part. Some amelioration might be effected by reducing the tannin-content of the beer, e.g., by using a smaller proportion of hops or extracting them with cold water before use, or by promoting the solution of proteins during mashing with a view to the subsequent precipitation of tannins. The fact that the maladies are almost confined to beers of 3% gravity or under, however, probably indicates that these beers are deficient in some protective constituent, which the author conceives to be free acid. He therefore advocates higher acidities in thin beers, and in this connection recommends treatment with lime as the most economical and effective means of decarbonating brewing waters.—J. H. L.

*Yeast cells: Diminution in the size of — in thin worts.* F. Schönfeld and C. Goslich. *Woch. Brau.*, 1918, 35, 153.

THE production of beers from worts of 3% gravity, as now practised in Germany, was found to yield yeast crops with cells appreciably smaller on the average than those from worts of higher or normal gravity.—J. H. L.

*Yeast: Influence of stimulants and of storage under water and beer, on the maltase-activity of —.* F. Schönfeld and M. Korn. *Woch. Brau.*, 1918, 35, 129–132.

TREATMENT of bottom-fermentation beer yeast with 1% solutions of monopotassium phosphate for several hours, was found to stimulate the activity not only of the zymase but also of the maltase (see this J., 1918, 161a). The maltase-activity was also increased by treatment with 0.6% solutions of lactic, phosphoric, and tartaric acids, especially by the first named. Yeast stored under water suffered a very slow reduction of maltase-activity, amounting to about 25% in 8 days. Storage under beer produced a more rapid change in the same direction. In general, conditions which affect the zymase of living yeast appear to exert a similar effect on the maltase.—J. H. L.

*Wine: Removal of acidity of — by addition of potassium tartrate.* W. I. Baragiola. *Schweiz. Ver. anal. Chem.*, May, 1918. *Chem.-Zeit.*, 1918, 42, 515–516.

THE addition of potassium tartrate to reduce the acidity of wine, either by precipitation of the tartaric acid as potassium bitartrate or by simply forming this salt in solution, may alter the relation between the quantities of total tartaric acid and non-volatile acids present in normal wines and thus render impossible the detection of added water and tartaric acid in sophisticated wines.—W. P. S.

#### PATENTS.

*Beer: Brewing — [without malt].* H. Boulard. Paris. Eng. Pat. 119,833, 7.5.18. (Appl. 7660/18.)

THE steeping and boiling of the grain and the addition of hops are all carried out in the same vessel. If the grain itself is not sufficiently acid, a small proportion of an organic acid, e.g. 3% of tartaric acid on the weight of the grain, is also added to assist the conversion of the starch into soluble dextrans, etc. The charge is boiled under a pressure of 4 atm. for about 35 mins., then cooled by water before opening the converter. A further quantity of hops may be added to the converted mash which is again boiled for 20 mins. In the open pan and a short time under pressure. The sterilised wort is saccharified in closed vessels at 38°–40° C. with pure cultures of *Mucor Boulard* No. 5, with a suitable supply of purified air so as



to convert the soluble starch and dextrins into fermentable sugars. The wort is again sterilised, if desired, by boiling and is then cooled and fermented either in separate vats or in the saccharifying vats, preferably by the use of pure cultures of yeast.—J. F. B.

*Fermentation: Process for accelerating* —. S. Fränkel and J. Fischl, Vienna. Ger. Pat. 307,545, 24.5.16. Int. Conv., 5.5.16.

FERMENTING liquids are treated with alcoholic extracts of embryos of plant seeds, of lower fungi (in particular dried yeast), or of animal embryos such as yolks of eggs.—J. H. L.

*Alcohol from sulphite [cellulose waste] liquors; Process of obtaining* —. R. H. McKee, Ridgefield Park, N.J., U.S.A. Eng. Pat. 120,520, 15.5.18. (Appl. 8158/18.)

SEE U.S. Pat. 1,273,392 of 1918; this J., 1918, 600 A.

*Beer; Art of brewing* —. L. Nathan, Zürich, Switzerland. U.S. Pat. 1,280,280, 1.10.18. Appl., 26.12.13.

SEE Eng. Pat. 564 of 1914; this J., 1915, 728.

*Cream of tartar*. U.S. Pat. 1,278,257. See VII.

## XIXA.—FOODS.

*Milk samples; Table for sorting* —. L. J. Harris. Analyst, 1918, 43, 375–377.

A TABLE is given by means of which it is possible to see at once, from the sp. gr. and total solids, whether a given sample of milk is above or below the minimum standard of the Board of Agriculture, and in the latter case whether the sample is deficient in fat, solids-not-fat, or both.—C. A. M.

*Milk; Refraction of* —. J. Pritzker, Schweiz. Ver. anal. Chem., May, 1918. Chem.-Zeit., 1918, 42, 528.

INCREASE of acidity of milk causes a corresponding increase in the refraction of the milk serum, the increase being due to solution of calcium phosphates by the lactic acid. When the quality of a milk is judged from its refraction, the acidity of the sample should be determined and taken into account.—W. P. S.

*Lactose [in milk]; Refractometric determination of* —. L. Panchaud and E. Auerbach. Schweiz. Ver. anal. Chem., May, 1918. Chem.-Zeit., 1918, 42, 528.

COMPARATIVE determinations of lactose in milk by means of the refractometer and by Allihn's method yielded concordant results, the differences not exceeding 0.03%. In the case of sour milk with an acidity of 28%, the result found by the refractometer was 0.7% too high; this was due to the conversion of the lactose into lactic acid, which has a higher refractive index. Milk from cows with diseased udders also gave high results for lactose when examined with the refractometer; this milk contained a high percentage of chlorides and of proteins which were not precipitated by calcium chloride.—W. P. S.

*Butter; Analysis of* —. P. Ereulisse and H. Dackweiler. Ann. Chim. Analyt., 1918, 23, 225–234.

THE authors determine the saponification value, the "silver value," and the "magnesium value" of the fat, and from these results calculate the

Reichert-Meissl, Polenske, and Hehner values of the fat, or figures which correspond with these values. This indirect method is stated to be more accurate than the usual direct method of determining the values. The saponification value is determined on 4 grms. of the fat; the neutral soap is then diluted to a definite volume and an aliquot portion of the solution is treated with an excess of N/10 silver nitrate solution, filtered, and the excess of silver titrated in the filtrate. All fatty acids, with the exception of butyric acid, are precipitated by the silver nitrate, and from the result is calculated what the authors term the true Reichert-Meissl value. Similarly, magnesium sulphate precipitates all acids higher than C<sub>12</sub>. The Polenske value calculated from the result of the magnesium precipitation is higher than the value determined in the usual way. Results of the examination of one sample of butter are recorded.

—W. P. S.

*Witgatboom, a substitute for chicory*. J. McCrae and A. Kloot. Analyst, 1918, 43, 373–374.

THE roots of four of the S. African trees known as "witgatboom" (white-hole tree) are used as chicory substitutes, viz., *Boscia transvaalensis* (most commonly used in the Transvaal), *B. rehmanni*, *Marua pedunculata* (Cape Province), and *Capparis albitrunca*. Commercial roasted samples showed under the microscope characteristic triangular pitted cells. On analysis they gave the following results:—Sp. gr. of 10% decoction, 1.0118 to 1.0244; total water-soluble matter, 31.0 to 68.5; ash in aqueous extract, 3.5 to 5.5; total ash, 4.3 to 6.9; and ash insol. in HCl, 1.1 to 1.7%. Results obtained with genuine samples of the roasted roots of (A) *Boscia transvaalensis* without the hard core; (B) the same root containing the core; and (C) *Marua pedunculata* are tabulated.—C. A. M.

*Silage; Chemistry of sweet clover — in comparison with alfalfa [lucerne] silage*. C. O. Swanson and E. L. Tague. J. Agric. Res., 1918, 15, 113–132.

LUCERNE, sweet clover, and a mixture of sweet clover and corn (maize) meal were placed in three series of bottles which were examined after increasing intervals of time. Good silage was produced in each case. Water and alcohol extracts were made and examined for acidity by electrometric and titration methods. It was found that the loss of weight in conversion into silage was approximately 1%. The titration acidity of the alcoholic extracts was greater than that of the water extracts, owing to the colouring matter extracted by the alcohol obscuring the end point, for when the electrometric method was used there was no appreciable difference between the acidities of the two extracts. Most of the acidity was developed in the first 15 days. The addition of maize meal to the sweet clover increased the production of acid. The amino-nitrogen was the same in the two extracts, but the lucerne silage yielded much more than the sweet clover silage. The amide-nitrogen formed about one-half of the total nitrogen. The solvent action of the water and the alcohol was about the same, two-thirds of the total nitrogen being extracted in each case. The practical conclusion is that silage can be made more easily from sweet clover than from lucerne.

—J. H. J.

*Rhubarb juice*. Besson. See XX.

*Drying plant materials*. André. See XXIII.

## PATENTS.

*Preserving [foods]; Vacuum process for — and other purposes.* W. S. Sellars, Brooklyn, N.Y., Assignor to American Can Co. U.S. Pat. 1,279,379, 17.9.18. Appl., 16.6.16.

In order to prevent the pin-holing of this of preserved food, the filled tins are placed in the liquid which is to be used as a preservative and are subjected to a vacuum, whereby air is withdrawn from the cells of the food and is replaced by the preserving liquid, which is allowed to fill the tins completely; they are then sealed.—J. H. J.

*Fodder; Process for preserving green —.* E. C. R. Marks, London. From C. Reinhardt, Berne, Switzerland. Eng. Pat. 120,256, 31.10.17. (Appl. 15,887/17.)

SEE U.S. Pat. 1,257,754 of 1918; this J., 1918, 280 A.

*Milk-sterilising apparatus.* F. H. Rogers, London. U.S. Pat. 1,280,851, 8.10.18. Appl., 15.8.17.

SEE Eng. Pat. 109,638 of 1916; this J., 1917, 1191.

*Drying fruit, etc.* Eng. Pat. 120,283. See I.

## XIXB.—WATER PURIFICATION; SANITATION.

## PATENTS.

*Filtering and purifying water; Device for —.* H. Bruck, Assignor to O. H. Krueger, Los Angeles, Cal., and M. E. Cline, San Francisco, Cal. U.S. Pat. 1,279,240, 17.9.18. Appl., 5.7.17.

The apparatus consists of a cylindrical filter-chamber with a hopper-shaped bottom. A stand-pipe with an enlarged base is supported a little way above the bottom of the hopper. The inlet pipe is near the top of the cylinder. Horizontal outlet pipes are arranged cross-wise near the lower part of the cylinder and are connected to openings in the side. There is also an outlet pipe in the bottom of the hopper. All the outlets lead into a single pipe provided with valves. A separator is placed in the space beneath the stand-pipe and above the outlet in the bottom of the hopper.—J. H. J.

*Water; Processes for softening —.* G. H. Uecke, Minneapolis, Minn., U.S.A. Eng. Pat. 120,293, 13.12.17. (Appl. 18,524/17.)

SEE U.S. Pat. 1,255,358 of 1918; this J., 1918, 221 A.

*Water; Apparatus for softening —.* G. H. Uecke, Minneapolis, Minn., U.S.A. Eng. Pat. 120,294, 30.12.17. (Appl. 18,525/17.)

SEE U.S. Pat. 1,255,359 of 1918; this J., 1918, 221 A.

*Disinfectant [formaldehyde]; Process of liberating a gaseous —.* A. Eckmann, Assignor to Schweiz. Serum- und Impfinstitut, Berne, Switzerland. U.S. Pat. 1,279,524, 21.9.18. Appl., 14.7.15.

SEE Eng. Pat. 9637 of 1915; this J., 1916, 941.

## XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

*Cinchona alkaloids. I. Cupreine, hydrocupreine, and their methyl and ethyl esters.* G. Giemsa and J. Halberkann. Ber., 1918, 51, 1325—1333.

Dihydrocupreine, prepared by the addition of hydrogen to cupreine in the presence of a palla-

dium or nickel catalyst, has m.pt. 201° C. Hesse (Annalen, 1887, 241, 281) gives 168°—170° C. Cupreine yields methyleupreine (quinine) by methylation by methyl sulphate or diazomethane. Methylidihydrocupreine is obtained by the catalytic reduction of quinine or by the methylation of dihydrocupreine. Ethyldihydrocupreine is prepared by similar methods.—C. S.

*Cinchona alkaloids. XX. Synthesis of quinotoxines.*

P. Rabe and K. Kindler. Ber., 1918, 51, 1360—1365.

N-BENZOYLHOMOCINCHOLEUFONE, prepared from benzoyldihydrocinchotoxine, is converted into its ethyl ester,  $C_{21}H_{27}O_7N$ , b.pt. 256° C. at 13 mm., which reacts with ethyl cinchonate and sodium ethoxide to form a substance, doubtless the  $\beta$ -ketonic ester, the hydrolysis of which by boiling 15% hydrochloric acid yields dihydrocinchotoxine (dihydrocinchonine). Since dihydrocinchotoxine has been converted into dihydrocinchoninone and the latter can be reduced to dihydrocinchonine and dihydrocinchonidine (future communication), the construction of cinchona alkaloids from derivatives of the quinoline and piperidine series has been accomplished. (See also J. Chem. Soc., Jan., 1919.)

—C. S.

*Strychnine alkaloids. XXIV. Cause of the violet reaction of cacotheline and of nitro-compounds of the brucine series allied to it.* H. Leuchs. Ber., 1918, 51, 1375—1389.

THE methonitrate of the cacotheline base,  $C_{21}H_{21}O_7N_3 \cdot CH_3 \cdot NO_2$ , obtained by the action of nitric acid on methylbrucine, is a nitrated quinone, since it is converted into the oxime,  $C_{22}H_{25}O_7N_4Cl$ ,  $2H_2O$ , yellow needles, of the methochloride of the cacotheline base by the action of hydroxylamine hydrochloride. It is converted by a hot aqueous solution of sodium bisulphite into a methosulphite of the cacotheline base,  $C_{21}H_{21}O_7N_3 \cdot CH_3 \cdot SO_3H$ , deep violet prisms. The latter contains the sulphite group in complex union, because sulphur dioxide is not eliminated by the action of strong acids, and is a nitrated quinol, since it is converted by nitric acid into a reddish-yellow methosulphite,  $C_9H_9O_2N_5 \cdot CH_3 \cdot SO_3H$ , which is reconverted into the violet compound by sulphurous acid or other reducing agents. Apart from their acidic groups, the methonitrate and the violet methosulphite are isomeric. The conversion of the former into the latter is, in the author's opinion, a case of intramolecular reduction. (See also J. Chem. Soc., Jan., 1919.)

—C. S.

*Nitro- and amino-arylarsonic acids.* W. A. Jacobs, M. Heidelberger, and I. P. Rolf. J. Amer. Chem. Soc., 1918, 40, 1580—1590.

THE preparation of a number of nitro- and amino-arylarsonic acids is described; the nitro-compounds were generally obtained by Bart's method (Ger. Pat. 250,264; Eng. Pat. 568 of 1911; this J., 1911, 1087), in which a diazo- or isodiazogroup is replaced by the arsenic acid residue. The procedure is particularly serviceable with *o*- and *p*-nitroamines; with *m*-nitroamines, on the other hand, the yields are poor though better with *m*-nitrotolylamines than with *m*-nitraniline. Reduction of the nitro- to the amino-group without disturbance of the arsenic acid residue was conveniently effected with cold alkaline ferrous hydroxide solution (compare Benda, Ber., 1911, 44, 3302). The following compounds have been prepared by these methods:—*o*-nitrophenylarsonic acid,  $NO_2 \cdot C_6H_4 \cdot AsO(OH)_2$ , m.pt. 235°—240° C. with decomp. (compare Bart, loc. cit.); *o*-aminophenylarsonic acid or *o*-arsanilic acid (compare Benda,



*loc. cit.*), needles, m.pt. 153° C.; *m*-aminophenylarsonic acid or *m*-arsanilic acid (compare Berthelm, this J., 1908, 622; Berthelm and Benda, Ber., 1911, 44, 3297), colourless rhombic prisms, m.pt. 213°–215° C. (decomp.); *p*-nitrophenylarsonic acid (compare Part, *loc. cit.*), pale yellow aggregates of minute leaflets which do not melt below 275° C.; *p*-aminophenylarsonic acid (*p*-arsanilic acid); 2-nitro-*p*-tolylarsonic acid, faintly yellow minute rods, m.pt. 255°–260° C. (decomp.); 2-amino-*p*-tolylarsonic acid, colourless needles, m.pt. 180° C. after softening and darkening; 6-nitro-*o*-tolylarsonic acid, pale yellow needles, decomposing at 228°–230° C.; 6-amino-*o*-tolylarsonic acid, rosettes or plates, decomposing at 175°–180° C.; 5-nitro-*p*-tolylarsonic acid (compare Michaelis, Annalen, 1902, 320, 321), cream-coloured needles, which do not melt below 285° C.; 5-amino-*p*-tolylarsonic acid, microscopic needles, m.pt. 172°–175° C.; 5-nitro-*o*-tolylarsonic acid (compare Karrer, Ber., 1915, 48, 311), m.pt. 261°–263° C. after melting or changing in appearance at about 225° C. according to the rate of heating; 5-amino-*o*-tolylarsonic acid, cream-coloured prisms, decomposing at 235°–245° C.; 4-nitro-*o*-tolylarsonic acid, minute needles, m.pt. 235°–240° C. (decomp.); 4-amino-*o*-tolylarsonic acid, microscopic needles, decomposing at 222°–224° C. (Benda and Kahn, Ber., 1908, 41, 1675, give 180° C.); 4-nitro-*p*-xylylarsonic acid, yellow crystals, m.pt. 290° C. (decomp.), which is not identical with the substance obtained by Michaelis (*loc. cit.*) by the nitration of *p*-xylylarsonic acid; 4-amino-*p*-xylylarsonic acid (compare Benda and Kahn, *loc. cit.*), colourless platelets, m.pt. 213°–214° C. (decomp.); 2-amino-4-hydroxyphenylarsonic acid, decomposing at 290° C. after darkening and softening at about 220° C.—H. W.

*Diethylaminoacetylsalicylic acid; Synthesis of derivatives of* — F. L. Hahn and M. Loos. Ber., 1918, 51, 1436–1447.

A NUMBER of substances were prepared partly with the object of obtaining a compound having certain advantages over aspirin. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Glycerophosphoric acid; Synthesis of optically active* — E. Abderhalden and E. Eichwald. Ber., 1918, 51, 1308–1312.

PHOSPHORYL chloride is added to a solution of *d*-α-bromohydrin in dry pyridine at a temperature below –10° C. The bromine is removed by means of lithium hydroxide and the product is isolated as nearly pure lithium *d*-glycerophosphate,  $\text{HO} \cdot \text{CH}_2 \cdot \text{CH}(\text{OH}) \cdot \text{CH}_2 \cdot \text{O} \cdot \text{PO}(\text{OLi})_2$ ,  $[\alpha]_D^{18} = +3.51^\circ$  in aqueous solution. Lithium *l*-glycerophosphate has  $[\alpha]_D^{18} = -3.02^\circ$ . (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Propyleneglycol; Optically active — and optically active β-hydroxybutyric acid.* E. Abderhalden and E. Eichwald. Ber., 1918, 51, 1312–1322.

β-CHLORO-α-PROPYLAMINE was resolved by *d*-tartaric acid and the *d*-tartrate, m.pt. 109.5° C.,  $[\alpha]_D^{18} = +35.72^\circ$  (in water), was converted by sodium nitrite into *d*-β-chloro-α-propanol, b.pt. 40°–41° C. at 15 mm. The chloropropanol was converted by aqueous potassium hydroxide at 50°–70° C. into *d*-propylene oxide, b.pt. 36.5°–38° C.,  $[\alpha]_D^{18} = +12.72^\circ$ , which by treatment with anhydrous formic acid and subsequent hydrolysis by 15% hydrochloric acid yielded *d*-propyleneglycol, b.pt. 95° C. at 15 mm.,  $[\alpha]_D^{18} = +13.71^\circ$  (in water). *d*-Propylene-oxide has been converted through *l*-β-bromoisopropyl alcohol and *l*-β-hydroxybutyronitrile into the biologically important *l*-β-hydroxybutyric acid, and *d*-β-chloro-α-propanol has been converted

through *l*-α-chloropropionic acid into *l*-alanine, and thus the configurative relations of these substances have been established. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Isoprene; Monohydrochloride of* — O. Aschan. Ber., 1918, 51, 1303–1307.

THE isoprene monohydrochloride described by Bouchardet in 1879 was almost certainly *tert*-isocamyl chloride. Isoprene monohydrochloride, prepared from carefully purified isoprene obtained from *d*-limonene, has b.pt. 109° C. and sp. gr. 0.9335 at 20° C. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Formic acid; Reducibility of* — K. A. Hofmann and H. Schibsted. Ber., 1918, 51, 1389–1398.

IN spite of all statements in the literature to the contrary, the authors have never obtained more than 4% of the expected yield in their attempts to reduce formic acid to formaldehyde and methyl alcohol by hydrogen under the most diverse experimental conditions. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Formaldehyde and methyl alcohol; Production of — from formates.* K. A. Hofmann and H. Schibsted. Ber., 1918, 51, 1398–1418. (Compare preceding abstract.)

METALLIC formates are able, to a degree dependent on the nature of the particular metal, to yield formaldehyde according to the equation  $2\text{H} \cdot \text{CO}_2\text{M} = \text{M}_2\text{CO}_3 + \text{CH}_2\text{O}$ . The temperature at which a distinct and sustained evolution of gas from the formate begins is in general higher the more strongly basic is the metallic oxide. For the following formates the temperatures are: Cu, 170° C.; Pb, 195°–200° C.; Ni, 210° C.; Zn, 240°–245° C.; Fe, 245°–250° C.; Mn, 295°–300° C.; Ba, 325° C.; Ca, 335° C.; Mg, 340°–345° C.; Sr, 355° C.; Li, 355° C.; Na, 355° C.; K, 375° C. The formaldehyde produced experiences, according to the nature of the metalliferous residue, diverse transformations, of which the most important is its conversion into methyl alcohol and formic acid. The vapour of formic acid, with or without hydrogen, yields carbon monoxide and steam or carbon dioxide and hydrogen when passed over a chemically unchangeable catalyst at a sufficiently high temperature. Considerable quantities of formaldehyde and methyl alcohol are produced, however, if the catalyst and the temperature of reaction are so selected that the formation of a formate is rendered possible. Zinc oxide and thoria are the best catalysts for this purpose. (See also J. Chem. Soc., Jan., 1919.)—C. S.

*Silver; Recovery of — from albumose-silver solutions. Methods for the analysis of these solutions.* G. Maue. Chem.-Zett., 1918, 42, 512–515.

LARGE quantities of 0.1% albumose-silver solutions are now used in certain hospitals and the recovery of the silver from the used solution is of importance. The following method is recommended for the purpose:—360 litres of the waste solution is treated with 60 grms. of sulphuric acid mixed with 200 c.c. of water, and 19 grms. of sodium chloride dissolved in 200 c.c. of water is added; the precipitate is allowed to settle, collected on a filter, dried, and fused with a mixture of potassium carbonate, 30, sodium carbonate, 30, and potassium nitrate, 15 grms. While still fluid, the mixture is poured into cold water and the metallic silver is collected. The cost of treating 360 litres of the solution is estimated to be M.0.12; the weight of silver recovered is 33.6 grms. having a

value of M.1375. An alternative method consists in precipitating the silver with ammonium thiocyanate in the presence of sulphuric acid; the silver thiocyanate is then treated as described. The estimated cost in this case is M.023. To determine silver in albumose-silver solution, the silver is first precipitated as metallic silver (by reduction) or as some insoluble salt which is fused with alkali carbonate and nitrate, and the silver is then dissolved in nitric acid and titrated. Methods using the following reagents yield accurate results: Lactose and sodium hydroxide, tannin, hydrochloric acid, potassium bromide and sulphuric acid, potassium iodide and sulphuric acid, ammonium thiocyanate and nitric acid, potassium ferrocyanide and acetic acid. Reduction with ferrous sulphate, or oxidation of the organic matter with permanganate, gives low results.—W. P. S.

*Ethylamine and diethylamine; Preparation of* —. E. A. Werner, Chem. Soc. Trans., 1918, 113, 899–902.

Five litres of 90% alcohol containing 490 grms. of ammonia was treated with 200 grms. of ethyl bromide and at intervals of 2 days eight diminishing quantities of ethyl bromide were added, the last quantity being 66 grms. and the total quantity 1186 grms. On the sixteenth day the solution was concentrated until nearly all the ammonium bromide had separated; it was then evaporated to dryness at 130°C. and the diethylammonium bromide, containing only about 5% of triethylammonium bromide, was separated from the ethylammonium bromide by extraction with chloroform. Diethylamine quantitatively displaces the tertiary amine from an alcoholic solution of triethylammonium chloride, and, similarly, ethylamine quantitatively displaces diethylamine. The three amines were separated by fractional addition of sodium hydroxide to an aqueous solution of the chlorides. Triethylamine behaved as the weakest base and the ethylamine as the strongest, behaviour which is contrary to the commonly accepted view.—C. S.

*Phenol [carrucrol] from the leaves of Coleus amboinicus*, Lour (*C. carnosus*, Hassk.). F. Weehuizen, Rec. Trav. Chim. Pays-Bas, 1918, 37, 355–356.

The fresh herb, *Coleus amboinicus*, Lour, gives a small yield of essential oil, 25 c.c. from 120 kilos., which contains a phenol identified as carrucrol.—W. G.

*Odorous substances; Spontaneous transformation of solutions of* — to a colloidal state by exposure to ultra-violet light. H. Zwaardemaker and F. Hogewind, Proc. K. Akad. Wetensch. Amsterdam, 1918, 21, 131–137.

SOLUTIONS of odorous substances in water, glycerol, or paraffin on keeping slowly pass into colloidal solutions which strongly exhibit the Tyndall effect. The change is accelerated by light, and particularly by ultra-violet light; thus a change which is effected in 30 minutes by means of ultra-violet light requires several weeks in daylight. (See also J. Chem. Soc., Jan., 1919.)—J. F. S.

*Rhubarb juice; Composition of* —. A. A. Besson, Schweiz. Ver. anal. Chem., May, 1918. Chem.-Zeit., 1918, 42, 527–528.

The following results were obtained on the analysis of 7 samples of rhubarb juice:—Total solids, 23.45 to 35.25; reducing substances, 4.5 to 12.7; ash,

4.1 to 6.9; ammonia-nitrogen, 0.13 to 0.45; protein-nitrogen, 0.05 to 0.22; oxalic acid, 2.11 to 3.53; tannin, 0.06 to 0.38 gm. per litre; total acidity 15.15 to 22.20 c.c., alkalinity of ash 42.0 to 82.8 c.c., and free organic acids 150.4 to 217.3 c.c. of N/1 solution.—W. P. S.

*Trypanosomes; Improved method for recovering* — from the blood of rats for antigen purposes in connection with complement fixation. F. H. Reynolds and H. W. Schoening, J. Agric. Res., 1918, 14, 573–576.

THE blood of rats which have been inoculated with trypanosomes for the purpose of the preparation of an antigen is collected in sodium citrate solution, and the mixture is strained through muslin and centrifuged for 20 mins. at 2000 revolutions per min. The clear liquid is drawn off and centrifuged again to recover any trypanosomes present. The first deposit of red corpuscles and trypanosomes is agitated with distilled water for 20 mins. in order to luke the corpuscles completely. The liquid is then centrifuged for half an hour, the red solution drawn off, and the deposit of trypanosomes is agitated with physiological salt solution and centrifuged again. The supernatant fluid is discarded, and preserving fluid is added to the deposited trypanosomes and stirred to produce a uniform suspension, which is then stored on ice. Experiments showed that the use of distilled water to luke the corpuscles had no detrimental effect on the antigen value of the trypanosomes, and the keeping qualities of the product were improved.—J. H. J.

*Mercury as a contact poison*. G. Bredig, Ber., 1918, 51, 1477.

THE poisoning of catalysts by mercury (compare Paal and Hartmann, this J., 1918, 560 A) has been previously noticed by Bredig and Berneck (Z. physik. Chem., 1899, 31, 327; compare this J., 1900, 1155) and others.—C. S.

#### PATENTS.

*Amyl acetate; Separation of* — from chloro-hydrocarbons. G. G. Oberfell and H. T. Boyd, Homer, Ohio, Assignors to The Ohio Fuel Supply Co., Pittsburgh, Pa. U.S. Pat. 1,278,198, 10.9.18. Appl., 22.1.18.

SYNTHETIC amyl acetate and its homologues, prepared by the esterification of chloro-hydrocarbons of the olefine or naphthene series, are purified by first fractionally distilling the esterified product, thereby separating the unsaturated olefine hydrocarbons formed in the reaction, then treating the residue formed by the decomposition of the chloro-compounds during esterification with sulphuric acid, which dissolves the amyl acetate and its homologues, leaving the other constituents, then separating the two layers and treating the amyl acetate solution to separate the amyl acetate and its homologues from the sulphuric acid.—J. F. B.

*Camphylcarbinol; Production of* —. H. Rupe, Basle. Ger. Pat. 307,357, 3.6.16.

ALCOHOLIC, aqueous alcoholic, or acetic acid solutions of oxymethylenecamphor, or aqueous solutions of its neutral alkali salts, are reduced by hydrogen under normal pressure, in presence of finely divided nickel or cobalt.—J. H. L.

*Catalytic substances*. Ger. Pat. 307,380. See VII.



## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

[Photographic] developing agents; Examination of organic ——. H. T. Clarke. Communication from Eastman Kodak Research Laboratory. Brit. J. Phot., 1918, 65, 499—503.

A SCHEME is outlined for the identification of 16 of the commoner developing agents, arranged in 4 groups according to their solubility in water, ether, and alcohol. Detailed descriptions are given of the characteristic reactions of each for qualitative examination and methods for quantitative estimation of most of them both singly and in mixtures. A number of typical analyses of developers actually on the market are also given showing in some cases a very considerable deviation from a reasonable standard of purity. For example a developer containing over 50% of lead chloride; a developer "identical to metol" containing 52% of quinol and only 32.8% *p*-methylaminophenol sulphate; metol substitutes containing respectively 33% of sucrose, 25.5% of pyrogallol with 73.4% of sodium sulphite, 85% of ammonium *p*-toluenesulphonate, and in two cases no methylated product at all. Numerous other adulterants were found, a list of 25 being given including starch, sucrose, citric acid, and various inorganic salts.—B. V. S.

### PATENTS.

*Colour photography.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,278,667, 10.9.18. Appl., 20.2.17.

Two colour-selection negatives, *e.g.* red and green, are first obtained. From the "red" negative is then obtained a blue-green positive and from the "green" negative a diapositive in some suitable colour contrasting with the blue-green. The blue-green film is then re-sensitised, exposed through the diapositive in register with it, and the image toned red.—B. V. S.

*Colour photograph or film and method of producing the same.* F. E. Ives, Philadelphia, Pa. U.S. Pat. 1,278,668, 10.9.18. Appl., 9.10.17.

A TRANSPARENT support provided with a film containing a sensitive silver salt is exposed through the support, developed, etc., then re-sensitised, exposed on the face, and developed to form a blue-green image. The silver image at the back is then copper-toned to a red colour.—B. V. S.

*Coloured [photographic] images; Process of producing —.* E. R. Bullock, Assignor to Eastman Kodak Co., Rochester, N.Y. U.S. Pat. 1,279,248, 17.9.18. Appl., 7.3.17.

A METALLIC image is oxidised and then reduced by an organic compound which forms, in the reduction process, an insoluble coloured compound. For example, a silver image is converted to the ferricyanide by the action of potassium ferricyanide and permanganate and then reduced by benzidine chloride. The silver salt is removed by fixing in "hypo" with the addition of a small quantity of an oxidising agent such as chromate.—B. V. S.

*Tinting process [for photographs] and product thereof.* J. I. Crabtree, Assignor to Eastman Kodak Co., Rochester, N.Y. U.S. Pat. 1,279,276, 17.9.18. Appl., 25.1.17.

THE gelatin of a photograph with an opaque image, such as silver, is tinted by treatment first with a solution of a suitable metal and then with a solution of a suitable precipitant, the coloured precipitate formed in this way being so finely divided as to be practically transparent.—B. V. S.

## XXII.—EXPLOSIVES; MATCHES.

*Explosives Physical Laboratory of the Bureau of Mines; Methods for routine work in the —.* S. P. Howell and J. E. Tiffany. U.S. Bureau of Mines. Tech. Paper 186, 1918. 63 pages.

DESCRIPTIONS are given of the methods used for determining the suitability of explosives for use in gaseous and dusty coal mines and in metal mines, tunnels, quarries, and engineering operations.

### PATENTS.

*Propellant-explosives; Gelatinised — and process of preparing same.* F. L. Nathan, W. Rintoul, and F. Baker, Stevenson, Assignors to Nobel's Explosives Co., Ltd., Glasgow. U.S. Pats. 1,280,278 and 1,280,279, 1.10.18. Appl., 26.2.14.

SEE Eng. Pats. 12,743 and 12,746 of 1912; this J., 1913, 991.

*Explosive.* A. Segay, Ware. U.S. Pat. 1,280,563, 1.10.18. Appl., 7.8.17.

SEE Eng. Pat. 113,083 of 1917; this J., 1918, 390 A.

## XXIII.—ANALYSIS.

*Silver-asbestos, lead chromate-asbestos, and lead-peroxide asbestos [for use in combustions].* O. Binder. Chem.-Zeit., 1918, 42, 522.

THE use of silver-asbestos is recommended in place of silver foil for the removal of chlorine in combustion analyses. It is prepared by reducing ammoniacal silver nitrate solution with metallic zinc, washing and drying the finely-divided precipitated silver, and then mixing it with ignited asbestos. A mixture of asbestos and lead chromate or lead peroxide is useful for the absorption of sulphur compounds.—W. P. S.

*Drying of plant materials at different temperatures.* G. André. Bull. Soc. Chim., 1918, 23, 430—437.

THE loss of water from plant materials, such as leaves or buds, when dried at 120° C. in a current of dry air, proceeds rapidly at first but soon slows down and there is a very slow but continuous loss over a long period of time. There is very little loss of organic matter during the drying at 120° C. in a current of dry air.—W. G.

*Metals; Electrolytic determination of — without the use of an external current.* M. François. Comptes rend., 1918, 167, 725—727.

A SELF-CONTAINED cell may be constructed by placing a strip of nickel across the top of a platinum crucible and suspending a zinc rod, 5 mm. in diam., from this strip; the lower end of the rod extends nearly to the bottom of the crucible. It is necessary to amalgamate the zinc rod at least 24 hrs. before it is used and during use it is provided with a filter-paper jacket held in position by cotton thread. For the deposition of silver or gold, the salt is dissolved in 2 c.c. of water in the crucible and 9 c.c. of 10% potassium cyanide solution, 5 c.c. of potassium hydroxide solution (36° B., sp. gr. 1.332), and 2 c.c. of ammonia (22° B., sp. gr. 1.18) are added. In the case of mercury the crucible should contain 20 c.c. of 10% sulphuric acid and 0.5 gm. of potassium iodide. The metals are deposited on the surface of the platinum crucible; the time required is 24 hrs., and the deposition is quantitative.—W. P. S.

*Germanium; Separation of — from arsenic, by distillation of the chloride in the presence of a chromate.* P. E. Browning and S. E. Scott. Amer. J. Sci., 1918, 46, 663—665.

ANOTHER modification of Buchanan's method (see this J., 1917, 1196) is described, chromic acid being used in place of permanganate or chlorate to oxidise the arsenious acid. The substance containing germanium oxide and not more than 0.25 gm. of arsenious oxide is warmed in a flask with 5 c.c. of 10% potassium bichromate solution and a few drops of sulphuric acid. About 10 c.c. of concentrated hydrochloric acid is then added and the mixture distilled; a current of carbon dioxide is passed through the apparatus during the distillation to facilitate distillation of the volatile germanium chloride and to remove any chlorine which might result from the action of the hydrochloric acid on the excess of chromic acid. The distillate is tested with hydrogen sulphide, a white precipitate of germanium sulphide being obtained in the presence of as little as 0.0005 gm. of germanium oxide.—W. P. S.

*Nitrogen; Pregl's micro-estimation of —.* H. Fischer. Ber., 1918, 51, 1322—1325.

UNTRUSTWORTHY results are obtained when the nitrogen in difficultly combustible substances is estimated by the micro-Dumas method (compare Kraemer, this J., 1918, 526A), and the author now uses only the original Pregl method ("Die quant. org. Mikroanalyse," 1917). The latter method is preferred to the Lassaigue test in detecting nitrogen in rare or very valuable organic compounds.—C. S.

*Temperature of gases.* Krelsinger and Barkley. See I.

*Calorimeter installation.* Davis and Wallace. See IIa.

*Oxidation of carbon monoxide.* Hofmann. See VII.

*Hydrogen phosphide in hydrogen.* Soyer. See VII.

*Scrap metals and cement copper.* Binder. See X.

*Sulphur in rubber.* Stevens. See XIV.

*Tannin estimation.* Lauffmann. See XV.

*Chromium estimation.* Lauffmann. See XV.

*Soil acidity.* Stephenson. See XVI.

*Raw sugars.* Horne. See XVII.

*Refraction of milk.* Pritzker. See XIXa.

*Lactose [in milk].* Panchaud and Auerbach. See XIXa.

*Butter analysis.* Erculisse and Dackweller. See XIXa.

*Albumose-silver solutions.* Maue. See XX.

*Photographic developers.* Clarke. See XXI.

## Patent List.

The dates given in this list are, in the case of Applications for Patents, those of application, and in the case of Complete Specifications accepted, those of the Official Journals in which the acceptance is announced. Complete Specifications thus advertised as accepted are open to inspection at the Patent Office immediately and to opposition within two months of the date given.

### I.—GENERAL; PLANT; MACHINERY.

#### APPLICATIONS.

Barber. Recovery of colloidal matters from liquids. 19,888. Dec. 2.

Barron and Barron. Impact grinders. 20,253. Dec. 6.

Bedford and Fuller. Furnaces. 20,734, 20,739. Dec. 12.

Berry, Hall, and Morgan. Heating, evaporating or calcining materials. 19,859. Dec. 2.

Bibby. Furnaces etc. 19,671. Nov. 28.

Christoffersen. Emulsifying liquid and like substances. 20,108. Dec. 4. (Denmark, 28,917.)

Davis and Twigg. Reversible regenerative gas-fired furnaces. 20,456. Dec. 9.

Dnnér. Filtering device for centrifugal separating machines. 19,577. Nov. 27. (Sweden, 22,117.)

Frey and Henz. Suppressing formation of froth or scum when boiling liquids. 20,169. Dec. 5. (Switz., 6,12,17.)

Kestner Evaporator and Engineering Co., and Reavell. Apparatus for cooling gases. 20,044. Dec. 4.

King and Rawson. Calcination. 20,533. Dec. 10.

Lamb and Pennington. 19,386. See II.

MacDonald. Production of extracts from vegetable materials. 20,355. Dec. 7.

Magrath. Apparatus for treating liquids with gases. 20,270. Dec. 6.

O'Brien. Continuous fractional distillation. 20,765. Dec. 13.

Pluperfect Refrigeration Co., and Ryder. Apparatus for making ice. 20,324. Dec. 7.

Wake. Grinding, crushing, and pulverising mills. 19,770. Nov. 29.

#### COMPLETE SPECIFICATIONS ACCEPTED.

12,291 (1917). Goyvaerts, Becquevort, and Bossut. Apparatus for fractional distillation of mixtures of liquids not miscible with water. (120,940.) Dec. 11.

16,355 (1917). Brooke. See VII.

17,064 (1917). Bates and Walker. Conveyor and its application to drying, mixing, screening, etc. (120,764.) Dec. 4.

17,982 (1917). Smith. See II.

18,349 (1917). Burns. See XI.

18,626 (1917). Hunter. Dry kiln. (120,809.) Dec. 4.

6062 (1918). Darasse. See VII.

6305 (1918). Schar. Grinding-mills. (115,424.) Dec. 18.

8011 (1918). Turner. Fractional or destructive distillation. (120,851.) Dec. 4.

8464 (1918). Carrier, and Carrier Engineering Corporation. Drying, conditioning, and regulating the moisture content of hygroscopic materials. (121,060.) Dec. 11.

12,966 (1918). Wilson. Filters. (120,867.) Dec. 4.

### II.—FUEL; GAS; MINERAL OILS AND WAXES; DESTRUCTIVE DISTILLATION; HEATING; LIGHTING.

#### APPLICATIONS.

Burnett. Apparatus for washing coal etc. 19,442. Nov. 26.



Chrystal. Producer gas plants. 19,940. Dec. 3.  
Clere. Gas generators. 20,098. Dec. 4. (Fr., 4,12,17.)

Climie and Lees. Gas producers. 20,390. Dec. 9.  
Climie and Lees. Gas-producing plant. 20,391, 20,392. Dec. 9.

Colson. Apparatus for synthetic production of gas from coal. 19,378. Nov. 25. (Fr., 26,11,17.)

Dalton. Artificial fuel composition and process. 19,645. Nov. 28. (U.S., 12,1,18.)

Davis and Twigg. 20,455. *See* I.

Dickinson (Ashworth). Apparatus for distilling or cracking heavy oils etc. 20,531. Dec. 10.

Findlater. Apparatus for continuous separation of paraffin wax from oil. 19,639. Nov. 28.

Ionides. Heating gaseous fluids. 19,827. Nov. 30.

Iverson. Gas-producers. 20,654. Dec. 11.

Lamb and Pennington. Treatment of coal, water, etc., and removal of substances therefrom. 19,386. Nov. 25.

Marks (Soc. Franco-Belge de Fours à Coke). Treatment of gases, producing distillation, coking, or gasifying coal etc. 19,677. Nov. 28.

Murphy. Manufacture of fuel from coke etc. 20,225. Dec. 6.

Smith. Gas-producers. 19,697 and 20,436. Nov. 28 and Dec. 9.

Temple. Means for gasification of coal, shale, etc. 20,880. Dec. 14.

Umsted. Manufacturing gas and obtaining by-products. 20,002. Dec. 3.

Westinghouse Lamp Co. Filaments of highly refractory metal. 19,460. Nov. 26. (U.S., 19,12,17.)

Westinghouse Lamp Co. Filaments. 19,595. Nov. 27. (U.S., 19,12,17.)

#### COMPLETE SPECIFICATIONS ACCEPTED.

15,445 (1917). Walster and Walster. Generating coal gas in retorts. (121,146.) Dec. 18.

16,879 (1917). Baker. *See* VII.

17,740 (1917). Bamber. Producer-gas plants. (121,161.) Dec. 18.

17,858 (1917). Penhale. Motor fuel. (120,792.) Dec. 4.

17,982 (1917). Smith. Cooling, cleansing, and filtering devices for gas. (121,177.) Dec. 18.

2445 (1918). Hayashi and Kaisha. *See* XVII.

6801 (1918). Doyle. Raising and drying peat. (120,850.) Dec. 4.

8011 (1918). Turner. *See* I.

9977 (1918). Zwilling. Converting beehive coke ovens into by-product ovens. (121,085.) Dec. 11.

13,533 (1918). Hamon. Fuel. (121,102.) Dec. 11.

#### III.—TAR AND TAR PRODUCTS.

##### APPLICATION.

Barrett Co. Process of sulphonating hydrocarbons and hydrocarbon derivatives. 19,686. Nov. 28. (U.S., 5,1,18.)

#### IV.—COLOURING MATTERS AND DYES.

##### APPLICATIONS.

Hart. Production of water-soluble dyes. 20,799. Dec. 13.

Imray (Soc. Chem. Ind. in Basle). Manufacture of mordant-dyeing dyestuffs etc. 20,534. Dec. 10.

Imrie (Geigy Soc. Anon.). Production of isatin and its substitution derivatives. 20,256. Dec. 6.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

##### APPLICATIONS.

Charlesworth. Machines for drying or carbonising wool, cotton, etc. 20,560. Dec. 11.

Distillates, Ltd., and Jarmain. Treatment of effluent from piece etc. scouring processes. 20,859. Dec. 14.

Soc. Gén. pour la Fabr. des Mat. Plastiques. Preparation of nitrocellulose. 20,283. Dec. 6. (Fr., 6,12,17.)

Testrup. and Wetcarbonizing, Ltd. Hydrolysing cellulosic materials. 19,389. Nov. 25.

#### COMPLETE SPECIFICATIONS ACCEPTED.

17,008 (1917). Frydensberg. Obtaining paper pulp and fibrous material from sea-tang. (120,761.) Dec. 4.

18,181 (1917). Knapp and Bartelds. Utilising old paper for producing new paper. (111,860.) Dec. 18.

7783 (1918). Unkle and Griley. Tub-beaters for paper pulp. (116,695.) Dec. 4.

#### VI.—BLEACHING; DYEING; PRINTING; FINISHING.

##### APPLICATIONS.

Dery. Mercerisation. 20,696. Dec. 12.

Lord. Apparatus for treating hanks of yarn with liquids. 20,116. Dec. 5.

#### COMPLETE SPECIFICATIONS ACCEPTED.

6017 (1918). Larivel. Machinery for dyeing and like treatment of textiles. (120,848.) Dec. 4.

7109 (1918). Lord and Lord. Machines for dyeing, washing, sizing, scouring, bleaching, and mercerising yarn in hank form. (121,075.) Dec. 11.

11,665 (1918). Clavel. Dyeing, bleaching, or like treatment of cops. (117,630.) Dec. 11.

#### VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

##### APPLICATIONS.

American Zinc Lead and Smelting Co. 20,208. *See* XIII.

Ashcroft. 20,714. *See* X.

Audienne. Heat-interchangers for gases applicable to the contact process for making sulphuric acid. 20,339. Dec. 7.

Bainbridge, Berry, Ollander, and Smith. Recovery of potash salts from furnace slag. 20,009. Dec. 3.

Cain, Couch, and Powell. Manufacture of potassium permanganate. 20,291. Dec. 6.

Hood. Purification of sulphur. 19,585. Nov. 27.

Norske Aktieselskab for Elektrokemisk Industri. Production of alumina from clay etc. 19,427. Nov. 26. (Norway, 24,1,18.)

Norske Aktieselskab for Elektrokemisk Industri. Removal of iron from solutions containing aluminium. 19,467. Nov. 26. (Norway, 23,2,18.)

Ramen. 19,826. *See* X.

#### COMPLETE SPECIFICATIONS ACCEPTED.

13,617 (1917). Clarke and Clarke. Separation of thorium from other rare earths and manufacture of thorium nitrate. (120,748.) Dec. 4.

16,355 (1917). Brooke. Concentration or evaporation of waste acid liquors. (120,951.) Dec. 11.

16,879 (1917). Baker. Recovery and utilisation of the cyanides in gas-works by-products. (120,759.) Dec. 4.

3767 (1918). Dutt and Dutt. Manufacture of alumina. (120,838.) Dec. 4.

6062 (1918). Darasse. Catalytic apparatus for producing oxychloride of carbon and other substances. (115,422.) Dec. 18.

9130 (1918). Linder. Manufacture of sulphate of ammonia. (121,082.) Dec. 11.

13,436 (1918). Lytle. Manufacture of lead arsenate. (121,101.) Dec. 11.

14,359 (1918). Dawson. Manufacture of nitric acid. (120,869.) Dec. 4.

## VIII.—GLASS; CERAMICS.

## APPLICATIONS.

- Bawtree. Staining glass for signalling. 19,171. Nov. 26.  
 British Thomson-Houston Co. (General Electric Co.). Composition for manufacture of glass. 19,592. Nov. 27.  
 Curd. Hydrogen-heated glass reservoir furnace. 20,306. Dec. 7.  
 Foster and Sankey. Manufacture of silica bricks, tiles, etc. 19,370. Nov. 25.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 11,513 (1917). Howard. Feeding molten glass from furnaces etc. (120,744.) Dec. 4.  
 17,474 (1917). Spinasse. Drawing glass. (114,819.) Dec. 11.  
 18,116 (1917). Feldenheimer. Treatment of clay. (121,191.) Dec. 18.  
 18,463 (1917). Maddock and Maddock. Kilns or ovens for use in making pottery, tiles, etc. (121,198.) Dec. 18.

## IX.—BUILDING MATERIALS.

## APPLICATIONS.

- Boehm, Ltd., and Reihl. 20,513. *See* XIII.  
 Melita. Manufacture of preservative for wood etc. 19,988. Dec. 3.

## X.—METALS; METALLURGY; INCLUDING ELECTRO-METALLURGY.

## APPLICATIONS.

- Acton. Process of using heat decomposition of nickel carbonyl to form protective coating of nickel on surfaces of oxidisable metal or alloy. 20,723. Dec. 12.  
 Amalgamated Zinc (De Bayays), Ltd., and Gancelin. Recovery of lead and silver from sulphide ores and metallurgical products. 20,761. Dec. 12.  
 Ashcroft. Cycle process for simultaneous production of magnesium metal or alloys and alkali chlorates. 20,714. Dec. 12.  
 Bainbridge and others. 20,009. *See* VII.  
 Charpy. Eliminating faults due to piping and segregation in ingots of steel etc. 20,377. Dec. 7. (Fr., 29,718.)  
 Dunningham, Hargreaves, and Hutchins. Manufacture of sodium. 20,634. Dec. 11.  
 Edser, Tucker, and Minerals Separation, Ltd. Concentration of ores. 20,541. Dec. 10.  
 Grover and Wigan. Aluminium alloys. 20,292. Dec. 6.  
 Harris, and National Metal and Chemical Bank, Ltd. Roasting sulphide ores. 19,993. Dec. 3.  
 Haywood. Copper-aluminium alloy. 20,550. Dec. 10.  
 Imbery. Hardening and tempering metals and apparatus therefor. 20,120, 20,375, 20,385. Dec. 5 and 7.  
 Leslie. Apparatus for testing hardness of metals etc. 19,866. Dec. 2.  
 McNab. Metal. 20,692. Dec. 12.  
 Metals Disintegrating Co. Process and apparatus for disintegrating metals. 20,080, 20,081. Dec. 1. (U.S., 20,12,17.)  
 Moore. Electric metallurgical furnaces. 20,378. Dec. 7.  
 Munden and Shilton. Crucible furnaces. 19,810. Nov. 20.  
 Queneau. Treatment of tin sulphide ores to separate metallic tin. 19,469. Nov. 26.  
 Ramen. Manufacture of burnt ore etc. bricks. 19,606. Nov. 27.  
 Ramen. Obtaining metals from end-lyes containing metal chlorides. 19,826. Nov. 30.  
 Rossi. Production of an acid-proof alloy. 20,840. Dec. 13.

- Westinghouse Lamp Co. 19,460 and 19,595. *See* 11.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 15,505 (1917). Jones. *See* XI.  
 16,197 (1917). Jensen. Preserving metallic surfaces. (121,150.) Dec. 18.  
 17,452 (1917). Roberts. Plating sheet metal. (112,125.) Dec. 11.  
 17,973 (1917). Minerals Separation, Ltd. Apparatus for concentrating ores. (112,019.) Dec. 18.  
 802 (1918). Tylor and Sons, Gaunt, White, and Brookfield. Smelting-furnaces. (121,213.) Dec. 18.  
 2936 (1918). Berglund. Extraction of lead. (114,303.) Dec. 4.  
 7039 (1918). Perry, and Industrial Inventions, Ltd. Production of malleable iron castings. (121,071.) Dec. 11.  
 8829 (1918). Mackie. Gold concentrators. (121,253.) Dec. 18.  
 10,626 (1918). Bennett. Furnaces for the heat treatment of steel. (118,598.) Dec. 18.

## XI.—ELECTRO-CHEMISTRY.

## APPLICATIONS.

- Elieson. Accumulator plates. 19,388. Nov. 25.  
 F.L.A.T. Soc. Anon. Electric furnaces. 20,746. Dec. 12. (Ital., 12,12,17.)  
 Harrison. Electric furnaces. 20,858. Dec. 14.  
 Imbery. Electric muffle furnaces. 20,375. Dec. 7.  
 Moore. 20,378. *See* X.  
 Soc. Electro-Metallurgique Française. Electrodes for electric furnaces. 19,756. Nov. 29. (Fr., 3,12,17.)  
 Tunnelly. Insulating materials. 20,489. Dec. 10.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 15,505 (1917). Jones. Electric muffle furnaces. (120,944.) Dec. 11.  
 17,612 (1917). Grammont. Electric furnaces. (120,786.) Dec. 4.  
 18,102 (1917). Chloride Electrical Storage Co., and Heap. Wood separators for secondary electric batteries. (121,188.) Dec. 18.  
 18,349 (1917). Burns. Electrical separation of suspended particles from gaseous bodies. (120,994.) Dec. 11.

## XII.—FATS; OILS; WAXES.

## APPLICATIONS.

- Blichfeldt. 20,079. *See* XIX.  
 Firth. Treatment of soya beans for recovery of oil and production of synthetic milk and flour. 19,672. Nov. 28.  
 Holderoff and MacIlwaine. Extraction of oil by volatile solvents. 20,125. Dec. 9.  
 King. Treatment of oils and fats. 20,175. Dec. 5.  
 Martin. Hydrogenating oils. 19,567. Nov. 27.  
 Park. Washing and drying copra. 19,609. Nov. 27.  
 Warburton. Refining oils. 20,733. Dec. 12.

## COMPLETE SPECIFICATIONS ACCEPTED.

- 13,135 (1917). Sizer. Apparatus for expressing oils etc. from seeds, nuts, etc., and for compressing the residuum into solid form. (120,747.) Dec. 4.  
 17,886 (1917). Escejay Co. Soaps and cleaning agents for use in laundries. (113,086.) Dec. 18.  
 17,996 (1917). Barton. Oil-cake and like hydraulic presses. (120,990.) Dec. 18.  
 19,305 (1917). Superior Oil and Process Co., and Schuck. Process of purifying oily materials. (120,820.) Dec. 4.  
 2445 (1918). Hayashi and Kaisha. *See* XVII.  
 3609 (1918). Norton. *See* XIX.  
 4523 (1918). Lockwood and Samuel. Treatment of fatty acids or their metallic salts, and manufacture of oxidised products therefrom. (121,237.) Dec. 18.



### XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

#### APPLICATIONS.

American Zinc Lead and Smelting Co. Preparing pigment zinc oxide. 20,208. Dec. 5. (U.S., 9,138.)

Boehm, Ltd., and Reihl. Manufacture of paint oils for cement, concrete, etc. 20,513. Dec. 10.

#### COMPLETE SPECIFICATIONS ACCEPTED.

18,342 (1917). Peachey. Plastic or solid material applicable also as varnish, lacquer, coating, or dope. (121,194.) Dec. 18.

4523 (1918). Lockwood and Samuel. *See XII.*

11,267 (1918). Peachey. Manufacture of a plastic or solid material applicable also as a varnish coating, dope, or lacquer. (121,091.) Dec. 11.

### XIV.—INDIA-RUBBER; GUTTA-PERCHA.

#### COMPLETE SPECIFICATION ACCEPTED.

147 (1918). Jackson (India Rubber Co.). Production of pigmented rubbers. (120,824.) Dec. 4.

### XV.—LEATHER; BONE; HORN; GLUE.

#### APPLICATIONS.

Bonamico. Tanning hides. 20,077. Dec. 4. (Italy, 4,12,17.)

Channon. Liquid glue. 19,897. Dec. 2.

Dorr Co. Recovery of proteids from waste liquors of hide-treating art. 20,284. Dec. 6. (U.S., 2,3,18.)

Drury. Leather compositions. 19,902. Dec. 2.

Forster. Treating hides and skins in manufacture of leather. 20,401. Dec. 9.

Stocks. Composition in lieu of leather etc. 19,763. Nov. 29. (Australia, 27,10,17.)

#### COMPLETE SPECIFICATION ACCEPTED.

18,176 (1917). White. Making composition from leather scrap. (120,802.) Dec. 4.

### XVI.—SOILS; FERTILISERS.

#### APPLICATION.

Allibon and Goulding. Apparatus for manufacture of superphosphates etc. 20,597. Dec. 11.

### XVII.—SUGARS; STARCHES; GUMS.

#### APPLICATIONS.

Goold (American Laundry Machinery Co.). Method and apparatus for preparing starch. 19,520, 19,529, 19,530. Nov. 27.

Martin. Manufacture of lactose or milk sugar. 19,692 and 20,836. Nov. 28 and Dec. 13.

Stephenson. Treatment or handling of sugar cane. 19,609. Dec. 11.

#### COMPLETE SPECIFICATION ACCEPTED.

2445 (1918). Hayashi and Kaisha. Manufacture of decolorising carbon. (121,035.) Dec. 11.

### XVIII.—FERMENTATION INDUSTRIES.

#### APPLICATION.

Wooldridge. Brewing. 19,764 and 20,429. Nov. 29 and Dec. 9.

### XIX.—FOODS; WATER PURIFICATION; SANITATION.

#### APPLICATIONS.

Berry, Mowbray, and Roberts and Co. Manufacture of baking powders and self-raising flours. 19,815. Nov. 30.

Blichfeldt. Apparatus for manufacture of margarine, etc. 20,079. Dec. 4.

Box. Destruction of house and other refuse and recovering useful materials. 20,427. Dec. 9.

Candy. Purification of water. 20,671. Dec. 12.

Cronsvive and Warnecke. Manufacture of feeding stuffs from marine plants. 20,197. Dec. 5. (Sweden, 30,8,15.)

Distillates, Ltd., and Jarman. 20,859. *See V.*  
Erslev. Manufacture of artificial milk. 19,673. Nov. 28. (Holland, 5,1,17.)

Erslev. Improving tobacco. 19,674. Nov. 28. (Holland, 15,12,17.)

Firth. 19,672. *See XII.*

Fruited Cereal Co. Prepared health food. 19,984. Dec. 3. (U.S., 4,12,17.)

Fuessler, Hepworth, and Wilton. Softening, treating, and filtering water. 20,216. Dec. 6.

Gee. Animal foodstuffs. 20,363. Dec. 7.

Hobley and Martin. Treatment of salvage flour, meal, spoilt cereals, etc. 20,013—20,015. Dec. 5.

Lamb and Pennington. 19,386. *See II.*

Liversedge. Treatment of sewage etc. 20,652. Dec. 11.

MacDonald. 20,355. *See I.*

Oxley. Alimentary product. 20,664. Dec. 12.

Tarrant. Obtaining substances of value from sewage. 19,974. Dec. 3.

#### COMPLETE SPECIFICATIONS ACCEPTED.

9291 (1917). Satow. Recovering proteids from vegetable proteid-containing material. (121,141.) Dec. 18.

17,442 (1917). Spencer. Preserving and sterilising milk, etc. (120,963.) Dec. 11.

17,458 (1917). Legg. Process of curing meats. (120,964.) Dec. 11.

18,044 (1917). Wilson. Apparatus for purifying water and other liquids. (121,180.) Dec. 18.

2123 (1918). Revis, Bolton, Downs, and Bellwood. Tanks or containers for ripening or souring milk or cream for use in making butter, margarine, etc. (121,033.) Dec. 11.

3609 (1918). Norton. Manufacture of margarine. (120,836.) Dec. 4.

### XX.—ORGANIC PRODUCTS; MEDICINAL SUBSTANCES; ESSENTIAL OILS.

#### APPLICATIONS.

Barbet et Fils et Cie., and Soc. Anon. de Prod. Chim. Etabl. Maletta. Manufacture of acetic ethers. 20,642. Dec. 11. (Fr., 20,10,17.)

Barrett Co. 19,686. *See III.*

Langley. Specific or medical compound. 20,702. Dec. 12.

#### COMPLETE SPECIFICATIONS ACCEPTED.

10,184 (1917). Marks (Nagai). Synthetic drugs. (120,936.) Dec. 11.

4523 (1918). Lockwood and Samuel. *See XII.*

### XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

#### APPLICATIONS.

Brown and Wellesley. Production of colour photography in natural colours on the film. 19,868. Dec. 2.

Rawlins. Photographic plates or negatives. 20,580. Dec. 11.

#### COMPLETE SPECIFICATION ACCEPTED.

4817 (1918). Scott. Coating films of celluloid etc. with sensitised photographic emulsion. (121,054.) Dec. 11.

### XXII.—EXPLOSIVES; MATCHES.

#### APPLICATIONS.

Marin. Explosives. 20,180. Dec. 5. (Belg., 12, 11, 17.)

Soc. Gén. pour la Fabr. des Mat. Plastiques. 20,283. *See V.*

### XXIII.—ANALYSIS.

#### APPLICATIONS.

Heathcote, and Rudge-Whitworth, Ltd. Means for ascertaining composition, nature, and effect of gases and vapours. 19,796. Nov. 30.

Leslie. 19,866. *See X.*













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